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EDITORIAL

This XXI Volume of the Journal of Food Physics is a special one. From 2 points of view. First, this is a proof of our 2 decades activity, because the first issue was published in 1988, i.e. 20 years ago. So with this issue we celebrate our 20 years of existence, as well. Thanks for your kind help, cooperation, support and understanding.

Secondly, this one is a special issue, because this is a proceedings of the 8th International Conference of Food Physics, which was organized in Bulgaria, 24-27 September, 2008, in Plovdiv at the Paisii Hilendarski University. I am sure we had a wonderful and very useful conference with appr. 80 participants from many different countries, from Albania via France to Turkey. As You probably know the first conference we organized in Budapest, Hungary, 1994, followed by the second one in Bucharest, Romania, 1996. The place of the third meeting was Poland, Lublin, 1998, and in 2000 we met in Turkey, Istanbul. Later we decided to organize the conference in Brno, Czech Republic, 2002, and 2 years later, in 2004 we came back again to Hungary, but the place was Pecs. The previous meeting we had in Serbia, in a beautiful small town, Senta, 2006. So we are really happy to have the possibility to continue the organisation of the ISFP conferences, and I myself would like to thank for the excellent work of the bulgarian colleagues taking part in organisation of the 8th ISFP conference in Plovdiv.

This issue gives opportunity also for those, who did not have the possibility to come to Plovdiv, and take part in the work of the meeting, but are interested in some special questions of food physics. The proceedings include not only the oral lectures but the poster presentations, as well. The topics are really wide, from food quality and safety to nondestructive techniques and nanoscience. Unfortunately some authors produced too long manuscripts, so we had to reduce significantly the volume of the papers.

Read and enjoy this issue! And do not forget to prepare for the next ISFP meeting, in Nitra, Agricultural University, Slovakia, 2010.

Prof. A.S. SZABÓ
Editor-in-chief

APPLICATION OF FLORAL SCENT ANALYSIS IN THE VERIFICATION OF HONEY AUTHENTICITY

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INTRODUCTION

The price of honey depends on its origin, monofloral honeys being the most highly appreciated. Certain types of honeys have been acknowledged by the European Union as a PDO (Protected Designation of Origin) product, which means among others that the product has unique quality. (*E.g.: Meli Elatis Menalou Vanilia, Mel de Galicia, Miel de Provence.)* Therefore (apart from deliberate honey adulteration, which is a major concern in honey trade and marketing) verification of the floral and geographical origin of a honey is in the focus of honey analysis. There are two main approaches to the problem. The first deals with several composition parameters (sugar ratio, moisture, ash content, enzyme activities etc.) and uses principal component analysis or other statistical devices for categorizing the samples [1]. The second searches for markers of the origin, *e.g.* special components deriving from the flower. [2], [3]

Our work applies this second approach searching for common compounds in the flower and the honey. In the recent study we have investigated three types of honeys and the corresponding flowers searching for identical compounds in them. The honey samples have been purchased from artisanal honey producers and derived from two consecutive harvest seasons. According to the producers' knowledge honeys were monofloral. Flower samples originate from bee pastures near to the nectar collecting fields. The honey samples were stored in screw-capped glass sample jars at room temperature in dark until required for analysis. Flower samples were processed immediately (*i.e.* during the ensuing day of the collection.)

MATERIALS AND METHODS

Sample preparation

Flower samples: Solidago, Tilia, Limonium sp. flowers
200 g sample, homogenization, chopping.
Internal standard: 0.4 mg undecan-1-ol
200 g sample+900 cm^3 distilled water+200 g NaCl (salting out)+internal standard – distillation in modified Likens-Nickerson distillation-extraction apparatus for 1,5 hour, extract in n-pentane
Extract drying over dehydrated sodium sulphate
Extract concentration by evaporation to 1 cm^3
1 ml extract gas chromatographed
Honey samples: Solidago, Tilia, Limonium unifloral honeys
900 g of honey sample+600 cm^3 distilled water+
0.4 mg ISTD+200 g NaCl – distilled according
to the flower method

GC-MS analysis

Instrument:	Hewlett Packard 5890/ II GC - 5971A MSD
Column:	60 m x 0.25 mm ID Supelcowax 10 (fused silica)
Film thickness:	0.25 mm
Initial temperature:	$T_1 = 60\ ^oC$,
Temperature progr. :	$v_{heat} = 4\ ^oC/min$
Final temperature:	$T_2 = 280\ ^oC$,
Det.temp. (tf.line):	$T_{det} = 280\ ^oC$
Carrier:	He, v_{lin}= 30.0 cm/s
Injector:	split/splitless, pin = 160 kPa, $T_{inj} = 250\ ^oC$
Injector mode:	splitless mode
Delay:	0.35 minute
Split ratio:	100 : 1
Ion source:	EI , excitation energy 70 eV
Mass range:	*m/z* = 35 - 350
Scan speed:	390 mass/s

RESULTS AND DISCUSSION

In the recent study three honey samples have been investigated which some way or other could be regarded as specialities.

Limonium species could be find on saliferous soils and its honey is very rare as monofloral nonetheless it is a delicious honey.
Solidago or goldenrod is a late bloomer, flowering in late summer into the fall and gives beekeepers the last opportunity of collecting honey. Solidago honey therefore appears as monofloral only in the "bad" harvest seasons, when producers are compelled to take advantage of the autumn collecting season. Solidago honey is dense and aromatic.
Linden honey is not exactly rare, but is highley appreciated and a pricey one. The cause is the erratic nature of Tilia trees, which give nectar mainly during the night and therefore beekeepers do not prefer. Because of high price it is prone to be adulterated.

The Likens-Nickerson simultaneous distillation-extraction equipment proved to be very suitable for sample preparation giving solutions rich in volatile components. The GC-MS analysis discovered numerous compounds in the distillates and made possible the characterisation of the samples.

Nevertheless no unique compound has been found in the distillates of the Limonium flower and honey in spite of the abundance of volatile and measurable components. The sample contained only derivatives that are ubiquitous in the plants and are not characteristic of the Limonium species.

In case of linden (Tilia) however three common compounds have been found in the flower and honey distillates. Linden ether (a name from the source, *i.e.* marker), cis-Rose-oxid and Chrysanthenon have not been found yet in any other investigated honey samples. These compounds could be used as marker compounds and could prove the floral origin of the honey.

Goldenrod (Solidago) honeys and flowers show very special volatile spectra due to the presence of sesquiterpene Germacrene D and its derivatives. Germacrene is not uncommon in plants because it is a key intermediate in the biosynthesis of many sesquiterpenes. Literary sources do not however report any occurence of it in honey samples neither it has appeared in our honey samples investigated so far. Germacrene D, delta-

Elemene and delta-Cadinene seem to be good marker compounds of the floral origin of Solidago honeys.

N°.	t_R (min)	PTRI	Compounds	Q %	Rel.Int.
			Terpenes, sesquiterpenes and derivatives		
1	4.066	1056	alpha-pinene	96	0.28
6	15.416	1397	cis-linalool oxide	91	27.81
8	16.459	1429	trans-linalool oxide	90	15.54
12	19.359	1516	linalool	97	1.24
13	20.925	1563	3,5,5-trimethylcyclohex-2-ene-1-on	91	2.53
14	21.466	1579	1-4-terpineol	97	1.69
15	21.759	1588	hotrienol	86	17.88
16	21.941	1593	.alpha.,4-dimethyl-3-cyclohexene-1-acetaldehyde	80	0.61
19	23.54	1642	2-hydroxy-3,5,5-trimethyl-2-cyclohexenone	94	1.41
22	25.886	1712	(Z)-(.+-.)-3,4,7,8,9,10-hexahydro-10-methyl-2H-oxecyn-2-one	90	1.29
23	26.385	1727	epoxylinalool	90	3.86
24	27.28	1754	epoxylinalool	91	4.27
44	42.108	2200	veridiflorol	91	1.65
55	46.566	2338	14B-pregnane	91	0.72
56	46.812	2343	14B-pregnane	91	0.89
60	49.242	2414	octahydro-alpha-camphorene	70	1.51
65	51.516	2483	nerolidol	95	52.97
71	55.764	2611	cis-bicyclo[4.3.0]-3-nonene	93	9.24
74	56.909	2645	trans-anti-trans-tricyclo[7.3.0.0(2,6)]-7-dodecene	81	14.49
79	61.041	2770	rimuene	86	8.47
			Open chain saturated and unsaturated hydrocarbons		
2	4.272	1062	1-decene	94	0.57
4	8.696	1195	1-dodecene	97	1.42
10	17.855	1471	n-pentadecane	96	0.33
18	23.358	1636	1-hexadecene	98	1.36
21	25.462	1700	heptadecane	95	1.35
29	32.567	1913	nonadecane	98	3.01
31	32.952	1925	1-heptadecene	91	1.22
34	35.886	2013	eicosane	97	0.58
40	39.223	2114	heneicosane	94	10.23
45	42.355	2208	docosane	98	3.12
51	45.62	2308	tricosane	98	49.64
52	45.819	2313	(E)-9-tricosene	99	5.00
53	46.034	2320	(Z)-9-tricosene	99	2.50
58	48.192	2384	hydrocarbon	94	2.30
59	48.828	2404	tetracosane	95	1.62
63	51.05	2470	10-methyl-eicosane	93	18.58
66	52.818	2522	1-nonadecene	98	4.15
72	56.161	2624	hydrocarbon		5.67
73	56.627	2637	hydrocarbon		8.56
75	58.036	2679	(Z)-9-tricosene	93	3.37
76	59.041	2711	hydrocarbon		2.79
			Open chain aldehydes and ketons		
3	7.023	1145	2-heptanone	94	0.51
5	13.692	1345	nonanal	95	0.48
7	15.695	1406	5-tetradecene	94	1.85
9	17.624	1464	decanal	87	0.58
35	36.194	2022	2-tridecanone	83	0.73
41	39.635	2126	6,10,14-trimethyl- 2-pentadecanone	91	6.70

			Esters		
11	19.056	1507	ethylnonanoate	95	2.20
27	30.614	1856	ethyldodecanoate	97	5.72
37	36.902	2044	isopropylmyristate	93	0.72
38	37.276	2055	ethyltetradecanoate	94	17.42
39	38.719	2098	3-hydroxytridecane acid ethylester	80	0.89
47	43.67	2247	ethylhexadecanoate	97	27.10
48	44.306	2266	ethyl-9-hexadecenoate	96	3.48
50	45.248	2294	decanedioic acid diethylester	86	7.34
61	49.65	2428	ethyloctadecanoate	99	3.59
62	50.225	2445	(Z)-9-octadeceneacid ethylester	99	100.00
64	51.35	2478	ethyl linoleate	99	20.18
67	53.111	2532	(Z,Z,Z)-9,12,15-octadecatriene acid, ethylester	99	43.31
78	60.629	2757	ethyldocosanoate	80	3.61
			Compounds with benzene ring		
25	27.551	1762	methylsalicylate	95	1.19
26	30.315	1845	3-phenyl-furan	95	2.27
28	32.29	1905	phenylethylalcohol	93	3.74
30	32.805	1920	benzeneacetonitril	81	1.35
32	33.785	1950	2-methoxy-4-methylphenol	74	0.64
33	35.292	1995	2-methyl-1,1-diphenyl-1-propene	74	1.45
36	36.432	2029	trans-cinnamonaldehyde (3-phenyl-2-propenal)	90	0.37
17	22.847	1621	benzeneacetaldehyde (Hyacinthin)	91	27.90
42	40.51	2152	eugenol (2-methoxy-4-(2-propenyl)-phenol)	98	2.59
43	41.189	2172	thymol (5-methyl-2-(1-methylethyl)-phenol)	91	3.41
46	43.235	2234	3-hydroxy-4-phenyl-2-butanone	74	2.05
49	45.077	2289	2,4-bis(1,1-dimethylethyl)-phenol	97	1.97
54	46.387	2328	3,4,5-trimethyl-phenol	93	2.41
57	47.274	2357	3,5-dimethoxy-benzoeacid methylester	97	1.57
68	53.376	2539	benzylbenzoate	96	9.05
			Organic acids		
20	24.832	1680	pentanoic acid	90	5.08
			O-containing heterocyclic substances		
69	54.185	2564	(Z)-octadec-9-ene-18-olide	95	19.05
70	55.441	2601	docosanolide	90	9.96
77	59.412	2722	cyclotetracosane	90	3.06

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MICROWAVE AND CONVENTIONAL HEATING EFFECTS ON SOME PHYSICOCHEMICAL PARAMETERS OF HIGH OLEIC AND LINOLEIC SUNFLOWER OIL

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ABSTRACT

The effect of microwave heating on some physicochemical parameters and oxidative stability of two sunflower oil (oleic and linoleic type), which have different lipid composition was studied. Each oil was heated by microwave energy of 360, 600 and 900W for 3, 6, 9 and 12 minutes. The results were juxtaposed to the ones, received by conventional heating and duration of 18 minutes. It was determined that by microwave and conventional heating of 900W for of sunflower oil no hydrolysis processes are observed. The oxidation degree in both types of oil increases with the increase of energy and the duration of microwave heating. The peroxide value and the conjugated dienes after 12 min of microwave heating at maximum power increase about 2 times, irrelevant of the fact that the two types of oil have a different oxidation stability and different degree of oxidation. The colour value increases, and the quantity of tocopherols decreases about 1.5 times. The nature of the oxidation changes in conventional heating does not differ significantly from those in microwave heating.

INTRODUCTION

Traditional conventional heating in the form of frying, baking and other types of culinary processing is the most commonly used in food preparation and production. Under the influence of high temperature and in the presence of moisture and atmospheric oxygen the processes of oxidation, hydrolysis, polymerization and destruction of oils are activated, which leads to changes in the sensory and nutritional qualities of foods. In the recent years microwave heating, which is more efficient and drastically reduces the duration of cooking, is more and more widely used

in thermal processing of foodstuff. The influence of microwaves on the nutritional value of products and their ingredients, however, is still not thoroughly studied. A great number of the studies on this issue are targeted towards one of the basic nutrients – lipids and even to a greater extent to the vegetable oils, rich in unsaturated fatty acids. Different in type and origin lipids were studied during microwave heating, and also the basic physicochemical values of vegetable oils [Stefanudaki E. et al., 1999], the degree of oxidation [Farag R. et al., 1992; Stefanudaki E. et al., 1999; Marinova E. et al., 2001], the changes in the triacylglycerols [Yoshida H. et al., 2002], fatty acids [Farag R. et al.,1992; Stefanudaki E. et al., 1999], tocopherols [Yoshida H. et al., 1991; Yoshida H. et al., 1992; Yoshida H. et al., 1999; Marinova E. et al., 2001] and phospholipids. Some of the authors [Stefanudaki E. et al., 1999] have established that in low and medium intensity of heating of the olive, corn and sunflower oils no significant changes in the quantity of free acid and the composition of fatty acids are observed. Most authors [Farag R. et al., 1992; Yoshida H. et al., 1991; Marinova E. et al., 2001] have determined significant changes in the basic physicochemical values and to oxidation changes of the lipids in sunflower. The peroxide value, the conjugated dienes and trienes are increased, and the quantity of unsaturated acids, tocopherols and phospholipids is decreased. The contradictory resource data on the behaviour of lipids during microwave heating, as well as the insufficient data about the main type of vegetable oil consumed in Bulgaria - sunflower oil, have attended our attention to its behaviour under these circumstances. The purpose of this paper is to carry out a comparative study of the changes in the physicochemical and chemical characteristics of two types of sunflower oil - oleic and linoleic type, which occur in microwave and conventional heating.

MATERIALS AND METHODS

Oils. Sunflower oil (the oleic type) and classical sunflower oil (the linoleic type) were obtained from the market network. Both types of oil were taken and examined 4 months after the production date.

Microwave and conventional heating. The samples of oil were placed in glass beakers of 100 ml and were heated in an *LG Gold* microwave oven at different, but traditionally used for cooking purposes powers at 360, 600 and 900 W for a duration of 3, 6, 9 and 12 min. Parallel to the

microwave heating, conventional heating was also conducted – on an electric hot-plate of 900W with duration of 6, 12 and 18 min. Oil samples were cooled at room temperature to 23 ± 1ºC for the analysis.

Analytical methods. The physicochemical values and the composition of the oil were determined by ISO methods - fatty acids composition [ISO 5508], acid value [ISO 3961], peroxide value [ISO 3960], Lovibond colour [ISO 15305], tocopherols [ISO 9936] and Rancimat oxidation stability at 100^0C [ISO 6886]. Absorption at 232 nm and UV-scans (220-320 nm) were determined following the analytical methods described by IUPAC (1979) method II.D.23 [Standard methods for the analysis of oils, fats and derivatives. 6. 1979]. Oil samples were dissolved in isooctane (0.2% solution). A Perkin-Elmer Lambda 15, UV/VIS Spectrophotometer device was used to determine absorptivity at UV spectrum. All data are presented as a mean value of three separate measurements ± standard deviation (SD, at P=0.05).

RESULTS AND DISCUSSION

The basic physicochemical characteristic, the fatty acids and tocopherols composition of the two studied types of sunflower oil are presented in Table 1.

Table 1
Characteristic values and composition of sunflower oil of oleic and linoleic type

Physicochemical characteristic	**Sunflower oil**	
	oleic type	**linoleic type**
Acid value, (mg KOH/g)	0.2±0.01	0.3±0.05
Peroxide value, ($meqO_2$/kg)	5.8±0.5	14.9±0.5
Tocopherols, (mg/kg)	145±10	336±10
Oxidation stability, (h)	23±0,5	10±0,5
Fatty acids composition, %		
Myristic ($C_{14:0}$)	tr.	0.2±0.1
Palmitic ($C_{16:0}$)	4.2±0.1	7.5±0.1
Palmitoleic ($C_{16:1}$)	tr.	0.5±0.1
Stearic ($C_{18:0}$)	3.0±0.1	4.2±0.1
Oleic ($C_{18:1}$)	81.5±0.1	36.4±0.1
Linoleic ($C_{18:2}$)	11.5±0.1	51.2±0.1

From the data in Table 1 it can be observed that the output oils have the anticipated fatty acids composition. The oleic type of oil contains 81.5% oleic acid, and the linoleic type – 51.1% linoleic acid. This composition determines the established more than twice greater stability of the oleic type of oil compared to that of the linoleic type. The linoleic type of oil has a greater quantity of tocopherols than the oleic type – respectively 336 and 145 mg/kg, with identical composition – about 98% α-tocopherol.

Table 2
Basic physicochemical values of sunflower oil (oleic and linoleic type), subjected to microwave and conventional heating for different periods of time and different power

Time	Acid value	Toco-pherols	Lovi-bond	Acid value	Toco-pherols	Lovi-bond
min	mgKOH/g	mg/kg	Colour	mgKOH/g	mg/kg	Colour
	Oleic type sunflower oil			Linoleic type sunflower oil		
0	0.2	145	2.8	0.3	336	4.2
Microwave heating at 360 W						
3	0.3	136	2.8	0.4	326	5.6
6	0.3	131	2.8	0.4	316	5.8
9	0.4	116	2.8	0.5	253	7.4
12	0.4	108	3.4	0.5	243	9.8
Microwave heating at 600 W						
3	0.4	126	2.8	0.4	300	5.4
6	0.4	116	2.8	0.5	286	6.4
9	0.4	110	3.1	0.5	256	9.4
12	0.4	102	4.4	0.5	203	12.6
Microwave heating at 900 W						
3	0.4	120	9.0	0.5	282	5.6
6	0.4	106	9.6	0.5	257	9.4
9	0.4	101	10.8	0.5	231	12.1
12	0.5	98	11.8	0.5	189	13.4
Conventional heating						
6	0.2	136	2.8	0.4	308	4.8
12	0.2	131	4.4	0.4	220	6.4
18	0.3	100	6.0	0.5	200	8.0
SD	±0.01	±10	±0.1	±0.01	±10	±0.1

The oils correspond to the requirements for quality assurance of vegetable oils [Bulgarian State Standard 1-77.Sunflower Oil] with the exception of the higher peroxide value of the oil (linoleic type). This shows that this type of oil is in the initial phase of oxidation, which could be explained with poor storage in the market network. Because of our desire to study the actual sunflower oil used by consumers, we studied the changes occurring in it during the next microwave heating, juxtaposed to the ones in using oleic type of oil, stored for the same period of time.

The results from the research carried out on the degree of hydrolysis and oxidation and the changes in the outward appearance of the two types of oil, subjected to microwave and conventional heating for different periods of time and different power settings, are presented in Table 2.

The data from Table 2, which follow the behavior of the linoleic type of oil during microwave and conventional heating give slightly distinguishable hydrolysis processes in the oil, which coincides with the data reported by other authors [Stefanudaki E. et al., 1999; Marinova E. et al., 2001]. The degree of deterioration does not differ from that observed in conventional heating. The results from studying the composition and basic physicochemical values of oleic type sunflower oil show changes occurring after microwave and conventional heating, but compared to the linoleic type of oil they are much more slightly distinguishable. The degree of hydrolysis at microwave heating is also very slightly distinguishable and coincides with the one in conventional heating.

Destructive processes also occur – the linoleic type sunflower oil gets darker – from 4.2 Lovibond colour value it reaches 9.8 – 13.4 after 12 min of microwave heating. The quantity of tocopherols decreases – after 12 min of heating at 900 W from 336 mg/kg to 189 mg/kg. The tocopherols in the oleic oil also decrease respectively from 145 mg/kg to 98-108 mg/kg, and the colour value increases from 2.8 to 3.4-11.8 – after 12 min of heating at 900 W.

More notable are the changes in the values reflecting the degree of oxidation of the samples. The peroxide value of the linoleic type of oil constantly increases with the increase of power and duration of microwave heating, and from 14.9 meqO_2/kg after 12 min heating it reaches values of 31.7 – 40.9 meqO_2/kg (Fig.1).

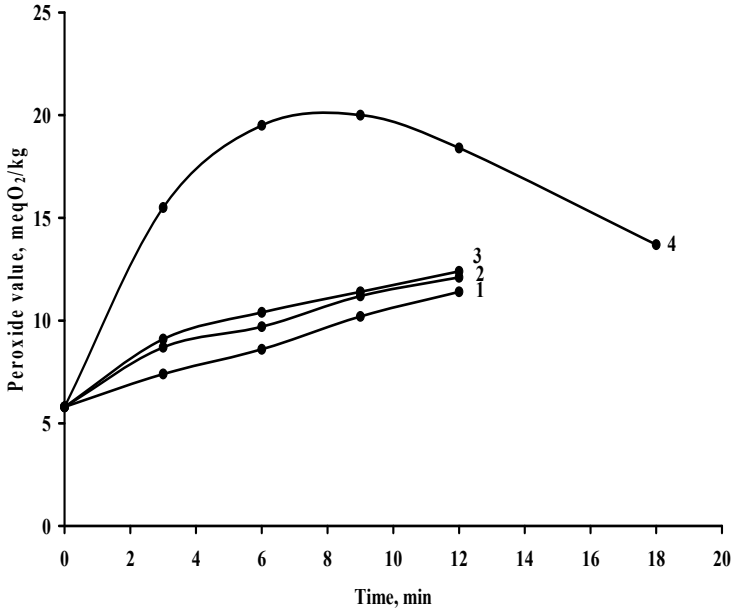

Figure 1
Change of the peroxide value

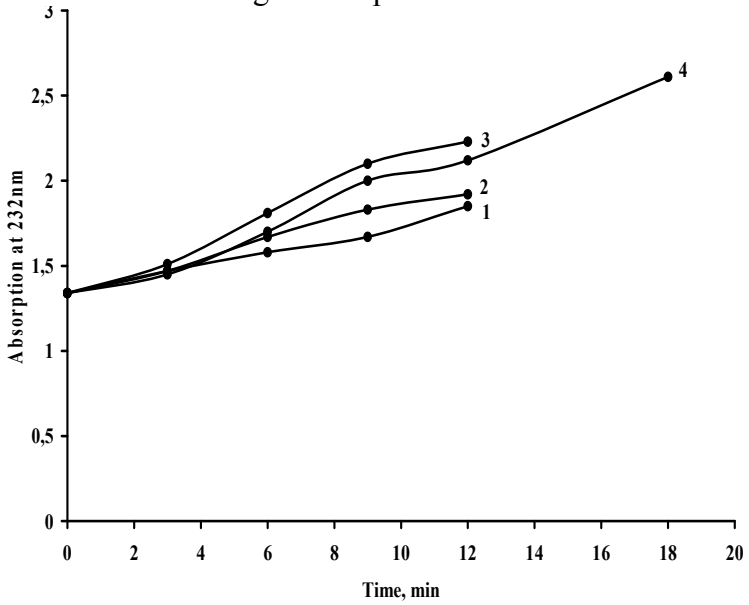

Figure 2
Change of the absorption at 232 nm

Change of the peroxide value (Fig. 1, Fig. 3) and of the absorption at 232nm (Fig. 2, Fig. 4) of sunflower oil of linoleic type during microwave and conventional heating: 1 - microwave heating at 360W, 2 - microwave heating at 600W, 3 - microwave heating at 900W, 4-conventional heating.
An indication for ongoing oxidation processes is also the increasing absorption at 232 nm, accounting for the formation of conjugated dienes. For example from initial absorption at 232 nm – 1.34 after 12 min of microwave heating, it reaches 1.92 – 2.23 (Fig. 2). The differences of these values as juxtaposed to the ones received from conventional heating are interesting. The peroxide value in conventional heating increases very abruptly from 14.9 to 42.5 meqO_2/kg during the first 6 min of heating, after which it gradually decreases (Fig.1, line 4). In conventional heating the temperature of the samples increases more rapidly compared to microwave heating and the first stage of oxidation processes – the formation of peroxides and hydroperoxides occurs with much greater speed. The changes in absorption at 232 nm are in the order of the data received from microwave heating (Fig.2, line 4).
The degree of oxidation of the oleic type is more slightly distinguishable compared to the linoleic type of oil, which is completely understandable having in mind the lower degree of unsaturation of the oleic type of oil and the higher initial degree of oxidation of the linoleic type of oil. The peroxide value from initial value of 5.8meqO_2/kg after 12min of microwave heating reaches 11.4–12.4 meqO_2/kg (Fig.3).
Despite the low content of linoleic acid in the oleic oil the increased content of conjugated dienes is also observed here – from initial absorption of 0.58 at 232nm to 0.68– 0.94 after 12 min of microwave heating (Fig.4). In conventional heating the same tendency for change in the peroxide value is noticed, which is observed in the linoleic type of oil – abrupt increase of the peroxide value up to the 6^{th} min of heating, after which it decreases (Fig.3, line 4). The absorption at 232, colour value, content of tocopherols also change, but they are in the order of the values observed in microwave heating (Table 2 and Fig. 4).

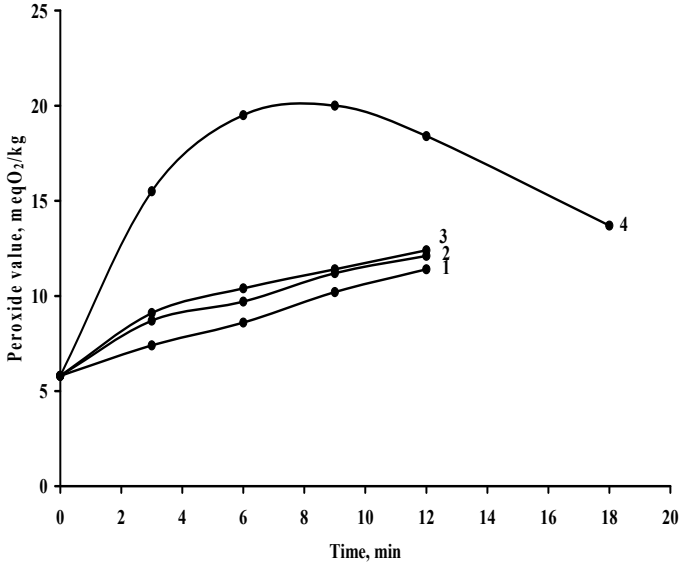

Figure 3
Change of the peroxide value

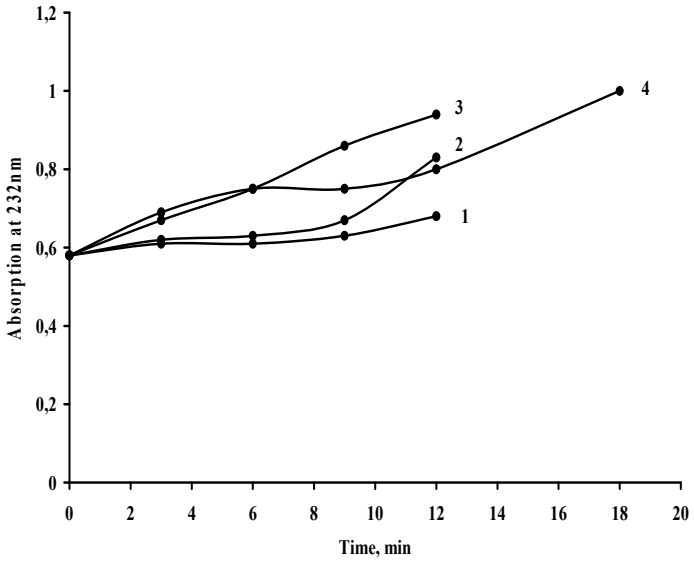

Figure 4
Change of the absorption at 232 nm

CONCLUSIONS

1. Hydrolysis processes are slightly distinguishable and the degree of change in microwave heating does not differ significantly from that in conventional heating.
2. Well distinguishable oxidation processes occur. The degree of oxidation increases with the increase of power and the duration of microwave heating.
3. The peroxide value in both types of oil after 12 min of microwave heating increases more than twice. In conventional heating it increases at a greater speed up to the 6^{th} min, after which it decreases.
4. In microwave heating under the specified conditions the quantity of conjugated dienes in the fatty oils increases about 2 times, the content of tocopherols decreases about 1.5 times and the oils get darker. The degree of change of these values in conventional heating does not differ significantly from those in microwave heating.

ACKNOWLEDGMENT

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MODIFICATION OF THE IRRADIATION EFFECT IN BARLEY PLANTS DURING THE VEGETATION PERIOD

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ABSTRACT

Vegetation pot experiments with spring barley, cultivar *Obzor* were carried out. The plants were grown under controlled conditions and in the phase of "*stem extension*" were irradiated with Cs-137 gamma rays at a dose of 5 Gy and dose intensity of 2 Gy/min.

On the day following the irradiation the plants were sprayed with synthetic growth regulators for the purpose of reducing the irradiation damage. Two types of protectors were tested: *4-Chlorophenoxyacetic acid* and *Phloroglucinol* – in concentration of 10^{-3} M. The degree of the irradiation damage and protection was recorded at the end of the vegetation period following some productivity indexes. The coefficient of protection of the applied modifiers was also determined.
It was established that in case of irradiation of spring barley with a dose of 5 Gy in the phase of stem extension, the reproductive organs were strongly damaged, which led to a considerable reduction of plant productivity.
The application of growth regulators after the irradiation improves to a different degree some of observed indexes, as a result of which the plant productivity is increased about 18% after treated with *4-Chlorophenoxyacetic acid.*

INTRODUCTION

In case of increased environmental radioactivity a necessity arises for urgent evaluation of the radiation situation and on time implementation of protective activities. This imposes availability of preliminary data for varieties depending radiosensitivity of plants as well as looking for possibilities to reduce losses from irradiation. It is determined that radioprotective abilities of plant can be increased and losses of plant production decreased with the help of chemical substances applied before or after irradiation (M. Aliev, 1983; D.M. Grodzinskij, I.N. Gudkov, 1972).
The study aims survey of possibilities for modification of radiation damage with the help of growing regulators after irradiation of barley during vegetation.

MATERIAL AND METHODS

Vegetation pot experiments with spring barley, cultivar *Obzor* were carried out. The experiments were done on Fluvisols/FAO soil in vegetative pots of 5 kg under controlled conditions. After the seeds sprout 15 normally grown plants were left in each pot. Irradiation was done in the most sensitive to radiation phase in cereal ontogenesis "*stem*

extension" at a dose of 5 Gy and dose intensity of 2 Gy/min. On the day following the irradiation the plants were treated with two synthetic growth regulators: *4-Chlorophenoxyacetic acid (P-1)* and *Phloroglucinol* (P-2) – in a concentration of 10^{-3} M aiming modification of the radiation damage. The following variants were set: control, plants irradiated with a dose of 5 Gy in the phase of stem extension, irradiated plants treated afterwards with *4-Chlorophenoxyacetic acid*, irradiated plants treated afterwards with *Phloroglucinol,* plants treated with *4-Chlorophenoxyacetic acid* и *Phloroglucinol* only.
Plants were grown to phase of full ripeness. The influence of radiation and the tested modifiers was checked on different characteristics forming plant productivity – plant survival, sterility, height of plants, total tiller, average number and weight of the grains per one plant. The protection coefficient of the applied growth regulators (K_3) was determined also (Grodzinskij, Gudkov, 1973).
The experiments were carried out in 3 repetitions.

RESULTS AND DISCUSSION

The obtained results are presented on figures 1 and 2. The data shows that acute gamma-irradiation with a dose of 5 Gy in the most radio-sensitive phase of ontogenesis strongly reduces the tested characteristics forming productivity.
The irradiation suppresses growth, thus decreasing the height of the irradiated plants. Some authors (Hillman, 1961) associate the suppressing of vegetation with the suppressing of aucsines synthesis and other physiologically active growth substances but according to others (Medvedev, 1970) the reason is accumulation of abnormal metabolites and inhibitors of growth. Stimulation of tiller for the irradiated variants compared to the control is observed. The survival index is not influenced by the irradiation because of the late phase in which irradiation was done and fading of growth processes. 100% outlasted plants are reported 23, 81% of which sterile. The number and weight of grains obtained average per plant decrease with 42.52% and 34.03% respectively compared to the unirradiated control. The productivity of the irradiated plants strongly decreases – with 43,74% compared to the control.
The considerable reduction of barley productivity due to radiation is an evidence for strong suppression of the meiotic processes and injury of the

generative organs during influence at this phase of ontogenesis. The results obtained correspond to the research of Grodzinski D.M. 1989, that in plant ontogenesis the stages of morphogenesis associated with the formation of generative organs characterize with lowest radioresistence.

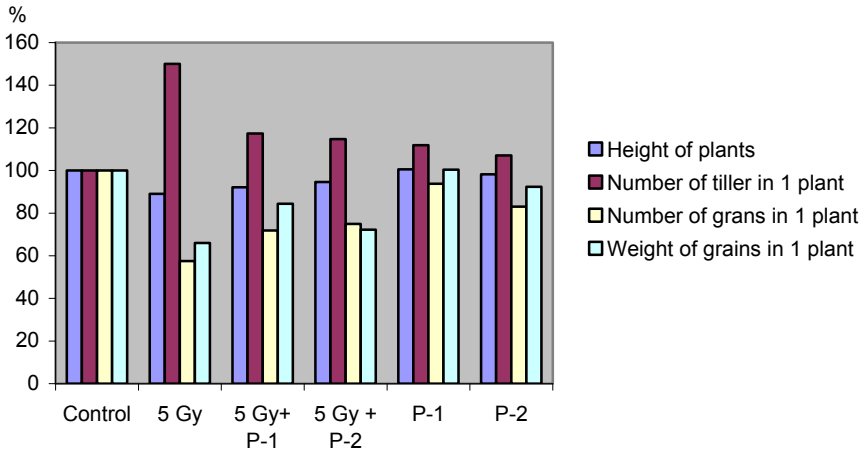

Figure 1
Effect of growth regulators on productivity of spring barley irradiated with a dose of 5 Gy in phase of "*stem extention*"

The results presented at fig. 1 and fig.2 show, that the application of *Phloroglucinol* have insignificant influence on the indices associated with productivity. The growth regulator studied does not stimulate the tiller of the irradiated plants. The percentage of sterile plants decreases insignificantly – with about 2% for those treated with the radiomodificator. The average number of grains per plant increases with 17.45%, while the weight of the grains obtained increases with just about 6,25%. This impacts also on the productivity of the plants restored which increases with only 6% compared to the irradiated and untreated variants.

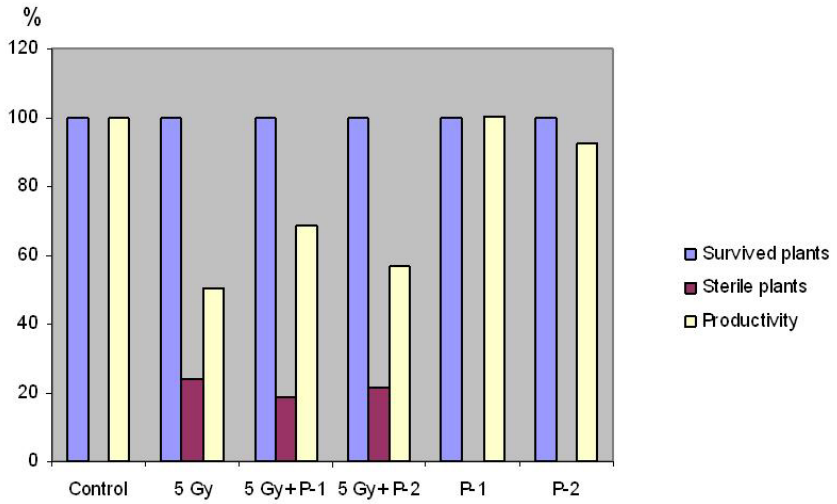

Figure 2
Effect of growth regulators on productivity of spring barley irradiated with a dose of 5 Gy in phase of "*stem extention*"
(K_{p-1}=1,36; K_{p-2}=1,12)
(P_1- *4-Chlorophenoxyacetic acid;* P_2 – *Phloroglucinol)*

The treatment of plants with *4-Chlorophenoxyacetic acid* after irradiation insignificantly affects the height of the irradiated variants. The tiller is not stimulated and the average number of the grains obtained increases with 14,4%. The average weight of grains per plant increases with 18,4% compared to the variant without treatment. The sterility reduces with about 5%. As a result the plant productivity increases with about 18%. A relatively high protection coefficient – 1,36 is recorded in this variant as well.

The results obtained by us confirm the statement that the modifying effect of different substances causing decrease of damaging effect of radiation is strongest during the period of reduced radioresistance of plants, when the potential abilities for protection and recovery are to the greatest extend manifested / Grodzinskij, D.M 1973; Aliev I.M.1983/.

The following conclusions can be done regarding the results of our research on radiosensitivity of spring barley cultivar *Obzor* and growth regulators studied for reduction of irradiation damage:

1 Irradiation of spring barley with a dose of 5 Gy in the phase of "*stem extension*" strongly damages the reproductive organs, which causes considerable reduction of plant productivity.
2. The treatment after irradiation with the growth regulators studied improves to a different extent of some yield indices like sterility, number and weight of the grains obtained. Therefore the productivity increases with 18% after treatment with *4-Chlorophenoxyacetic acid.*
Application of *4-Chlorophenoxyacetic acid* considerably influences the productivity of the irradiated plants and therefore its use in the practice for irradiated sowing can be recommended.

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SOME THERMOPHYSICAL CHARACTERISTICS OF MILK AND MILK PRODUCTS

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This article deals with thermophysical characteristics of milk and milk products. If we want to protect quality of food we need to know its physical properties. One of the most important are thermophysical parameters as temperature, thermal conductivity and thermal diffusivity.

For thermophysical parameters measurements was used PS method and also Hot Wire method. In the first series of measurements we measured relations between thermal conductivity and thermal diffusivity in temperature range (5–25) C for milk. In the second series of measurement was measured relation between thermal conductivity and relative fat content for milk. There were also measured some thermophysical parameters of cheese, processed cheese and acidophilus milk. The results of measurements for milk samples showed that temperature stabilisation process and relative fat content have influence to variation of thermophysical parameters. All measured relations for milk samples during temperature stabilisation have linear increasing progress – fig. 2-3. Figure 4 shows that increasing relative fat content has decreasing influence on milk thermal conductivity. Results for other milk products are summarised in table 1.

INTRODUCTION

The quality assessment and guarantee of the safety of foodstuff belong to the main priorities in food industry. HACCP – Hazard Analysis and Critical Control Point System represents scientifically sophisticated approach to the problems of the protection of foodstuff quality. It is internationally accepted system guarantying the foodstuff safety aimed to the identification, evaluation and risks control in the whole technological procedure of foodstuff production. Its successful application is subject to the complete knowledge of physical properties of foodstuff (Jiřičková – Pavlík - Černý, 2006). One of most important are thermophysical parameters of food which are usually changed during the processing, storage and manipulation. In national standards are usually used for thermophysical parameters measurements transient methods. Transient methods represent a large group of techniques where measuring probes, i.e. the heat source and the thermometer, are placed inside the specimen. The temperature of the specimen is stabilized and made uniform. Then the dynamic heat flow in the form of a pulse or step - wise function is generated inside the specimen. From the temperature response to this small disturbance, the thermophysical parameters of the specimen can be calculated.

MATERIALS AND METHODS

Measured samples of milk and milk products were provided in storage boxes at the temperature from 4°C to 5°C and 90% of the air moisture content during 24 hours before measurement and relations of thermophysical parameters to the temperature for milk were measured during temperature stabilization of samples. Measurements of thermal conductivity and thermal diffusivity for cheese, processed cheese and acidophilus milk were realised in room temperature, and presented values are averages from one hundred measurements for every thermophysical parameter. All measurements were made in laboratory settings. The measurement was realized for milk with relative fat content 0.5 %, 1.5% and 3.5% in temperature range (5– 25) ºC. Methods of measurements were selected according to structural characteristics of the sample. For grated liquids materials is convenient Hot wire method. For samples with compact and suspenzoid structure are convenient PS method, which are described in follows text.

Hot wire method (HW) - The simple measurement consists in measuring the temperature rise *vs*. time evaluation of an electically heated wire embedded in the tested material. The thermal conductivity is derived from the resulting change in temperature over a known time interval. The ideal analytical model assumes an ideal – infinitely thin and infinitely long line heat source (hot wire), operating in an infinite, homogenous and isotropic material with uniform initial temperature T_0. If the hot wire is heated for the time $t = 0$ with constant heat flux q per unit wire length, the radial heat flow around the wire will occur. The temperature rise $\Delta T(r,t)$ in any distance r from the wire as a function of time is described by the simplified equation (1) (Carslaw H. S. - J. C. Jeager, 1999).

$$\Delta(r,t) = \frac{q}{4\pi\lambda} \ln \frac{4at}{r^2 C} \tag{1}$$

Where:λ – the thermal conductivity, a – thermal diffusivity, $C = \exp(\gamma)$ with γ the Eulers´constant. The thermal conductivity is calculated from the slope S of the temperature rise $\Delta T(r,t)$ *vs*. the natural logarithm of the time $\ln t$ evolution using the formula

$$\lambda = \frac{q}{4\pi S} \tag{2}$$

Several corrections have been introduced to account for the heat capacity of the wire, the thermal contact resistance between the wire and the test material, the finite dimension of the sample and the finite dimension of the wire embedded in the sample. (Liang, 1995, Assael- Wakeham, 1992)

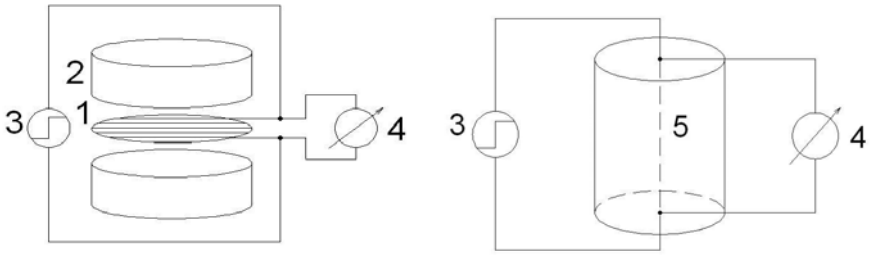

Figure 1
Plane source method Hot wire method
1 – PS sensor, 2 – samples, 3 – current source, 4 – milivoltmeter,
5 – heat source and thermometer

Plane source method (PS) - is based on using an ideal plane sensor – PS. The PS sensor acts both as heat source and temperature detector. The plane source method is arranged for a one dimensional heat flow into a finite sample.

The theory considers ideal experimental conditions – ideal heater (negligible thickness and mass), perfect thermal contact between PS sensor and the sample, zero thermal resistance between the sample and the material surrounding sample, zero heat losses from the lateral surfaces of the sample. (Krarawacki, Suleiman , Hang , Nhi, 1992)

If q is the total output of power per unit area dissipated by the heater, then the temperature increase as function of time is given by (Beck, Arnold, 2003).

$$\Delta T(x,t) = 2\frac{q\sqrt{at}}{\lambda} ierf\left(\frac{x}{2\sqrt{at}}\right) \qquad (3)$$

Where a is thermal diffusivity, λ is thermal conductivity of the sample and *ierfc* is the error function [3].

We consider the PS sensor, which is placed between two identical samples having the same cross section as the sensor in the plane $x = 0$. The temperature increase in the sample as a function of time conforms.

$$T(0,t) = \frac{q\sqrt{a}}{\lambda\sqrt{\pi}}\sqrt{t} \qquad (4)$$

Which corresponds to the linear heat flow into an infinite medium (Karawacki, Suleiman, 2001). The sensor is made of a Ni-foil, 23 μm thick protected from both sides by an insulating layer made of kapton of 25 μm thick made on Institute of Physics, SAS.

RESULTS AND DISCUSSION

In the 1st series of measurements were obtained values of thermal conductivity (Fig. 2) and thermal diffusivity (Fig. 3) during the temperature stabilization of milk with different relative fat content 0.5%, 1.5% a 3.5%.

Table 1
Results of thermal conductivity and thermal diffusivity measurements for selected milk products

Sample	Thermal Diffusivity [m^2/s]	Thermal Conductivity [W/m .K]
Acidophilus milk	18.5 x E-8	0.51
Processed cheese	16.5 x E-8	0.71
Cheese	15.1 x E-8	0.63

For thermophysical parameters measurement was used temperature range (5 - 25) °C. In the 2nd series was measured relation between thermal conductivity and relative fat content (Fig. 4). Results of thermal conductivity and thermal diffusivity measurements for milk products are showed in table 1.

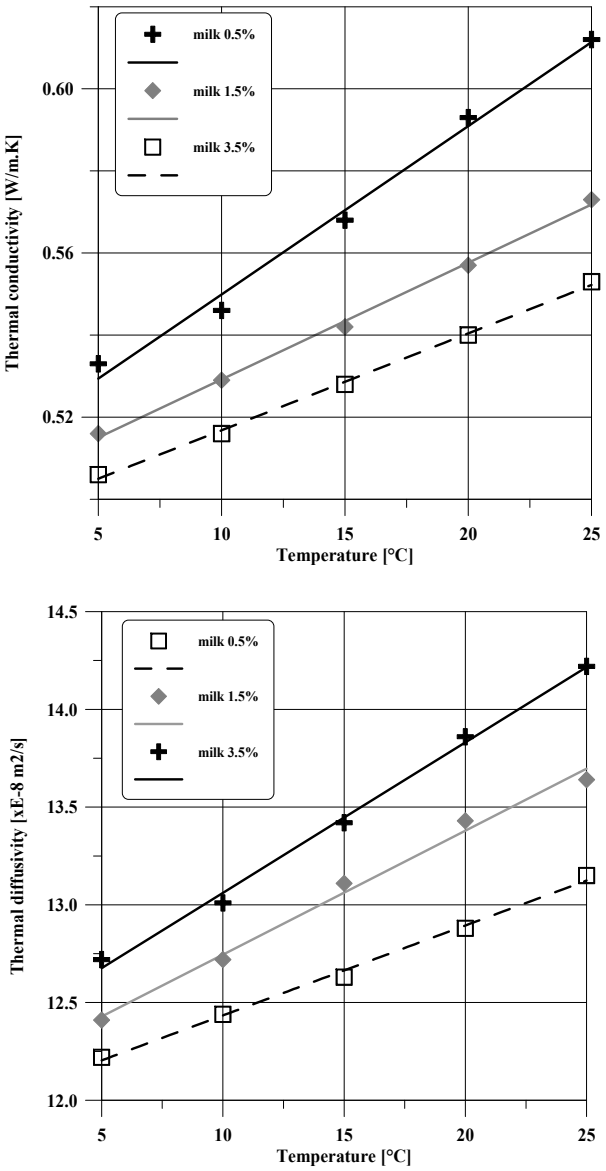

Figure 2-3
Relations of thermal conductivity and thermal diffusivity
to temperature for milk with different relative fat content

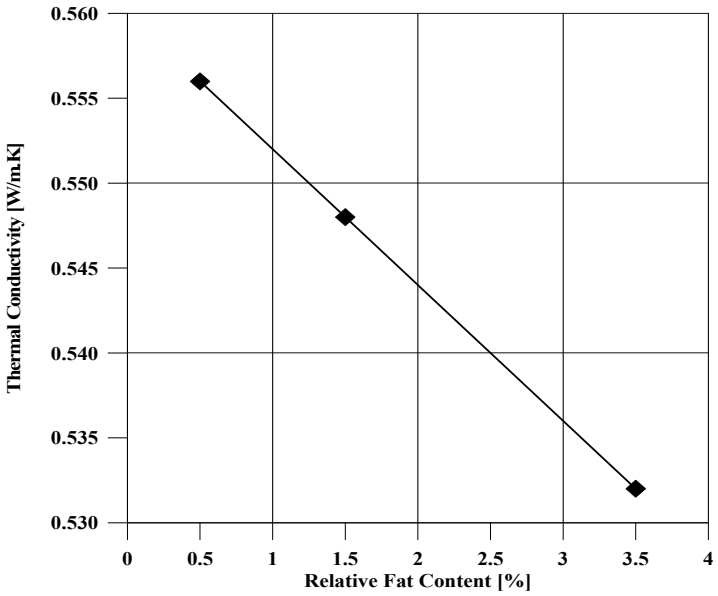

Figure 4
Relations of milk thermal conductivity

Conclusion

Study of relationships between thermal conductivity, thermal diffusivity and temperature which results are showed on figures 2 – 3. Figure 4 – show, that increasing relative fat content has decreasing influence on milk thermal conductivity. It shows, that the thermal conductivity of low viscosity liquids can be measured with Hot wire method and the three last concentrations indicate a perfectly linear dependence of the thermal conductivity on the fat concentration. There were also measured some thermophysical parameters of cheese, processed cheese and acidophilus milk. For samples with compact and suspenzoid structure were used PS method. The results of measurements which are presented in table 1 show that different structure of material and different way of processing have significant influence on thermal conductivity and thermal diffusivity of milk products. For data reliability protection there were realized series of measurements for every point in graphics characteristics and presented table values with number of hundred measurements and results were

obtained as valued averages. Based on presented results, it is necessary to have knowledge of the dependence of thermophysical parameters on temperature, structure and relative fat content of milk and milk products if we need to protect quality during the processing and storage.

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INSTRUMENTAL MEASURING OF COLOUR AS A MARKER OF ORIGIN OF SOME VARIETIES OF POTATOES

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ABSTRACT

In this work are described results of first year of research of spectrofotometric measuring of colour. We observed an improvement of instrumental measuring of colour as a marker of origin of some varieties of potatoes.
We tested ten varieties of potatoes. The potatoes were tested raw and boiled. Three raw potato tubers were parted in two pieces in transverse and lengthwise direction. Every half was measured in three repetition. In the same way were prepared other three raw and three boiled potatoes which were measured over the PE sheeting.
The optical system is using diffused illustration and reflected light is measured under coal 8°(d/8) with SCE function (specular component excluded) for elimination of mirror radiance. Slot diameter is 30 nm.
All spectrum between 380-780 nm was measured. The colour is defined in L*a*b* colour system (CIELab).
The first record shows that it is possible to diagnose the varieties of potatoes only by spectrophotometical measuring of colour. Just now we are trying to confirm this in other tests in In-house grant of Mendel University.

INTRODUCTION

There is lot of causes why to check quality of food. In cause of potatoes is point to check mainly its varieties. Quickness, simplicity, low costs and scarcely any other special requirements to serve person – this is a few basic conditions to implant new method to practice.

Our Method - spektrofotomectric measuring of colour fulfil all this condition. The difference between spectrophotometric and colourimetric measuring of colour is that the spectrophotometric measuring defined the colour of the object like the human eye seems it. Spectrophotometric measuring does it too, but in addition it measures all spectrum between 380-780 nm and the colour is defined in L*a*b* colour system (CIELab). So we can find unique part of spectrum if it exists and describe for example origin of some varieties of potatoes.

MATERIALS AND METOD

Ten varieties of potatoes were tested (Aneta, Rosara, Korela, Lolita, Jitka, Ditta, Red Anne, Katka, Karin a Keřkovské rohlíčky).
Preparation of samples:
The potatoes were tested raw and boiled. Three raw potato tubers were parted in two pieces in transverse and lengthwise direction. Every half was measured in three repetition. In the same way were prepared other three raw and three boiled potatoes which were measured over the PE sheeting.
Boiled samples were boiled 15 minutes.

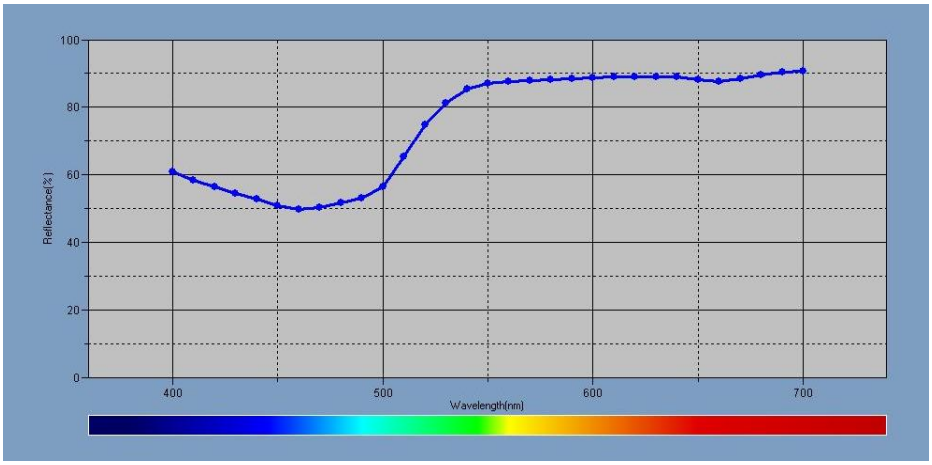

Figure 1
The curve of spectrum between 380 -780 nm

The optical system is using diffused illustration and reflected light is measured under coal 8 (d/8) with SCE function (specular component excluded) for elimination of mirror radiance.
Slot diameter is 30 mm.
All spectrum between 380-780 nm was measured. The curve of measured spectrum of one sample is on Fig. 1.

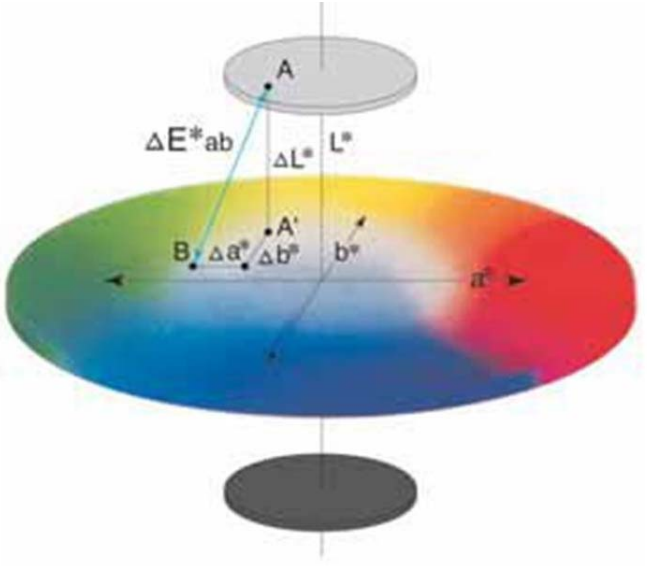

Figure 2
L*a*b* colour system

The colour is defined in L*a*b* colour system (CIELab). (Fig. 2) In this system value L* is luminance and values a* and b* are axis of colour. + a* is red direction, -a* is green direction, + b* is yellow direction and -b* is blue direction.

RESULTS AND DISCUSSION

It was used ten varieties of potatoes. From every variety was choose 12 samples. Six Samples were boiled, other six we used raw. Three samples from each groups we cut in cross direction and other three in longitudinal. Each half of sample was measured three times. So we got 72 measurement from each variety of potatoes.

For statistic characterising we use Tukey-B test. It show (Fig. 3) that origin of variety of potatoes can be defined for P<0,05 on base of measurement of a* dates (red colour area).

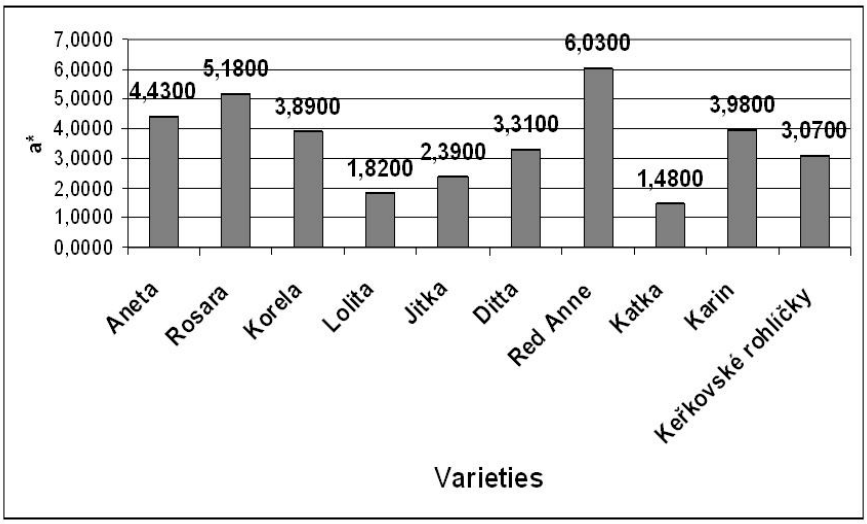

Figure 3
Mean of a* data in measurement of potatoes

This dates can not be use like conclusive. We have to confirm our first results in few harvest and take into account other factors like different growing plans, area, weather, stocking etc.

ACKNOWLEDGEMENT

The results presented in that paper are the summary of activities developed at Mendel University of Agriculture and Forestry in Brno with the support of In-house Grant Agency at MUAF in Brno .

ACOUSTICS METHOD OF THE CHEESE RIPENING EVALUATION

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ABSTRACT

The paper is focused on the study of the feasibility of using the acoustic impulse-response technique to evaluate Edam cheese texture and maturity. Cheese blocks were hit with an impact bar and the acoustic response at the eight points on the cheese surface was recorded. The response signals (impact force, surface displacement and surface velocities) were detected by the laser-vibrometers. In connection with the results of the classical compression tests performed on the tested cheeses the constitutive equation of the cheese behaviour has been designed. This equation has been used in the numerical simulation of the cheese block response. This simulation has been performed using of the LS DYNA 3D finite element code. The numerical results show reasonable agreement with experimental ones.

INTRODUCTION

The texture of the cheese are traditionally determined by destructive sensory and instrumental measurements. Texture Profile Analysis (TPA), uniaxial compression and puncture tests have been widely used to assess cheese texture, providing information on both the deformation and fracture properties of food products. At present, new non-destructive techniques such as small displacement probes, vibrating rheometers, near infrared spectroscopy (NIR), computer vision, biosensors, ultrasonic analysis and sonic measurements are more and more used. One of these non destructive methods is the acoustic impulse–response technique where the food is excited by being struck with a probe and the response functions are obtained. This technique was used to detect surface cracks in eggshells or voids in watermelons (Cho,2000). Furthermore, this

methodology has been applied on fruits such as peaches, apples and pears, in order to quantify changes in firmness during ripening.

These ultrasonic techniques were also used to assess the degree of Manchego cheese maturity (Benedito et al., 2006). The texture of some other cheeses like Mahon or Cheddar cheese has been also studied. The acoustic impulse –response technique used for the study of the cheeses has used response function in form of sound waves detected by the microphones. In the given paper the modified technique has been used where the response of the cheese is described by the time histories of the surface displacement and surface velocity.

MATERIALS AND METHODS

The experiments were carried out on blocks of Certified Origin EDAM cheese (Content of the fat 30%), manufactured by a company located in South Moravia. The pieces were matured in chambers where relative humidity and temperature were maintained according to the company procedures. The blocks of cheese have been tested at 16,39,60,79 and 107 days after the production.

Two types of the experiments have been realized. First of all the simple compression and stress relaxation tests have been performed using of the TIRA testing device. The crosshead velocities have been chosen to be 1,10,100 and 400 mm/min. Results of these tests have been used to obtain the constitutive parameters of the non-linear viscoelastic materials like cheeses etc. The details of this procedure are described in (Goh et al.,2004).

The impact tests were carried out using an impact device specially designed and built for cheese measurements. The impact set-up consisted of a free-falling cylindrical bar(6 mm in diameter, 200 mm in height – made from aluminium alloy). The bar is instrumented by strain gauges . This instrumentation enables to record the time history of the force at the interface between cheese and bar. At the distances 30, 45, 60,75, 90, 105, 120 and 135 mm from the point of the bar impact the surface displacement as well as the surface velocity are measured using of the laser-vibrometer.

The cheese response was picked up through an amplifier and a commercial A/D PC board to the PC, which simultaneously served as the data acquisition system. An optical sensor was used to trigger the

acquisition. The signal was sampled at a rate of 200,000 samples/s for a period of 15 ms. Instead of the time dependent response functions, the MATLAB computer program transformed the response from time to frequency domain by means of Fast Fourier Transform (FFT). The impact velocity of the bar has been kept to be constant (1.2 m/s).

RESULTS AND DISCUSSION

In the Fig.1 the experimental records of the surface displacement - time are displayed. This displacement is connected with the propagation of the surface wave from the point of the impact. The results show that the surface wave exhibits the significant attenuation in the direction of the stress wave propagation. This attenuation also increases with the time of the cheese ripening – see Fig.1. This phenomenon indicates that the behaviour of the cheese is more or less viscoelastic. The damping of the stress wave in the direction of its propagation can be also described in the frequency domain .If we substitute the displacement p(t) by its Fourier transform P(ω), where ω is the angular frequency we can define the transfer function T(ω) :

$$T(\omega) = \frac{dP(\omega)}{dx}$$

Where x is the distance in the direction of the wave propagation

The example of the transfer function amplitude is displayed in the Fig.2. The analysis of the experimental data found that this dependence was typical for the different stages of the cheese ripening. One can see that the description of the wave attenuation in the frequency domain is more simple than that in the time domain.

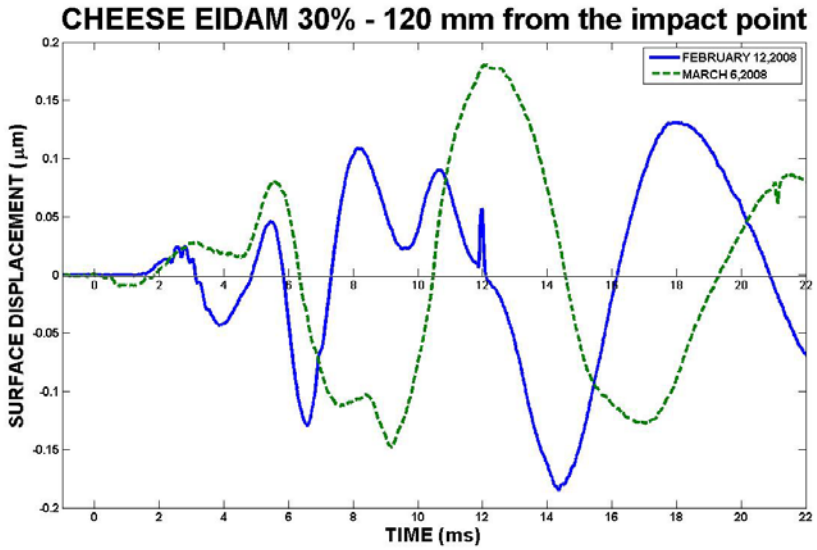

Figure 1.
Surface displaement vs.
Time at the point x=90 mm from the point of the bar impact.

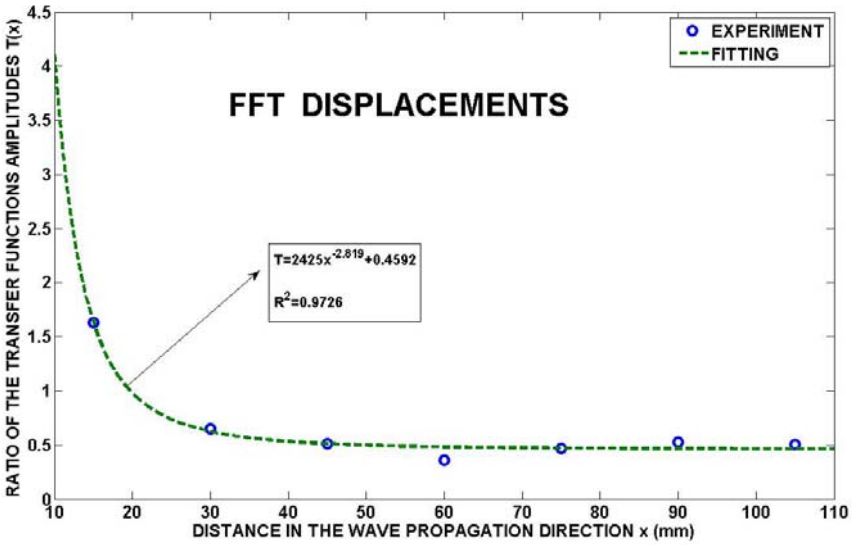

Figure 2
Transfer function amplitude .Cheese tested on March 6, 2008.

In the next step the displacement at the different points has been evaluated using of the constitutive equation obtained from the simple compression and relaxation tests. According to the procedure outlined in (Goh et al.,2004) the parameters of the non-linear viscoelastic model have been evaluated. The detail description of this work will be subject of the forthcoming papers. The obtained model of the cheese mechanical behaviour has been used for the numerical simulation of the experiments. In the Fig.3 the comparison of the experimental and numerical results is shown. One can see that there is very reasonable agreement between experiment and numerical simulation.

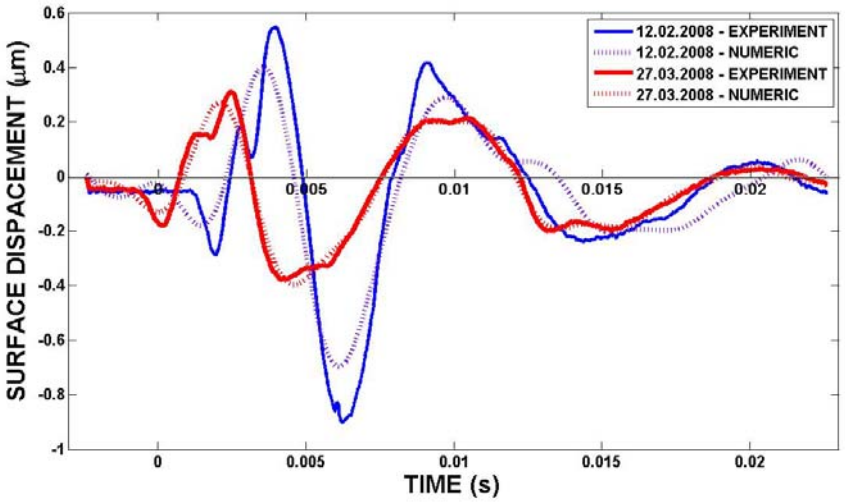

Figure 3
Comparison between experiment and numerical results - an example.

Generally one can see that the proposed method of the cheese properties evaluation seems be a promising tool for the next research. There is only one critical limitation of this procedure which follows from the role of the stress wave propagation. The origin of some voids or some holes in the cheese can effects the wave propagation much more significantly than some changes of the cheese texture, e.g. during its ripening. The presence of the holes in the cheese on the stress wave propagation should be a subject of some next research.

ACKNOWLEDGEMENTS

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DETERMINATION OF SOME PHYSICAL AND SENSORY PROPERTIES OF MILK, DARK AND WHITE CHOCOLATES ENRICHMENT WITH SUNFLOWER SEED, FLAX SEED, OAT AND DRIED DAMSON PLUM

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ABSTRACT

Chocolate and cocoa products are preferred by a large community and as well as being a pleasure to eat. In the last decade, the studies about all sort of chocolate enrichment with functional foods during the shelf life are very limited. And in the examined works, there are no declarations about chocolate enrichment with sunflower seed, flax seed, oat and dried damson plum.The aim of this project is enrichment the milk, dark and white chocolates with sunflower seed, flax seed, oat and dried

damsonplum, and determine some physical, and sensory properties of enrichment milk, dark and white chocolate.

INTRODUCTION

Cocoa and chocolate products have been delicacies for hundreds of years and are preferred by a large community. Chocolate is one of the most popular foods all over the world and is a highly nutritious energy source, with a fast metabolism and good digestibility. Nowadays, one of the most important trends in food manufacturing is originated by the consumers' demand of functional or health-promoting foods, i.e., foods that not only cause no harm, but also remedy or prevent illnesses such as heart disease, osteoporosis, cancer, diabetes, etc. In the last decade, the studies about all sort of chocolate enrichment with functional foods are very limited. And in the examined works, there are no declarations about chocolate enrichment with sunflower seed, flax seed, oat and dried damson plum.

In this study, it is aimed to enrichment the milk, dark and white chocolates with sunflower seed, flax seed, oat and dried damson plum, and determine some physical and sensory properties of enrichment milk, dark and white chocolate. For this reason, milk, dark and white chocolates enriched with sunflower seed, flax seed, oat/rice and dried damson plum which are in different amount and different particul size. After the prelimanirary experiments the amount and particle size were optimised.

MATERIALS AND METHODS

Materials

The industrial production of enrichment dark, white and milk chocolate samples were done in Nestle Turkey Food Industry, Karacabey, Bursa, Turkey.

The amount and particle size was optimised by prelimanirary experiments. Different amount and different particle sized sunflower seed, flax seed, oat and dried damson plum were added to dark, white and milk chocolate. The trained panellists were evaluated chocolate samples i.e. colour, texture, flavour and overall acceptability by using a 7-point hedonic scale (1 for dislike extremely, 4 for moderate, 7 for like extremely) (Larmond, 1997). The result of sensory attributes showed that

15% and 1400-1600µm flaxseed, 20% and ½ broken sunflower seed, 15% minced dried damson plum, 10% and 3 mm extruded 30: 70% oat: rice had been mostly preferred samples.

Methods

Determination of Melting point

The melting point of chocolate samples was determined by using Büchi 510 Melting Point Determinator (Büchi Lab. Teck. AG, Flawil, Swiss). All measurements were done at 20°C.

Determination of texture

Hardness or degree of softening was measured by determining the maximum penetration
force of chocolate samples. For measuring the depth of penetration a Texture Analyzer Model

TA-XT plus was used. The measurement parameters were; penetration depth 6 mm; probe needle PN, pre-speed 1.0 $mm \cdot s^{-1}$, test speed = 1.1$mm \cdot s^{-1}$, post speed = 10.0 $mm \cdot s^{-1}$ and all measurements were done at 20°C (Ali et al., 2001).

Determination of colour

The colour was measured by determining Hunter L (lightness), a (redness/greenness) and b (yellowness/blueness) values of chocolate samples. For measuring a Minolta Chroma-Meter CR-310 reflectance colorimeter was used and all measurements were done at 20°C.

Sensory evaluation

Sensory attributes of chocolate samples i.e. appearance, texture, flavour and overall acceptability were evaluated by using a scoring scale which was constituted for these new products by trained panellists.

Scoring Scale for enrichment chocolate samples

Appearance

1. Own typical chocolate colour; smooth and bright surface; homogeneous and smooth chocolate structure; homogeneous disperse of enrichment components

2. Own typical chocolate colour; a light decrease smooth and bright surface; homogeneous and smooth chocolate structure; homogeneous disperse of enrichment components

3. Own typical chocolate colour; a light decrease of smooth and bright surface; a light decrease of homogeneous and smooth chocolate structure; not homogeneous disperse of enrichment components
4. A little fat or sugar bloom; rough and/or mat surface; not homogeneous and smooth chocolate structure; not homogeneous disperse of enrichment components
5. Unacceptable fat or sugar bloom; excessive rough and/or mat surface; not homogeneous and smooth chocolate structure; not homogeneous disperse of enrichment components

Texture
1. Ideal hardness and crispness; melting in mouth and tender structure; and affiliated to sort of enrichment components:
Sunflower seed: Ideal hardness, light gum mastic taste, easy chewing
Flax seed: Ideal hardness, sticky to teeth
Oat: Ideal hardness and crispness
Dried damson plum:Light sticky, easy chewing
2. Decrease/increase of hardness and crispness; a light softness/sticky structure; and affiliated to sort of enrichment components:
Sunflower seed: Decrease/increase of hardness and decrease/increase of gum mastic taste
Flax seed: Decrease/increase of hardness, sticky to teeth
Oat: Decrease/increase of hardness and crispness
Dried damson plum: Decrease/increase of sticky, light softness/hardness
Excessive hard/soft; fragile; easy dispersed; excessive loose in mouth; sticky and oily/dry structure; and affiliated to sort of enrichment components.
Sunflower seed: Excessive decrease/increase of hardness and excessive decrease/increase of gum mastic taste, hard chewing
Flax seed: Excessive decrease/increase of hardness, excessive sticky to teeth, hard chewing
Oat: Excess decrease/increase of hardness and crispness
Dried damson plum: Excessive decrease/increase of sticky, excessive softness/hardness

Flavour
1. Own typical chocolate flavour and sweetness; typical enrichment components flavour

2. A light decrease of typical chocolate flavour and sweetness; typical enrichment components
flavour
3. A light decrease of typical chocolate flavour and sweetness; a light tasteless of enrichment
components flavour
4. An evident strange flavour; tasteless or oxidized of enrichment components flavour
5. Unacceptable strange flavour; spoil taste; excessive tasteless or oxidized of enrichment components flavour

Table 1. Melting point and texture properties of enrichment chocolate samples[1]

Enrichment chocolate samples	**Melting point (^{0}C)**	**Hardness (kg force)**
White chocolate	29.13±*0.12*^{a}	*1.91±0.09*^{b}
White chocolate with flaxseed	22.67±*0.42*^{d}	2.00±*0.09*^{a}
White chocolate with sunflower seed	21.93±*0.21*^{e}	1.75±*0.03*^{c}
White chocolate with oat and rice	27.07±*0.06*^{c}	1.88±*0.01*^{b}
White chocolate with damson plum	27.20±*0.17*^{b}	1.63±*0.01*^{d}
Milk chocolate	29.97±*0.06*^{a}	*2.90±0.05*^{a}
Milk chocolate with flaxseed	27.03±*0.06*^{d}	2.39±*0.07*^{c}
Milk chocolate with sunflower seed	26.00±*0.10*^{e}	2.39±*0.00*^{c}
Milk chocolate with oat and rice	28.27±*0.06*^{c}	2.68±*0.00*^{b}
Milk chocolate with damson plum	*29.10±0.10*^{b}	2.41±*0.13*^{c}
Dark chocolate	30.57±*0.06*^{a}	3.51±*0.01*^{a}
Dark chocolate with flaxseed	27.03±*0.06*^{d}	3.25±*0.03*^{c}
Dark chocolate with sunflower seed	26.77±*0.25*^{e}	2.79±*0.05*^{e}
Dark chocolate with oat and rice	29.57±*0.06*^{c}	3.45±*0.01*^{b}
Dark chocolate with damson plum	29.93±*0.06*^{b}	2.88±*0.18*^{d}

[1] Measurements were done at 20°C, mean of five determinations ± SD, means followed by the same letter within each coloumn are significantly different at $p<0.05$ level (Duncan's multiple range test)

Overall acceptability
5. Like extremely
4. Like
3. Moderate
2. Dislike
1. Dislike extremely

Statistical analysis

The results were expressed as mean values ± SD. The statistical analyses for determining analysis of variance and degree of significance were carried out using the SPSS 15.00.

Table 2. *L*, a*, b** values of enrichment chocolate samples [1]

Enrichment chocolate samples	*L**	*a**	*b**
White chocolate	81.09±*0.05*^{a}	-3.86±*0.02*^{c}	26.07±*0.21*^{a}
White chocolate with flaxseed	71.75±*0.13*^{e}	-3.70±*0.00*^{b}	16.65±*0.01*^{e}
White chocolate with sunflower seed	77.46±*0.07*^{b}	-4.87±*0.02*^{d}	21.49±*0.05*^{c}
White chocolate with oat and rice	73.38±*0.20*^{d}	-0.83±*0.27*^{a}	22.25±*0.08*^{b}
White chocolate with damson plum	75.96±*0.92*^{c}	-5.00±*0.16*^{e}	20.99±*1.23*^{d}
Milk chocolate	35.22±*0.05*^{a}	6.44±*0.01*^{c}	9.06±*0.05*^{c}
Milk chocolate with flaxseed	34.71±*0.35*^{b}	6.78±*0.19*^{b}	9.30±*0.18*^{b}
Milk chocolate with sunflower seed	34.97±*0.60*^{c}	6.72±*0.06*^{b}	9.19±*0.12*^{b}
Milk chocolate with oat and rice	34.70±*0.04*^{b}	8.65±*0.08*^{a}	11.72±*0.00*^{c}
Milk chocolate with damson plum	34.34±*0.42*^{d}	6.34±*0.17*^{d}	8.77±*0.06*^{d}
Dark chocolate	29.71±*0.08*^{a}	2.73±*0.08*^{e}	3.98±*0.14*^{c}
Dark chocolate with flaxseed	29.74±*0.23*^{a}	3.32±*0.11*^{b}	4.41±*0.15*^{b}
Dark chocolate with sunflower seed	29.36±*0.25*^{b}	3.06±*0.10*^{c}	4.04±*0.03*^{c}
Dark chocolate with oat and rice	27.72±*0.02*^{c}	4.94±*0.03*^{a}	5.33±*0.02*^{a}
Dark chocolate with damson plum	29.24±*0.02*^{d}	2.85±*0.20*^{d}	4.03±*0.14*^{c}

[1] Measurements were done at 20°C, mean of five determinations ± SD, means followed by the same letter within each coloumn are significantly different at $p<0.05$ level (Duncan's multiple range test)

RESULTS AND DISCUSSION

The results of the melting point and hardness of enrichment chocolate samples are presented in Table 1. The melting point of samples was arranged in order white chocolate< milk chocolate < dark chocolate. The enrichment of chocolate samples with sunflower seed, flax seed, oat and dried damson plum was affected hardness significantly ($p<0.05$).

Table 3. Sensory attributes of enrichment chocolate samples[1]

Enrichment chocolate samples	**Appearance**	**Texture**	**Flavour**	**Overall acceptability**
White chocolate with flaxseed	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.50±*0.53*^{c}
White chocolate with sunflower seed	4.75±*0.46*^{c}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.88±*0.35*^{a}
White chocolate with oat and rice	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.88±*0.35*^{a}
White chocolate with damson plum	4.88±*0.35*^{b}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.75±*0.46*^{b}
Milk chocolate with flaxseed	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.38±*0.74*^{c}
Milk chocolate with sunflower seed	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.75±*0.46*^{b}
Milk chocolate with oat and rice	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	5.00±*0.00*^{a}
Milk chocolate with damson plum	4.89±*0.33*^{b}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	5.00±*0.00*^{a}
Dark chocolate with flaxseed	5.00±*0.00*^{a}	3.00±*0.00*^{a}	4.86±*0.38*^{b}	4.43±*0.53*^{c}
Dark chocolate with sunflower seed	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	4.86±*0.38*^{b}
Dark chocolate with oat and rice	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	5.00±*0.00*^{a}
Dark chocolate with damson plum	5.00±*0.00*^{a}	3.00±*0.00*^{a}	5.00±*0.00*^{a}	5.00±*0.00*^{a}

[1]Means followed by the same letter within each coloumn are significantly different at $p<0.05$ level (Duncan's multiple range test)

The results of colour and sensory evaluation of chocolate samples are presented in Tables 2 and 3. As a matter fact there was a significant difference between L^*, a^* and b^* values of samples ($p<0.05$). The sensory attributes of enriched chocolate samples were mostly taken whole point. This indicates that enrichment of chocolate samples with sunflower seed, flax seed, oat and dried damson plum were possible.

CONCLUSION

New chocolate products were improved by the result of this study and for sensory evaluation a scoring scale was constituted for new chocolate product.

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USE PHYSICAL AND CHEMICAL PARAMETERS FOR THE CHARACTERISTIC OF HONEY QUALITY

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ABSTRACT

For the characteristic of honey quality it is possible to use different physical and chemical parameters. For example, activity of enzymes, the content of sugars, etc. The content of carbohydrates in honey is very diverse. It depends on kinds of honey and many other things factors. The content of sugars in honey allows define quality of honey and also characterizes specific properties of honey. Activity of enzyme invertase also as activity of enzyme amylases depends from heating and storage of honey. Therefore invertase can serve as a parameter of quality of honey.

Amino acid proline is one of the main amino acids of honey. Natural honey should contain this acid. Therefore definition the content of proline in honey can serve as a parameter of quality of honey. Each carbohydrate has a specific angle of rotation of polarized light (specific rotation). It depends on relations and content of carbohydrates in honey. The aim of the present research was to establish the relationship between honey sorts and content of carbohydrates as well as specific rotation and possibilities of using these criteria (content of carbohydrates, specific rotation, and activity of invertase) for characterization of honey quality. Following parameters were determined with different physical – chemical methods: specific rotation – by method of polarimetry, content of sugars with high pressure liquid chromatography, activity of invertase and content of proline – spectrophotometrically. The obtained results indicated that content of carbohydrates partially dependent on honey sorts. Content of sacharose depends from invertase activity in honey. Invertase is good parameter for honey characterization. Specific angle of rotation of polarized light is not available for identification of honey sorts.

INTRODUCTION

Honey is a complex natural product, containing more than 400 different substances, e.g. various carbohydrates, organic acids, proteins, amino acids, enzymes, aroma substances, mineral substances, pigments, waxes, etc (Belitz *et al.,* 2008).

In market comes honey sometimes with unfamiliar origin. Therefore quality control of honey is important. The scientists discussed about necessity of introduction new criteria of honey quality in a place old, for example activity of amylases. These criteria are activity of invertase and content of proline in honey.

Invertase hydrolyzes sacharose about fructose and glucose. Invertase is more sensitive to heat than amylases and loses activity during storage faster compared to amylases. That is why in few countries (Italy, Switzerland) invertase is used as additional criteria to characterize honey quality. As freshness indicator invertase is also used in honey standards of the beekeepers association in Germany, Belgium and Spain (Bogdanov, 1999).

In conformity to EU recommendations it was proposed that fresh and unheated honey should have an invertase number (IN) higher than 10, but

for honey with low enzymatic activity IN higher than 4 is recommended (Bogdanov, 1999).

Amino acid proline is one of the main amino acids of honey. Natural honey should contain this acid. Therefore definition the content of proline in honey can serve as a parameter of quality of honey. In conformity to EU recommendations it was proposed that natural honey should have proline content not less than 180 mg kg^{-1} (Bogdanov, 1999; Muli *et al*., 2007).

One of the main parameters for an estimation of honey quality is the content of sugars in honey. The name of mix from sugars glucose and fructose is invert sugars. The content of glucose and fructose in honey average is 31.3% and 38%. In her turn content of maltose in honey is to 9%, but content of sacharose to 8%, in several honey sorts yet more. Proportions of glucose and fructose partially determine the crystallizations speed of honey. Fructose determines the hygroscopic features of honey, but glucose – the speed of honey crystallization. Honey with partially crystallization, the top liquid layer, basically contains fructose (Farmer, 2003; Kasenburger, 2006; Belitz *et al*., 2008).

The highest content of sacharose and lowest content of invert sugars (glucose and fructose) characterize the bad maturing of honey or else about bee feeding with sacharose (Шабаршов, 2002).

EU and Latvia are adopted the following standards of quality control for honey sugars: invert sugars in flower honey – no less than 60%, in honeydew honey – no less than 45%; sacharose – no more than 5%, in some exceptions – 10% (Council Directive 2001/110/EC, 2002). The estimation the content of other sugars in honey characterizes specific properties of honey.

Each carbohydrate has a specific angle of rotation of polarized light (specific rotation). It is depending on relations and content of carbohydrates in honey. As now, that specific rotation of carbohydrate fructose is – 92.4°, specific rotation of glucose +52.7°, specific rotation of sacharose +66.5°, but specific rotation of maltose 130.4° (Чепурной, 2002). There are discussions about possible use of specific rotation for identification of separate kinds of honey.

The aim of the present research was to establish the relationship between honey sorts and content of carbohydrates as well as specific rotation and possibilities of using these criteria (content of carbohydrates, specific rotation, proline content and activity of invertase) for characterization of

honey quality. Research the relationship between invertase activity and content of sacharose in honey.

MATERIALS AND METHODS

Following parameters were determined with different physical – chemical methods.

- Content of carbohydrates in honey was determined by method of high pressure liquid chromatography (HPLC).

Principle. After filtration of the solutions, the sugar content is determined by HPLC with RI detection. Peaks are identified on the basis of their retention times.

Parameters for method:

column: Altima Amino 100A 5 u;
flow rate: 1.3 ml min^{-1};
mobile phase: acetonitrile/water (70:30 v/v);
column and detector temperature: 30 °C;
sample volume: 10 µl.

- The activity of invertase in honey samples was determined by method of spectrophotometry.

Principle. p - nitrophenyl - α - D – glucopyranoside (pNPG) is used as substrate for the determination of the invertase number in honey. pNPG is split into glucose and p – nitrophenol by invertase. By adjusting the pH value to 9.5 the enzymatic reaction is stopped and at the same time nitrophenol is transformed into nitrophenolate anion, witch corresponds to the amount of converted substrate and is determined spectrophotometrically at 400 nm.

- The content of proline in honey samples was determined by method of spectrophotometry.

Principle. Proline and ninhydrin form a coloured complex. After adding 2-propanol, the extinction of the sample solution and a reference solution at a wavelength maximum is determined. The proline content is determined from the ratio.

- Specific rotation was determined by method of polarimetry.

Principle. The angular rotation of a clear, filtered aqueous solution is measured by means of a polarimeter. The value is related to the carbohydrate composition.

RESULTS AND DISCUSSIONS

In our work was analyzed various honey sorts from different regions of Latvia. Results of investigations of different kinds of honey are shown in Table 1. From the received results it is possible to draw conclusions, that at some kinds of honey the raised content of separate carbohydrates. Example, the raised content of turanose has honey of heather and phacelia flowers, but at other carbohydrate the content in honey approximately identical. It is possible to ascertain, that the content of carbohydrates are not full depending from honey kinds. At analyzed honey samples the ratio fructose/glucose is in an interval 0.98 – 1.26. It is known, that the crystallization of some honey kinds are faster, and the crystallization of some honey kinds are more slowly. Speed of crystallization in honey is defined with a proportion and the content of carbohydrates in honey.

Table 1
Content of carbohydrates in honey, %

Kinds of honey	Place of gathering	Content of carboxydrates, %			
		Sach.*	Gluc.*	Fruc.*	Malt.*
Various flowers	Ludza	2.32	33.13	38.64	2.08
Various flowers	Jekabpils	1.92	35.23	37.10	3.92
Various flowers	Cesis	2.28	36.13	36.17	3.02
Wild flowers	Cesis	2.12	33.09	41.74	2.73
Wild.flowers	Madona	2.31	33.93	35.32	4.95
Lime blossom	Riga	1.98	35.21	37.72	1.95
Lime blossom	Talsi	2.17	36.31	38.04	2.11
Dropwort flow.	Valka	2.14	33.17	40.50	3.81
Heather flowers	Limbazi	2.40	30.20	37.97	2.24
Meadow flowers	Riga	2.32	44.58	36.94	1.21
Buckwheat flow.	Saldus	1.72	33.53	38.69	2.62
Phacelia flowers	Jelgava	2.31	35.25	40.52	1.03
Sweet flowers	Riga	2.69	35.86	37.30	4.99

Kinds of honey	Place of gathering	Content of carboxydrates, %		
		Turan.*	Malttr.*	Meliz.*
Various flowers	Ludza	2.14	0.09	0.96
Various flowers	Jekabpils	2.18	0.62	0.56
Various flowers	Cesis	1.83	0.12	0.10
Wild flowers	Cesis	1.17	0.65	0.67
Wild.flowers	Madona	2.26	2.22	0.02
Lime blossom	Riga	2.12	0.82	0.35
Lime blossom	Talsi	1.83	0.72	0.56
Dropwort flow.	Valka	1.38	-	0.61
Heather flowers	Limbazi	3.78	1.46	0.82
Meadow flowers	Riga	0.97	0.44	-
Buckwheat flow.	Saldus	0.92	0.09	
Phacelia flowers	Jelgava	5.01	1.09	0.97
Sweet flowers	Riga	1.80	0.21	0.11

* Sach. – sacharose; Gluc. – glucose; Fruc. – fructose; Malt. – maltose; Turan. – turanose; Malttr. – maltotriose; Meliz. – melizitose.

It is known, that carbohydrate glucose promotes the crystallization of honey, and however carbohydrate fructose breaks crystallization of honey. Crystallization of such kind as heather blossom honey is slowly. About the analysis of ours research can see, that the content of glucose in heather blossom honey one of the smaller. It is necessary to note, what even in boundaries of one kinds of honey, honey can have a different speed of the crystallization. The content of reducing sugars is differentiated in quality standards of flowers and honeydew honey. The data of our analyses give evidence that content of reducing sugars and sacharose in explored honey's samples complies with requirements of the EU quality standarts.

The content of proline in honey, specific rotation and activity of invertase is summarised in Table 2.

In conformity to EU recommendations it was proposed that fresh and unheated honey should have an invertase units (IU) higher than 73, but for honey with low enzymatic activity IU higher than 29 is recommended (Bogdanov, 1999).

Table 2
Content of proline, specific rotation and activity of invertase in honey.

Kinds of honey	Place of gathering	Activity of invertase, IU	Content of proline, mg kg^{-1}	Specific rotation, $[\alpha]_D^{20}$
Various flowers	Ludza	138	734	-12
Various flowers	Jekabpils	201	820	-9
Various flowers	Cesis	72	643	-14
Wild flowers	Cesis	68	920	-5
Wild flowers	Madona	74	1100	-16
Lime blossom	Riga	91	500	-7
Lime blossom	Talsi		380	-8
Dropwort flowers	Valka	63	490	-16
Heather flowers	Limbazi	95	1195	-10
Meadow flowers	Riga	77	1400	-14
Buckwheat flowers	Saldus	208	800	-16
Phacelia flowers	Jelgava	74	320	-14
Sweet flowers	Riga	58	740	-8

From our results we can see, that in greet deal of honey samples invertase activity is higher then 10 IN or 73 IU. The correlation between activity of invertase and the content of sacharose in honey was determined. Our observations are disclosed in Figure 1.

From the results of regression analysis (significance level 0.05) ascertained, that there is the some dependence between the content of sacharose in honey and activity of invertase. As can see from Figure 1, the more the content of sacharose in honey, then less is activity invertase in honey. Despite of rather low activity of invertase at some samples of honey, it is impossible to approve, that in these samples the lowered quality.

Proline is one of the main amino acids in honey. It is used as indicator of ingenuousness of honey. From the received results (Table 2) it is possible to draw conclusions, that content of proline in analysed honey samples is higher then the minimal requirement (180 mg kg^{-1}) of "International Honey Commission" for natural honey. So it is evident that all samples analyzed were natural honey.

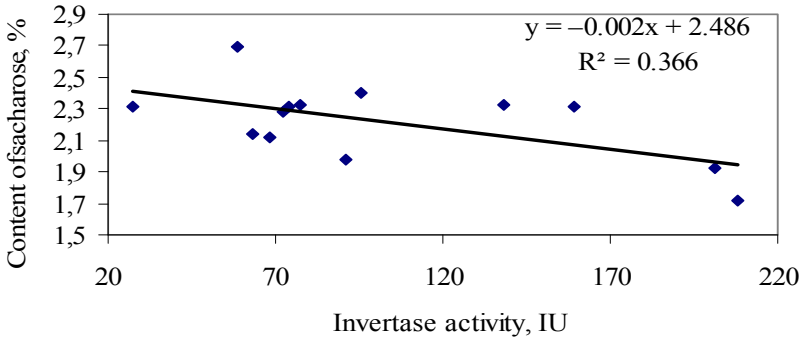

Figure 1
Influence of invertase activity in honey on the content of sacharose.

From results of specific rotation (Table 2) it is possible to ascertain, that specific rotation of honey is not depending from a honey kinds. It means that the parameter of specific rotation cannot be used for identification of honey kinds. It is necessary to note, that at all samples of honey specific rotation is a negative size. Polarized light of all analyzed honey samples turn on left. Specific rotation could be used, as auxiliary parameter for identification of natural honey.

CONCLUSION

- Invertase activity can be used as one of the parameters for characteristic of honey quality.
- Proline can be used for characterizing ingenuousness of honey.
- In great deal of analyzed honey samples activity of invertase was not lower as 73 IU, but content of proline was higher as 180 mg kg^{-1}. It's certify good quality of honey.
- Content of invertsugars (glucose and fructose) and sacharose in honey correspond with quality criteria of EU.

- Specific rotation of light cannot be used for identification of honey kinds.
- For natural honey specific rotation of light should be negative.
- For objective appraisal of honey quality it is necessary to consider all parameters in a place.
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RESPONSE SURFACE METHODOLOGY IN RHEOLOGICAL CHARACTERIZATION OF QUINCE PUREE

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ABSTRACT

Rheological properties of quince puree were investigated with respect to processing conditions by using response surface methodology. Effects of total solids content (10-18%), pH (3-4) and temperature (25-75°C) that could be encountered during processing on rheological properties of quince puree were determined in a central composite design. Flow behavior of quince puree was found to be pseudoplastic that could be described by Herschel-Bulkley model under all conditions studied. Temperature and total solids content were found effective on the consistency coefficient of quince puree. Yield stress of quince puree was found to change with pH and total solids content. Thixotropy in the samples decreased with increase in temperature. Interactive effects of some processing parameters were found significant on consistency coefficient, yield stress and thixotropy of quince puree. Rheological properties of quince puree were found to depend on processing conditions.

INTRODUCTION

The quince (*Cydonia oblonga*) is a pome type fruit with firm flesh and characteristic fragrance. The yearly production is approximately 381 000 tons in the world and about 27% of which is produced in Turkey (Gül and Akpınar, 2006). Because of its high pectin content, it is valuable both industrially as a gelling agent and nutritionally as a dietary fiber source. Quince is also a good source of phenolic compounds and minerals (Silva et al., 2002). It is largely used in jelly, preserves, marmalade and nectar production. Numerous studies have been conducted on rheological properties of fruit pulp, purees and paste (Guerrero and Alzamora, 1997; Ahmed and Ramaswamy, 2004; Haminiuk et al., 2006; Maceiras et al., 2007; Nindo et al., 2007). Ramos and Ibarz (1998) investigated the thixotropy of quince puree. The aim of this study was to investigate the

rheological properties of quince puree with respect to processing conditions by using response surface methodology. In this context, the effects of dry matter content (10-18%), pH (3-4) and temperature (25-75°C) that could be encountered during processing on rheological properties of quince puree were determined in a central composite design.

MATERIALS AND METHODS

Material

Quince puree was kindly provided by DIMES Food Industry and Trade Inc. Co., Tokat, Turkey in 5 kg sterile pouches. The total soluble solids content was 15.4 Brix, total solids content was 19.26%, ash content was 0.33% and its pH was 3.62 all of which were evaluated according to the standart method of AOAC (2000). The amounts of alcohol insoluble solids, water soluble pectins and acid soluble pectins were determined as 5.93%, 1.45%, 0.56%, respectively by using the methods reported by Günes et al. (2001) and Haminiuk et al. (2006).

Quince puree packed in plastic pouches (500 g) were kept frozen at -18°C until analysis. Prior to analysis, a frozen sample was thawed and equilibriated to the room temperature. Test samples containing 10, 14 and 18 % of total solids were prepared by adding calculated amount of distilled water to the sample. pH of the samples were adjusted by using citric acid or sodium hydroxide. The samples were analysed at the same day and measurements were always carried out in the freshly prepared samples.

Experimental design

Response surface methodology was used used in designing this experiment. A three factor and three level central composite design (α=1) consisting of 20 experimental runs was employed including six replicates at the center point. The independent variables were the temperature (X_1, °C), pH (X_2) and total solids (X_3, %). The levels of independent variables were selected based on the conditions to be encountered in a processing plant (Table 1). The responses were consistency index, flow behaviour index, yield stress and tixotropy. The order of the experiments were randomized in order to minimize the effect of extraneous factors on the observed response. Experimental design was generated and analysed by using MiniTab 14.2 (MiniTab Inc., USA).

Table 1
Independent variables and their coded and actual values used in the experiments

Independent variable	Units	Symbol	Coded levels		
			-1	0	+1
Temperature	°C	X_1	25	50	75
pH		X_2	3.0	3.5	4.0
Total solids	% (w/w)	X_3	10	14	18

Rheological measurements
The rheological measurements were made by using plate-plate sensor and rheometer (Rheostress 1, Haake, Germany). Approximately 1 ml sample was poured to lower plate of the sensor very slowly and the sample was allowed to equilibriate for 5 min before starting the experiment. To prevent the evaporation of the water, the surface of the sample exposed to air was covered with a thin layer of liquid paraffin. An efficient temperature control was provided by the rheometer used in this study.
Flow curves were obtained at controlled shear rate mode during which the shear rate was increased linearly from 0 to 300 s^{-1} in 360 seconds and immediately decreased from 300 s^{-1} to 0 s^{-1} in the next 360 seconds. All the measurements were duplicated and every time a new sample was loaded into the measuring unit. Fitting of the flow curve data to the rheological models was performed by using nonlinear regression method in SPSS statistical software (SPSS version 10, SPSS Inc., USA).

RESULTS AND DISCUSSION

Rheological properties of quince puree
All of the quince puree samples exhibited pseudoplasticity with a yield stress (Fig. 1). Herschel Bulkley model (Eq. 1) was found efficient to describe the relationship between shear stress and shear rate ($R^2 \geq 0{,}997$) of all the quince puree samples in this study.

$$\tau = \tau_0 + K(\dot{\gamma})^n \qquad \text{(Equation 1)}$$

where, τ is the shear stress (Pa), τ_0 is the yield stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), k is the consistency index ($Pa.s^n$) and n is the dimensionless

flow behaviour index. Rheological properties of quince puree samples were calculated from the upper flow curve in accordance with the above model. The area enclosed by the hysterisis loop between up and down flow curves was determined as an indication of thixotropy.

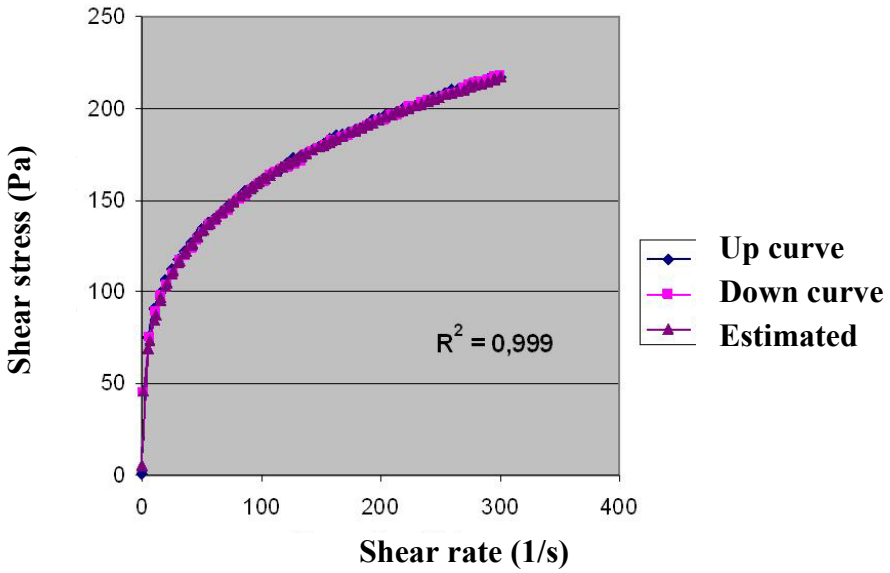

Figure 1
Experimental and estimated (Herschel-Bulkley model)
flow curve of quince puree at 18% total solids, pH 4 and 25°C.

The values of flow behaviour index (n) were less than 1 (n<1) for all conditions in the study indicating that quince puree exhibited a shear thinning behaviour (Table 2). The same behaviour was also reported in previous studies done with whole araça pulp (Haminiuk et al.,2006) and mango pulp (Bhattacharya, 1999). Thixotropic behaviour was also reported for quince puree by Ramos and Ibarz (1998) and mango pulp by Ahmed et al. (2005).

Effects of processing parameters on rheological properties of quince puree

Each rheological property obtained from the Herschel Bulkley model was fitted to a second order polynomial model (Eq. 2) which was used in the response surface analysis:

Table 2.
erimental design and data for the responses; consistency index (k), flow index (n), yield stress (τ_o) and thixotropy of quince puree under different conditions

T (^oC) (*X1*)	**pH** (*X2*)	**C (%)** (*X3*)	**k ($Pa.s^n$)** (*Y1*)	**n** (*Y2*)	**τ_o (Pa)** (*Y3*)	**Thixotropy (Pa/s)** (Y_4)
25	4	10	2.2	0.49	3.6	23
25	3	18	18.8	0.38	71.2	2472
75	4	18	6.1	0.48	36.8	1091
50	3.5	14	5.2	0.43	13.0	664
50	3.5	14	5.3	0.43	14.5	654
75	3	10	0.8	0.54	4.6	3
50	3.5	14	4.5	0.46	14.6	587
75	3	18	4.5	0.51	37.6	1426
50	3.5	14	4.7	0.46	12.7	474
25	3	10	2.0	0.50	4.1	58
75	4	10	1.0	0.52	3.5	2
25	4	18	29.1	0.33	49.0	2694
50	3	14	3.3	0.49	20.8	870
25	3.5	14	9.4	0.39	18.0	746
50	3.5	18	9.8	0.46	46.5	1587
50	3.5	14	4.9	0.45	17.1	692
50	3.5	14	4.9	0.46	19.9	736
50	4	14	4.6	0.45	20.4	797
50	3.5	10	1.3	0.51	2.1	36
75	3.5	14	3.7	0.43	8.4	412

Table 3.
Regression coefficients and correlation coefficient (R^2) for the response functions†

Coefficient of independent variables	**k (Pa.s^n)** (Y1)	**n** (Y2)	**τ_o (Pa)** (Y3)	**Thixotropy (Pa/s)** (Y4)
b_0	6.646	1.163	1.834	0.246
T (X1) (b_1)	-0.35	-0.004	0.192	**-57.95***
pH (X2) (b_2)	1.646	-0.173	**-42.422***	-51.71
C (X3) (b_3)	**4.29***	-0.043	-2.488	73.76
TxT (X1xX1) (b_{11})	**-0.003***	-0.003	-0.003	-0.071
pHxpH (X2xX2) (b_{22})	-1.816	0.0311	**-20.81***	11.87
CxC (X3xX3) (b_{33})	**0.316****	0.001	**0.558***	**847.94***
TxpH (X1xX2) (b_{12})	-0.024	0.004	**0.209***	-5.24
TxC (X1xX3) (b_{13})	**-0.062****	0.002	**-0.058***	**-3.22****
pHxC (X2xX3) (b_{23})	0.345	-0.005	**-1.338***	-4.74
R^2	**0.995**	**0.971**	**0.991**	**0.984**

† b: coefficients of polynomial equation; b_0 (constant); b_1. b_2 and b_3 (linear effects); b_{11}. b_{22} and b_{33} (quadratic effects); and b_{12}. b_{13} and b_{23} (interaction effects).

* Significant at $p \leq 0.05$.

** Significant at $p \leq 0.001$.

$$Y = b_0 + \sum_{n=1}^{3} b_n x_n + \sum_{n=1}^{3} b_{nn} x_n^2 + \sum_{n=m=1}^{3} b_{nm} x_n x_m \qquad \text{(Equation 2)}$$

where the coefficients of the polynomial were represented by b_0 (constant term); b_1, b_2 and b_3 (linear effects); b_{11}, b_{22} and b_{33} (quadratic effects); and

b_{12}, b_{13} and b_{23} (interaction effects). The coefficients of the independent variables and corresponding correlation coefficient (R^2) for the models are shown in Table 3. The statistical analysis indicated that the proposed models were adequate with satisfactory values of R^2.

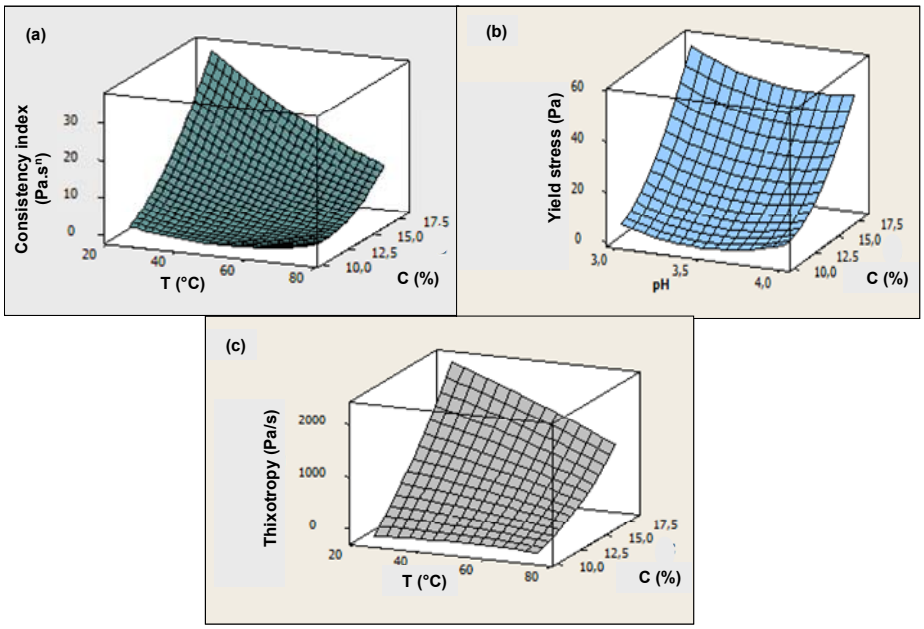

Figure 2
Interactive effects of processing parameters on consistency index (a), yield stress (b) and thixotropy (c) of quince puree

There were no significant effects of the processing parameters on flow behaviour index (Table 3). Linear effect of concentration, quadratic effects of both temperature and concentration and interactive effect of only temperature and concentration were found significant on consistency index of quince puree (Fig. 2a). Linear and quadratic effects of pH, quadratic effect of concentration and interactive effects of all processing parameters were found significant on yield stress of quince puree (Fig. 2b). Significant linear effect of temperature, quadratic effect of concentration and interactive effect of temperature and concentration were observed on thixotropy of quince puree (Fig. 2c).

Conclusions

Quince puree was found to be pseudoplastic with a yield stress in nature. Herschel-Bulkley model was found applicable to describe flow curve of quince puree. Thixotropy was also observed and intensity of which was found to be dependent on the processing conditions. Interactive effects of processing parameters had significant effects on the consistency index, yield stress and thixotropy of quince puree.

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CHANGES IN RHEOLOGICAL AND FOOD-CHEMICAL PARAMETERS IN SWEET MELON VARIETIES DURING THE POST-HARVEST TREATMENTS.

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ABSTRACT

The aim of our research was to study the effect of the different storage system different post-harvest handling for the 4 type of sweet melon varieties in different maturity stage on different storage temperature (4 and 8 ^{o}C) for the storability measured on different way and test the change sugar content storage.

The storage results of different variety shown clearly the long-shelf life varieties were Main results of our research work was the evaluation of the storage results of the melon fruits which were harvested in different

maturity stage. Results of dropping-treatment, model of ill-treatment shown dramatically deterioration loss.

INTRODUCTION

In Hungary the sweet melon consumption is relatively low comparing to another European countries. Hungarian traditional melon growing based on early, normal maturity, quick over-ripening varieties, in that case the harvest time and the time of the fresh consumption from local production are short. Chance to increase the Hungarian consumption of melon are the introduction and entering on the market the new, long-shelf life (LSL) varieties(Füstös ,2005,2007.). The adopted method of the short storage and post-harvest handling is not in Hungarian trade practice. We had experiments to study the effect of the different storage temperature, different post-harvest handling for the 4 type of sweet melon varieties in different maturity stages

MATERIAL AND METHODS

We tested registered melon varieties in EU Common Catalogues. The fruits were grown in the Variety Trial Station of Central Agricultural Office in Kecskemét, Hungary.
*Cantalup type melon varieties
Fiata :short shelf life, early maturity, orange flesh, quick over-maturity
Proteo: long shelf life, early variety, orange flesh, slow over-maturity
*Galia type melon varieties
Candy: short shelf life, early maturity, white-green flesh, quick over-maturity
Solarking, long shelf life, middle or late maturity, green flesh, slow over-maturity

Melon fruits of the varieties were harvested at two maturity stages: 50 % and 75%.
We stored the fruits in experimental storage chamber without any packing on 4 ^{o}C and 8 ^{o}C, at 90-95 % relative humidity, during 28 days. We sampled the stored fruits 4 times weekly. To simulate poor handling technique some fruits were dropped from height of 50 cm 2 or 4 times for

studying the effect of bruising on fruit firmness and weight loss. The treated fruits were stored and measured same than other ones.
Measurement methods were used for studying the changes in rheological parameters of fruits: weight of fruits - weight loss, skin and flesh firmness (Pankotai et al,2004, 2007) by manual penetrometer – FT 327, 0,8 mm diameter cylindrical probe, Firmness kg /mm , stiffness by acoustic method. We measured by PC with sound Card: Sound Blaster PCI512
Acoustic sensor: condenser microphone, preamplifer
Sample holder above the microphone
Software: Stiff,Measured: resonant frequence (f), Hz mass of the sample (m),g
Siffness = f 2 * m / 1 000 000 N/mm
Food-chemical parameters were detected from samples of three maturity stage: 50 % and 75%.100% : refraction (TSS) % by refractometer, sugars – glucose, fructose, sucrose by enzimatic method . The sweetness index was calculated.

RESULTS AND DISCUSSION

1.Table Weight loss (%) of varieties on different storage temperature.

Variety	Temperature	1,week	2.week	3.week	4.week
Cantalup type					
Fiata	***4°C***	***2,32***	***4,46***	***6,38***	***8,67***
	8°C	2,59	4,77	7,79	
Proteo	***4°C***	***1,96***	***3,24***	***4,02***	***5,74***
	8°C	2,18	4,32	6,2	
Galia type					
Candy	***4°C***	***2,31***	***4,04***	***6,25***	***8,11***
	8°C	2,48	4,57	6,93	
Solarking	***4°C***	***2,32***	***4,15***	***5,31***	***6,82***
	8°C	2,65	4,91	5,77	

All varieties were less weight loss, better storage results in the lower, 4°C temperature, In the chamber where the temperature was 8°C after 4 weeks were no edible fruits.

2. Table Weight loss (%) of Cantalup varieties cvs. maturity stages stored on 8°C

Variety	Maturity	1,week	2.week	3.week	4.week
Cantalup type					
Fiata	***50%***	***1,79***	***3,29***	***5,51***	***9,81***
	75%	3,39	6,25	10,08	
Proteo	***50%***	***1,99***	***3,80***	***4,98***	***7,11***
	75%	2,38	4,84	7,43	

The melons were harvested in earlier maturity stage got a better storage results of two Cantalup type varieties. The melons were harvested in 75% maturity stage after 4 weeks were no edible fruits.

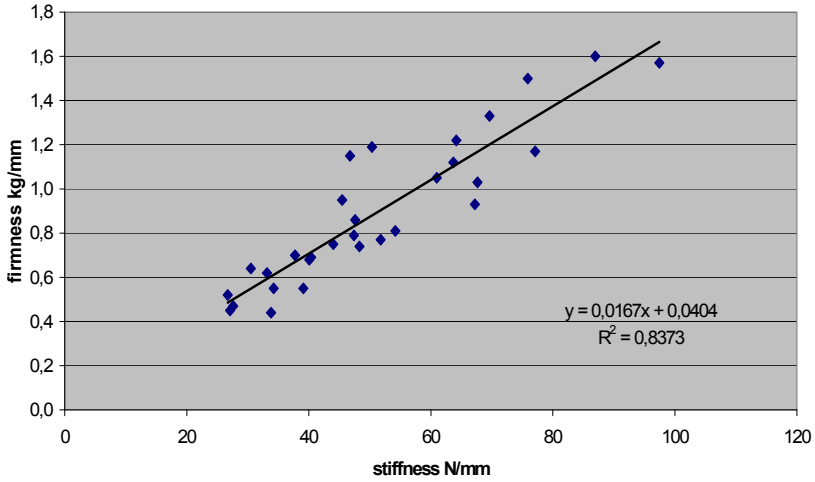

Figure 1
The correlation between the
destructive measurement by penetrometer (firmness) and
nondestructive measurement by acoustic method (stiffness)

The correlation of destructive and non-destructive measurement methods was very close.
We will present in the following the data of stiffness loss, characterized the quality of melons during storage

3.Ttable Stiffness loss (%) of varieties cvs. maturity stages stored on 8°C

Variety	Maturity	1,week	2.week	3.week	4.week
Cantalup type					
Fiata	***50%***	***43,97***	***51,00***	***59,00***	***70,46***
	75%	56,18	68,04	74,54	
Proteo	***50%***	***31,23***	***36,84***	***45,14***	***55,01***
	75%	46,65	54,25	64,65	
Galia type					
Candy	***50%***	***43,17***	***43,70***	***47,67***	***57,01***
	75%	50,31	52,28	56,71	61,43
Solarking	***50%***	***43,03***	***49,26***	***55,29***	***58,29***
	75%	33,12	37,3	47,78	

The melons were harvested in earlier maturity stage generally got a better storage quality instead of Solarking variety.

4. Table. Changes of sugar content of varieties during the storage

Variety, maturity	sucrose	fructose	glucose	total sugar	sweetness index
Fiata					
fresh	3,43	2,71	1,98	8,04	9,05
1. week	5,07	2,19	1,28	8,54	9,39
2. week	5,17	1,85	0,95	7,94	8,74
3. week	0,10	1,57	1,04	2,71	3,28
Proteo (LSL)					
fresh	2,50	1,85	1,37	5,71	6,34
1. week	4,47	1,26	1,07	6,80	7,19
2. week	5,02	1,37	1,00	7,38	7,85
3. week	4,64	1,07	0,63	6,33	6,76
4 week	3,09	0,88	0,55	4,52	4,85

The sugar content of Fiata variety decreased after 3 weeks. The sugar content of Proteo variety were less changeable.
Summary the results of poor handling experiments shown the big differences among the quality of intact and bruised melons. After dropping immediately 27-48 % firmness loss were detected in fruits, the ratio increased during storage time.

5.. Table. Results of dropping trials on firmness and weight loss average of tested varieties

	Storage period (days)		
	7	**14**	**21**
Stiffness loss %			
Control	33,10	40,05	48,48
2 drops	39,68	44,35	51,02
4 drops	47,23	53,8	61,73
Weight loss %			
Control	2,11	3,97	5,94
2 drops	2,22	4,03	5,73
4 drops	2,49	4,51	6,43

Conclusions

The storage results of different varieties shown clearly the long-shelf life varieties and earlier maturity stage gave better storage quality.
In lower temperature (4 ^{O}C) we measured better storage results.
Results of dropping-treatment, model poor handling shown dramatically deterioration loss.
The non-destructive acoustic measurement method is suitable for control of the melon fruit quality during storage time
The sugar contents decrease in normal maturity type and stable in LSL type during storage
Our research results add some detailed information for the better storage results of the different melon variety types. We hope help to supply for longer season of our market with better quality melon product, which will increase Hungarian melon consumption

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LEARNING OF PHYSICOCHEMICAL PARAMETERS OF THE SAUSAGE GOODS

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Meat and meat foods are one of the major components of feeding of a person. It is source of proteins and vitamins necessary for normal developing of an organism. The purposes of given job is study and introduction in educational process of express methodology by identification in meat products:

1. Content of nitrates and nitrites.
2. Calcium contents by method of complexometric titration.
3. pH by colorimetric method.
4. Quantitative moisture content.

During done work to comparative analysis of physicochemical parameters 5 sorts of summer sausages of superior quality were studied. All sorts of summer sausages was from different producers and were in one price range. They are:

1. Sausage Avstrijskaja - Grodno meat-packing plant.
2. Sausage Ivanovskaja - Volkovysskij meat-packing plant.
3. Sausage Slutskaja - Slutskij meat-packing plant.
4. Sausage Prazdnichnaja - Berezovskij meat-packing plant.
5. Sausage Italianskaja - Lidskij meat-packing plant.

Organoleptic characteristics.

In a course of organoleptic estimation correspondence of main quality characreristics (external view from a surface and on sectional view, smell, taste, consistency) of products to State Standards requests is established. Researched sample (stick of sausage) release from envelope and cut up along to diameter. At an assessment of external view pays attention to colour, uniformity of colouring, frame and state of separate components. Stickness and sliming presence is established by fingering of product. Smell in depth of product defines after cutting of envelope and surface layer. Consistency determines by light pressing of finger on fresh section of stick.
In researched samples of summer sausages on a surface of stick were not watched contaminations, sticking of mould, slips, fimbrias and bristle remainders. Consistency of these samples is dense. Colour of samples on cut is from pink to dark red with interlayer of intermuscular fat and inclusion of spices. Taste is slightly acute, saltish with expressed aroma of smoking and spices and without of strange smack.

Identification of pH

Identification of pH samples of summer sausages was spent by colorimetric method. This method is grounded on property of indicators to variety its colouring depending from pH solution. The universal indicator consisting of mixed indicators (1 - methyl red, 2 - bromthymol blue, 4 - phenolphthalein dissolved in ethanol) was used to research.
For measuring of acidity of researched samples in mass 5 gram were taken. They were carefully crumbled up to homogeneous mass which one was run with distilled water and mixed. In 30 minutes 1 ml of tested solution brought into test glass and added 3-5 drops of universal indicator. Occur bed colouring compared with data about colouring of the indicator against pH value. Following results have been received:

Name of sample	**pH**
Sausage Avstrijskaja	4,2
Sausage Ivanovskaja	5
Sausage Slutskaja	5,6
Sausage Prazdnichnaja	4,6
Sausage Italianskaja	4,8

At repeated determination of acidity on 14 days later in samples №1 sausage Avstrijskaja and №5 sausage Italianskaja insignificant increasing of pH on 0,1 was watched. Increasing of acidity at other samples was not seen.
Received results match to fixed legitimate values pH of summer sausages. Weak-acid environment contributes to a more long term of storage of finish products which keeping time reaches several menthes.

Identification of nitrites and nitrates.

At production of meat products traditionally in small doses sodium nitrate and potassium nitrate are used. Their using stipulates colour, taste and aroma of finished products and also inhibit growth of micro flora and formation of toxins. According to the State Standard 29299-92 mass fraction of nitrates should not exceed 0,005 %.
Contents of nitrites and nitrates are determined by method of direct potentiometry under the graduate diagram. For constructing of diagram from crystalline potassium nitrate prepare solutions with concentration from 10^{-1} to 10^{-5} mole/l. Potential (E, мВ) of each solution is in turn measured by received results the graduate diagram of dependence of potential from concentration of nitrate ions is graph.
Then receive an extract from tested samples flooding exact masses of ground samples by hot water and mixture puts in vibromixer and extracts 30 minutes. Content cools and filters.
Remained proteins deposit with mixture of sodium hydroxide of zinc sulphate. Flacks warm 5 minutes on the water bath at temperature of boils and then cool off and filter again. In received results potentials are measures. Then under the graduated graph discover an initial concentration of nitrates in analyse extracts.
For identification of their nitrites samples are previously oxidised with ammonium persulfate to nitrates and again measured potential. Under the graduate graph discover concentration of nitrates after oxidising of nitrites. Difference between total contents of nitrates and their initial concentration is equal to concentration of nitrites in analysable solution.

Name of sample	ω, NO^{3-} %	ω, NO^{2-} %
Sausage Avstrijskaja	0,000147324	0,00427219
Sausage Ivanovskaja	0,000136635	0,00317955
Sausage Slutskaja	0,0000888537	0,00514152
Sausage Prazdnichnaja	0,0000873153	0,00370634
Sausage Italianskaja	0,000118107	0,00496494

During researches were established that all samples contain small quantity of nitrates which in small amount are contained in meat and also oxidation of nitrites. The lowest content of nitrites was fixed in sausage Ivanovskoj of Volkovyskij meat-packing plant. In sausage Slutskaja productions of Slutskij meat-packing plant contents of nitrites is insignificantly exceeded on 2,83 %. At other samples the index of content of nitrites matches to State Standards Republic of Belarus.

Calcium content identification.

Calcium content identification is grounded on preliminary incineration of analysable samples, obtaining of ashes solution and cimplexometric titration in the presence of mureksid.
Exact masses of researched samples are transported to crucibles and charred on an electric stove. Then samples are putted in muffler and burn at 450-500 ^{0}C to constant mass. After burning crucibles with ash put on boiling water bath and dissolve ash by hydrochloric acid. Received solutions filter through a filter paper «blue band» in measuring bottle.
Then aliquot parts of filtrates neutralise on indicator paper «kongo» adding by drops solution NaOH and deposit on an end of spatula dry mix of mureksid with NaCl. After that flow by 2 ml of solution NaOH and titrate by solution of complex on III before transferring of pink coloration in violet. Ground titrating data the calcareous content is found:

Name of sample	ω%, Ca^{2+}
Sausage Avstrijskaja	0,002514
Sausage Ivanovskaja	0,004815
Sausage Slutskaja	0,003279
Sausage Prazdnichnaja	0,003197
Sausage Italianskaja	0,003183

During researches exact calcareous content at samples of summer sausages were established. Its mass fraction is extremely insignificant. It means that sausage cannot be attributed to products rich on this microelement.

Identification of humidity content.
For moisture content identification in weighing bottles put clean sand in quantity approximately in 2-3 times exceeding mass of researched samples. Also in each weighing bottle put a glass rod and dry up in drying box at the temperature of 150°C within 30 minutes. Then cool off in exsiccator to room temperature and weigh. After to weighing bottles with sand put exact mass of product in mass 3gram and carefully mix with sand. They dry up in drying box in open weighing bottle at the temperature of 150°C within 1 h. After during weighing bottles cover, cool off in exsiccator to room temperature and weigh. Analogous instrumentation was conducted later 14 days:

Name of sample	Mass fraction of moisture (primary)	Mass fraction of moisture (after 14 days)
Sausage Avstrijskaja	30,77	24,45
Sausage Ivanovskaja	21,48	17,37
Sausage Slutskaja	20,88	17,42
Sausage Prazdnichnaja	25,67	16,95
Sausage Italianskaja	23,18	18,16

Originally exceeding of norm of moisture content was watched only in sample №1 sausage Avstrijskaja. Rest samples precisely match to State Standard norms. Later 14 days (storage with damaged envelope) moisture content was watched in all formation samples on the average on 5,526 %.

CHANGES OF THE DARK BEER RHEOLOGIC PROPERTIES DURING STORAGE

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ABSTRACT

Results from measuring of rheologic properties of dark beer are shown in this paper. Dependencies of dynamic viscosity on rotational frequency of probe are shown. Dependencies of dynamic viscosity, kinematic viscosity and fluidity on temperature and on time of storing are described. Dependencies of dynamic viscosity on rotational frequency of probe had increasing shape. Dynamic viscosity had increased with time of storing. Dependencies of dynamic viscosity and kinematic viscosity on temperature had decreasing shape. Dynamic viscosity and kinematic viscosity had increased a bit with time of storing. Dependencies of dark beer fluidity are increasing with temperature. Fluidity of used sample had decreased a bit with time of storing.

INTRODUCTION

In this paper are shown results from measuring of rheologic properties of dark beer. We can include into rheologic properties these parameters: dynamic viscosity, kinematic viscosity, fluidity and tangential tension. Materials, where internal friction is generated, can be characterized by viscosity. Dynamic viscosity $\boldsymbol{\eta}$ is defined as a constant between tangential tension $\boldsymbol{\tau}$ and gradient of layer velocity **grad** $\boldsymbol{v}$.

$$\tau = \eta \ \mathrm{grad}\, v$$

Physical unit of dynamic viscosity is Pa.s and unit of tangential tension is Pa. Kinematical viscosity $\boldsymbol{\nu}$ is defined as a ratio between dynamic viscosity $\boldsymbol{\eta}$ and density of used material $\boldsymbol{\rho}$.

$$\nu = \frac{\eta}{\rho} \quad (1)$$

Physical unit of kinematic viscosity is $m^2.s^{-1}$. Reciprocal value of dynamic viscosity η is called fluidity φ and physical unit of fluidity is $Pa^{-1}.s^{-1}$.

$$\varphi = \frac{1}{\eta} \quad (2)$$

Rheologic properties were measured by many authors. Buchar et al. (2005, 2003), Severa et al. (2007) investigated these properties of eggs yolk, milk products and ketchups. Marudova and Zsivánovits (2005) described rheologic properties of pectin films. Biczó et al. (2005) examined methods for determination of rheologic properties of chocolate mass. At quality valuation of food material is important to know their physical properties particularly mechanical, rheologic and thermophysical (Božiková, 2005). Automatically controlled processes at manufacturing, at handling and holding require exact knowledge about physical quantities of materials. Still are detected new methods that are utilizing new modern apparatuses and microscopic components. Very fast development is possible to observe at utilization of microwave at measuring properties of soil and food (Hlaváčová, 2002).

MATERIAL AND METHODS

Used sample of dark beer was obtained from Department of Animal Husbandry and Food Production of Slovak University of Agriculture in Nitra. Measuring was performed by digital viscosimeter Anton Paar (DV-3P). Principle of measuring by this viscosimeter is based on dependency of sample resistance against the probe rotation. Probe with signification R2 was used in our measurements. We were able to choose frequency of probe rotation from 0.3 min^{-1} to 200 min^{-1}.

Dependency of dynamic viscosity on temperature can be described by Arrhenius equation $\eta = \eta_0 e^{-\frac{E_A}{RT}}$ (3)

where η_0 is reference value of dynamic viscosity, E_A is activation energy, R is gas constant and T is temperature. This equation has decreasing exponential shape.
Sample of dark beer was stored in special cool box in temperature 3 °C and was measured in different days during two weeks. Measurements were done after the temperature stabilization from 7 °C to laboratory temperature. Dependencies of dynamic viscosity on frequency of probe rotation and on temperature are constructed. Dependencies of kinematic viscosity on temperature and temperature dependencies of fluidity are discussed. Dependency of all these parameters on time of storing is described.

RESULTS AND DISCUSSION

For illustration on Fig. 1 are dependencies of dark beer dynamic viscosity on frequency of probe rotation after different time of storing. Progress of graphic dependencies can be described by increasing linear function

$$\eta = M\left(\frac{f}{f_0}\right) + N \qquad (4)$$

where f is frequency of probe rotation, f_0 = 1 min^{-1}; M, N are constants dependent on kind of material, and on ways of processing and storing.

In Tab. 1 can be seen coefficients M, N of regression equation (4), and also that coefficient of determination had high values for all measurements.
Dependencies of dark beer dynamic viscosity on frequency of probe rotation had increasing linear shape for all measurements and it can be seen that dynamic viscosity had increased a bit with time of storing (Fig. 1). Dependencies of dark beer dynamic viscosity on temperature after different time of storing are shown on Fig. 2. Progress of graphic dependencies can be described by decreasing exponential function (5) or by decreasing linear function (6). Differences between these exponential and linear functions in this temperature range are not very significant for all dependencies. Small differences can be found only in coefficients of determination.

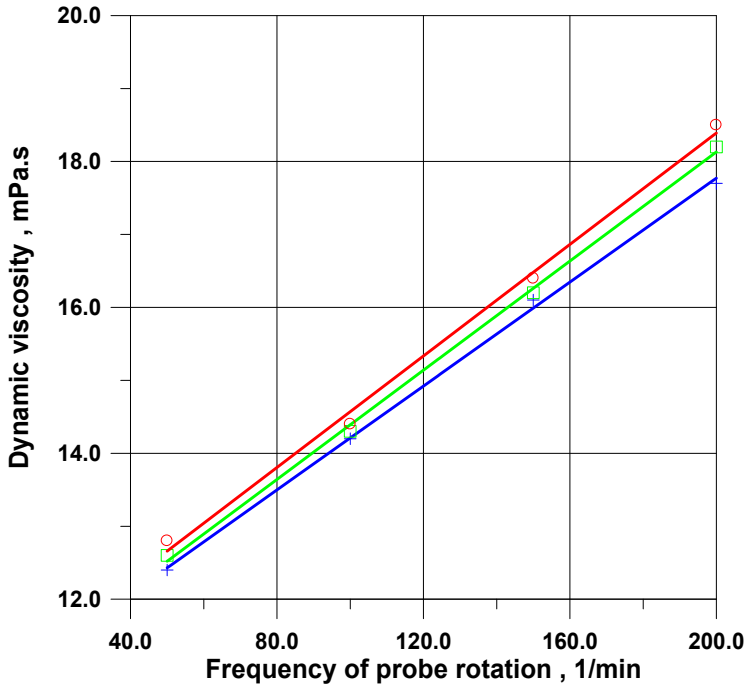

Figure 1

Dependencies of dark beer dynamic viscosity on frequency of probe rotation after different time of storing: first measurement (+), second measurement after one week of storing (□), third measurement after two weeks of storing (○).

Table 1

Coefficients M, N of regression equation (4), and coefficients of determination

Measurement Coefficients	First measurement	Second measurement	Third measurement
M	0.035 6	0.037 4	0.038 2
N	10.65	10.65	10.75
R^2	0.998 865	0.998 686	0.996 340

$$\eta = A e^{-B\left(\frac{t}{t_0}\right)} \tag{5}$$

$$\eta = -C\left(\frac{t}{t_0}\right) + D \tag{6}$$

where t is temperature, t_0 = 1 °C; A, B, C, D are constants dependent on kind of material, and on ways of processing and storing .
Temperature dependencies of dark beer dynamic viscosity had decreasing exponential shape and in this temperature range it is almost linear shape for all measurements (Fig. 2) (exponential function on the left side, linear function on the right side) and it is also evident that dynamic viscosity had increased a bit with time of storing (Fig. 2).

Table 2
Coefficients A, B, C, D of regression equations (5, 6), and coefficients of determination

Measurement Coefficients	**First measurement**	**Second measurement**	**Third measurement**
A	24.378 4	24.522 7	24.740 3
B	0.013 183 6	0.013 237 2	0.013 322 7
R^2	0.987 173	0.992 260	0.992 312
C	0.262 424	0.263 636	0.267 576
D	23.998 8	24.118 2	24.331 2
R^2	0.980 412	0.989 489	0.988 368

In Tab. 2 can be seen coefficients A, B, C, D of regression equations (5, 6), and also that coefficients of determination had high values for all measurements. Coefficients of determination are bit higher in case of decreasing exponential function than in decreasing linear function.

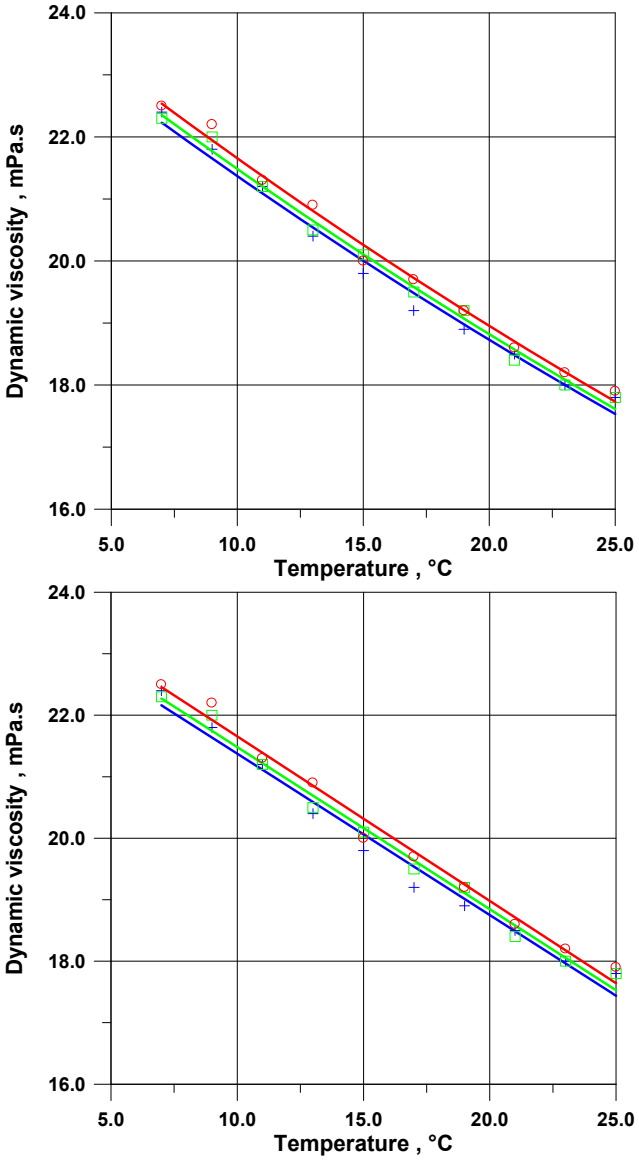

Figure 2

Dependencies of dark beer dynamic viscosity on temperature after different time of storing : first measurement (+), second measurement after one week of storing (□), third measurement after two weeks of storing (○).
(exponential function on the left side, linear function on the right side)

Kinematic viscosity and fluidity were calculated from equations (1), (2). Similar decreasing relations were found in temperature dependencies of dark beer kinematic viscosity. We also found out that dark beer fluidity is increasing with temperature. Kinematic viscosity of dark beer has increased with time of storing. Time of storing dependencies of dark beer fluidity has decreasing shape.

Discussion and conclusion

At quality valuation of food material is important to know their physical properties particularly mechanical, electric, rheologic and thermophysical. Automatically controlled processes at manufacturing, at handling and holding require exact knowledge about physical quantities of materials. Rheologic properties were measured by many authors.
Temperature dependencies of dark beer dynamic viscosity had decreasing exponential shape and in this temperature range it is almost linear shape for all measurements. Coefficients of determination are bit higher in case of decreasing exponential function than in decreasing linear function. Arrhenius equation (3) has decreasing exponential shape, so the dependency of dynamic viscosity on temperature can be described by it. It is also evident that dynamic viscosity had increased a bit with time of storing. Similar decreasing relations were found in temperature dependencies of dark beer kinematic viscosity. We also found out that dark beer fluidity is increasing with temperature. Kinematic viscosity of dark beer has increased with time of storing. Time of storing dependencies of dark beer fluidity has decreasing shape.
Measured values of dynamic viscosity and calculated values of kinematic viscosity and fluidity were obtained with good precision and all drawn dependencies had very high coefficients of determination.

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SOME ELECTRICAL PROPERTIES OF DRIED QUINCES CYDONIA OBLONGA

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ABSTRACT

The electrical measurements are utilized at appraisal of various fruits quality. Samples of dried quince were delivered by Faculty of Agriculture of University in Novi Sad. The samples were dried in osmotic drier on the beginning and in convective drier after it. Electrical resistance, impedance and capacitance were measured by LCR meter Good Will LCR-821. Measurements had been realized at frequencies from 100 Hz to 200 kHz. Frequency dependencies of electrical properties were determined for all samples. The capacitance, resistance, impedance and relative permittivity as well decrease powerly in dependence on the frequency. The differences between values of capacitance belonged to different quinces are very small. We can deduce that the method of drying ensured the same properties of all apricot pieces.

INTRODUCTION

The electrical measurements are utilized at appraisal of various fruits quality. For example Gordeev (1998) developed an apparatus for investigation the electrical parameters of fruit tissue, viz. polarization capacity and conductivity. Vozáry et al. (1999) described the impedance parameter characterized apple bruise. Mészáros et al. (2005) measured the impedance spectra of apple slices during drying and correlated impedance parameters to moisture content in the different drying periods. Electric capacity measurement was utilized for watermelon quality determination by Kato (1997). Muha et al. (2005) investigated of apricots maturity by non-destructive methods. Arnold et al. (1998) described electrical impedance methods for assessing fruit quality. Montoya et al. (1994) utilized a technique for measuring the electrical conductivity of intact

fruits. They measured the electrical conductivity of avocado fruits during cold storage and ripening. The health state of some fruit can be observed on ground of their electrical properties. Than et al. (1996) investigated the effect of pineapple blackheart on electrical resistance of pulp tissues. The electrical resistance of pulp from harvested pineapples with blackheart was lower than that of tissue from healthy pineapples. Resistance decreased with increase in disease severity. The measurement of the electrical resistance thus provides a rapid and convenient method of diagnosing the blackheart. Hlaváčová and Hlaváč (2003) measured the electrical properties of apricot flesh. It was found that the decay of apricot flesh influences its electric conductivity, impedance and capacity, which are caused by damage of cell membranes. The impedance decreased on values from 250 Ω till 900 Ω for decayed apricots; on the contrary the impedance of flesh intact with decay attained the values more than 13 kΩ.

MATERIAL AND METHOD

Samples of dried quince were delivered by Faculty of Agriculture of University in Novi Sad. Quince is a fruit with hard flesh, with many pips or seeds; it has high pectin content, and a wonderful fragrance. The samples were dried in osmotic drier in sugar solution with concentration of 0.85 and temperature 45 °C during 120 min on the beginning. Outcome moisture content wet basis was about 32 %. During the soaking in concentrated solution three mass transfer flows were taking place. One was water outflow from the tissue to the surrounding solution, the second was solute movement from the medium to the bioproduct, and the third mass transfer flow was the leaching of food solutes to the medium.
The samples were dried in convective drier after osmotic drying at temperature of 50 °C during 240 min. The method of drying is described by Babić Lj. et al. (2002). Final moisture content w. b. of quinces was about 20 %. For example these properties of dried fruits were noted: relative increase of sugar content and vitamin C content, and the taste improvement as well.
30 pieces of dried fruits were chosen and sliced on thickness of 5 mm. The dried pieces of fruits were located between 2 plates of capacitor. We measured the resistance, impedance and capacitance of samples by LCR meter Good Will 821 in the frequency range from 50 Hz to 200 kHz. The measured values were loaded by PC. Each electrical property was

measured at all frequencies three times. Average value has been computed from these ones.

RESULTS AND DISCUSSION

Frequency dependencies of resistance, impedance and capacitance were constructed from measured values. For illustration, on Fig. 1 the frequency dependencies of impedance for 6 samples of dried quince are shown.

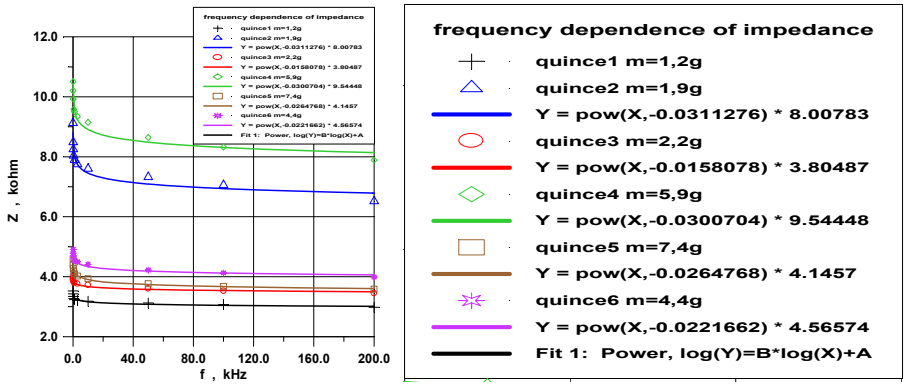

Figure 1
Frequency dependencies of impedance
for 6 samples of dried quince

The impedance decreases powerly with frequency according to the equation

$$Z = Z_o \left(\frac{f}{f_o} \right)^{-k} \qquad (1)$$

where: Z – impedance, Z_o – reference impedance, f – frequency, f_o = 1 kHz, k – constant. The coefficients of determination of this equation have high value for all samples. On Fig. 2 the frequency dependencies of resistance and impedance for sample 6 are shown. These dependencies have the same shape and differences are negligible. It is evident that in this frequency range the reactances of quince haven't influence on their impedance. On Fig. 3 the frequency dependencies of capacitance for 6 samples of dried quince are shown. The regression equation of these dependencies has the same shape as equation (1)

$$C = C_o\left(\frac{f}{f_o}\right)^{-k} \qquad (2)$$

where: C – capacitance, C_o – reference capacitance, f – frequency, f_o = 1 kHz, k – constant.

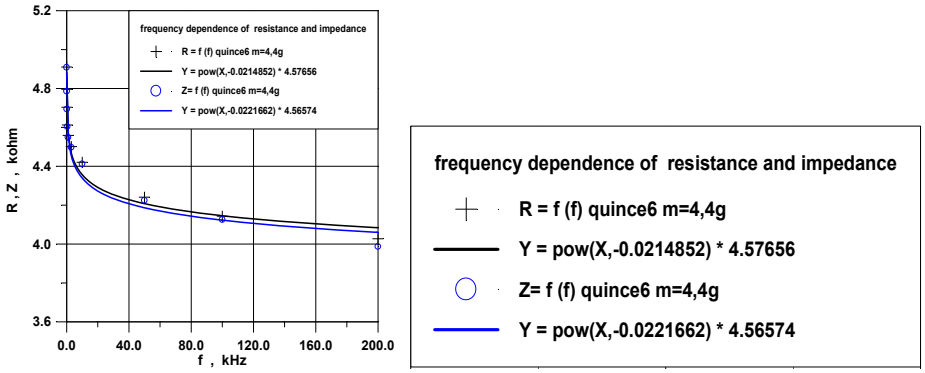

Figure 2
Frequency dependencies of resistance (+) and impedance (o) for sample 6

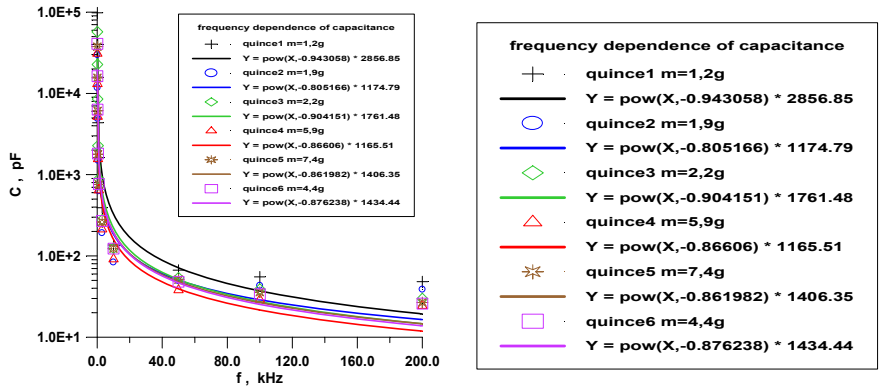

Figure 3
Frequency dependencies of capacitance for 6 samples of dried quince

The coefficients of determination have high value for all samples in this case, as well. The displacement between frequency dependencies of capacitance for 6 samples is very small. Average relative permittivity

calculated on the base of the capacitance and dimensions of samples has value about 12.

Conclusion

The resistance, impedance and capacitance decrease with frequency. The regression equation has the shape of decreasing power function as (1), (2). These equations have high coefficients of determination. The differences between dependencies for resistance and impedance are negligible. It is evident that in this frequency range the reactance of quinces hasn't influence on their impedance. The displacement between frequency dependencies of capacitance is very small. From this we can dedicate that the method of drying ensured the same properties of all quince pieces. Electrical properties can be used at the control of quince pieces identical moisture content after drying. As we had presented in Hlaváčová (2007) mix formula for the permittivity of the system of water and dry porous material has the shape

$$\omega = \frac{\sqrt{\varepsilon'} - \sqrt{\varepsilon'_m}}{\sqrt{\varepsilon'_w} - \sqrt{\varepsilon'_m}}$$

where: ω - moisture content w. b., ε' - the resulting permittivity, ε_w' - permittivity of (free) water, ε_m' - permittivity of the dry material. From this equation can be calculated the moisture content of dried fruits.

ACKNOWLEDGEMENT

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PHYSICAL QUALITY OF POTATO VARIETIES (SOLANUM TUBEROSUM L.)

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ABSTRACT

Ten potato varieties were studied for firmness of raw tubers and texture of the boiled product. Textural properties of selected varieties of potatoes were evaluated using unaxial compression test on a device Tira test 27025. Cylindrical specimens of the exact size were prepared from raw and boiled potatoes. The force needed for compression was recorded and assessed.
In raw potatoes the varieties with the biggest hardness was variety Red Anna (201,2 N), Keřkovské rohlíčky (186,5 N) or Korela (186,2 N). The variety Jitka needed only the force of 133,5 N to be compressed.
The most hard boiled potatoes was the varieties Rosara (12,0 N) and Ditta (11,3 N). Both of them are classed to cooking type AB. Minimal force for compression needed the variety Katka (3,8 N) which is cooking type BC. According to the results of the tests there is evident relation between the cooking type and hardness of boiled potatoes. It is valuable and could help us to observe some planting conditions, like physiological maturity in relation of potato quality for consumers.

INTRODUCTION

Texture of potatoes is qualitatively and economically important factor. According to the texture of boiled tuber are potatoes divided into various cooking types such as salads (A), multi-purpose (B) or floury (C), including transitional groups. It is based on disintegration, consistency, mealiness, dryness and structure. These are determined particularly by the starch content. Human perception of the texture may be imitated by instrumental methods in several forms such as chewing or biting. Now, there are identified some differentials in texture parameter during potato storage. Some varieties through the storage have different cooking type at the storage opening distinct of ending (SOLOMON, JINDAL, 2007). These used methods are accurate, repeatable and representative. It is also possible to investigate the relationship between the texture of raw and cooked potatoes.

MATERIAL AND METHOD

Potatoes *Solanum tuberosum L*. of the varieties Aneta, Ditta, Jitka, Karin, Katka, Keřkovské rohlíčky, Korela, Lolita, Red Anne and Rosara were used as the experimental material. The crop was from the School Farm in Žabčice which was harvested in September 2007. These varieties were including various cooking types of the potatoes ranging from the A to B and C type commonly used in Czech gastronomy. The tubers were stored under controlled conditions before the analysis. For testing the medium size tuber were used.
The laboratory device Tira test 27025 was used for evaluating the texture of potatoes. Cylindrical samples were prepared from raw tubers from the medium part of the tuber and not from the vascular ring using cork borer no.12 (diameter 12 mm, height 10 mm).

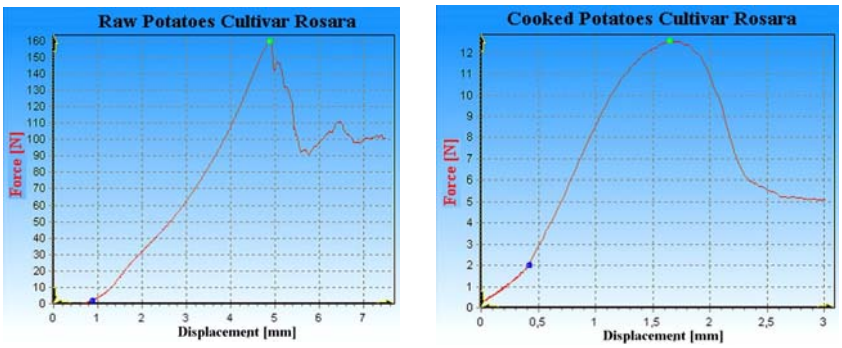

Figure 1
Force/displacement curves recorded
during Rosara specimen unaxial compression test

Raw samples were cooked in the distilled water for a period of 7 minutes. There were 50 specimens in the series. For evaluating of the texture was used uniaxial compression test. Raw samples was compressed between two steel plates to 75 % of its original height, cooked one to 30 %. The loading rate was 50 mm per minute. The dependance of the force on the time of compression was recorded (Fig.1). The hardness of the tubers of different varieties was compared and statistically evaluated (Unistat 5.1).

RESULTS

The classification by varieties was significant factor influencing the force needed for compression of the sample. Results are in Table 1 and Fig. 2. There were statistically significantly differences of the hardness between potato varieties. In raw potatoes the varieties with the biggest hardness was variety Red Anna, Keřkovské rohlíčky or Korela. The variety Jitka needed only the force of 133,5 N to be compressed.
The most hard boiled potatoes was the varieties Rosara and Ditta. Both of them are classed to cooking type AB. Minimal force for compression needed the variety Katka, which is cooking type BC. Generally there was no dependance of raw potato texture on the cooking type. But concerning cooked potatoes hardness it was found that less hard are cooking type BC and B while for cooking type AB force increased.

Table 1.
Results of force [N] necessary to compress the specimens

Potato varieties	Raw specimens			Boiled specimens		
	mean	st.dev.	var.	mean	st.dev.	var.
Aneta	152,0	24,8	0,2	7,8	3,1	0,4
Ditta	172,5	15,7	0,1	11,3	4,2	0,4
Jitka	133,5	12,0	0,1	9,0	3,1	0,3
Karin	149,1	14,3	0,1	5,2	1,7	0,3
Katka	154,6	24,1	0,2	3,8	0,7	0,2
Keřkovské rohlíčky	186,5	35,5	0,2	5,8	1,9	0,3
Korela	186,2	22,2	0,1	5,2	1,6	0,3
Lolita	165,4	17,2	0,1	7,4	2,5	0,3
Red Anne	201,2	26,0	0,1	6,6	2,1	0,3
Rosara	159,6	17,8	0,1	12,0	4,4	0,4

DISCUSSION AND CONCLUSION

Potato tuber is heterogeneous material and specimens varied a lot. The variation is larger for cooked samples than for raw potatoes, according to THYBO, VAN DEN BERG (2002). The findings of this study illustrate obvious differences between textural properties of potato varieties. It is

not only the variety what is the important influence. For example it was investigated by NUNN et al. (2006) that some vegetables had different values of texture score depending on type of boiling. Important influences are also the soil, a fertilizers or weather condition. But only with real values we can compare sufficiently different groups of specimens under various conditions (SOLOMON, JINDAL, 2007). Next our research will concentrate on these external influences on the texture of the potatoes. Our aim is to create useful database which would characterize each potato variety by texture and other analyses.

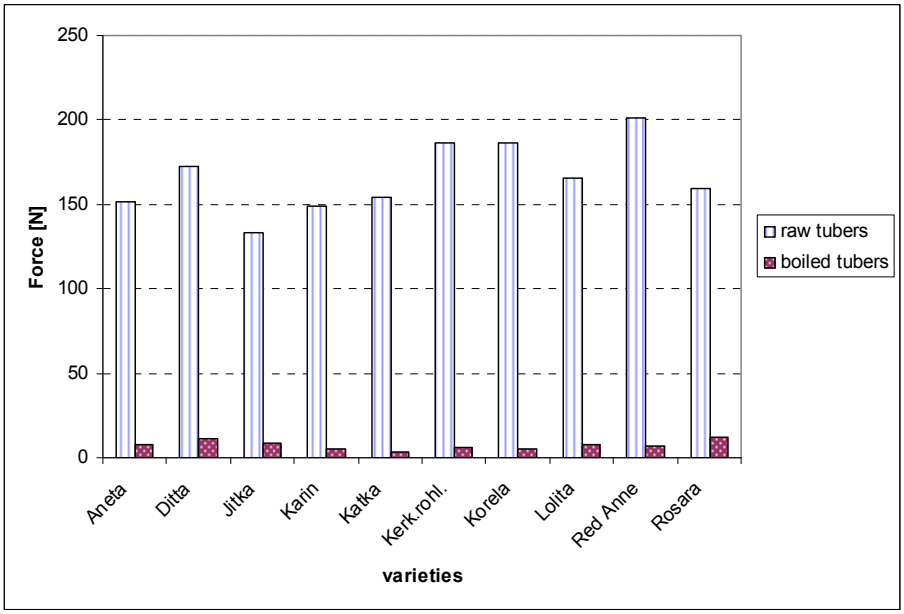

Figure 2
Results of the tubers hardness pursuant to different varieties

ACKNOWLEDGEMENT

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NON-DESTRUCTIVE SORTING OF POTATOES

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ABSTRACT

The internal quality of potatoes and other vegetables and fruits is an important quality factor for both consumers and food industry. There are several diseases and defects, which have no effect to the quality of potato skin. Therefore internal defects remain invisible to human and also to ordinary camera.

V at V-camera stands for "virtually peeling". In this method and technology the potato, or other skinned fruit or vegetable, is virtually peeled by measuring the NIR transmission of potatoes and simultaneously measuring the NIR reflection from the potato skin.

INTRODUCTION

Problem status

It is well known that the quality of potato tubers intended to processing is determined by the quality status of the tubers flesh notwithstanding the quality of the peel which is to be removed. In case of surface defects due to diseases, pests, mechanical injuries, and soil pollution the machines so far known most frequently are sorting such tubers as defectives although their fleshy content is flawless.

The sorting machines based on the light reflected by the objects, i.e. based on their appearance only, can not detects that are hidden not only from man's sensory receptors but also from all kinds of instruments working with reflected light.

Therefore, because of this problem, the tubers having a flawless peel but with internal cavities, internal discolorations, vitreousness, and other kinds of internal defects are erroneously assessed and discarded by sorting as unfit for processing the sorting machines based on measuring the light transmitted by the objects (INAME AQS 602 et al) can defect both **external and internal defects** but without distinguishing between those type of defects. Therefore, tubers with flawless flesh but having defects on the skin due to Rhizoctonia mechanical injuries etc. are frequently assessed as defectives and discarded as unfit for processing. This leads to incomplete use of the raw material and losses to both the producers and processors.

METHOD AND MATERIALS

The new machine, here presented, has not the above mentioned drawbacks. It is based on a method patented by Prof. G. Krivoshiev in 1999 year BG Pat. 62304. In scientific community this method is known under the name "Seeing through layer" (STL). The author is declaring it as being a virtual method (V) because by means of optical and mathematical mans id predicted (measured) the internal spectrum of the fruits without they being peeled or sliced. This is equivalent to a virtual removing of the skin (peeling) and allows for defection of internal diseases and defects otherwise unseen by observation. Moreover, up to now such defects could not be detected reliably by spectral instruments as

well because the disturbing effect of the skin strongly prevented from measuring of the internal spectrum.

STL - method

The STL method is created in 1996 by Prof. Krivoshiev and it is originally called "the method for a virtual peeling of the skin" (V-method). The word "skin" in this case means the skin of potato tubers, which hinders to fulfil a precise assessment of fruit flesh quality. It is a method for a nondestructive measurement of the internal (of the fruit flesh) optical density by eliminating the disturbing skin spectrum using the diffuse reflectance. In the year 2000, D. Dham gives to the new method the name "Seeing Through Layers" (STL).

The method STL has built the foundation for the more sizable studies in the framework of the research project NIQAT financed by the European Commission

The nondestructive instrumental measurement of the internal optical density of potato tubers

The studies with potatoes are carried out in the course of three selection years by using 29 cultivars of potatoes, with proven origin, supplied from Bulgaria, Germany, Holland, England and Finland.

Acquisition of NIR data

It is used spectral computer system of Bentham Instruments Ltd, UK, to which has been integrated the developed by CANRI-Plovdiv, Bulgaria, photometric camera.

The camera allows the simultaneous measurement of the transmittance and reflectance spectra in both the visible and NIR regions by using two geometry patterns - T 0/180 and R0/45. Two variants were made: Option 1 - for smaller objects with dia up to 50 mm, and Option 2 - for objects with dia. up to 100 mm.

Statistical analyses

The software packages GRAM developed at ICFT Plovdiv, Bulgaria and The Unscrambler 6.11 version, of CAMO - Norway were used. The computation procedure essentially consists in determination of the coefficients of a MLR model with reselected architecture for each

wavelength (with a gap of 5 nm). The architecture of the MLR models was built according to the STL - method.

RESULTS AND DISCUSSION

The STL method is illustrated by fig. 1 wherein for one potato tuber, are shown: a spectrum of the whole intact tuber that of the physically peeled one, and the spectrum computed by means of the STL model. It is evident the nearness between the measured spectrum and computed one within the region from 600 to 940 nm.

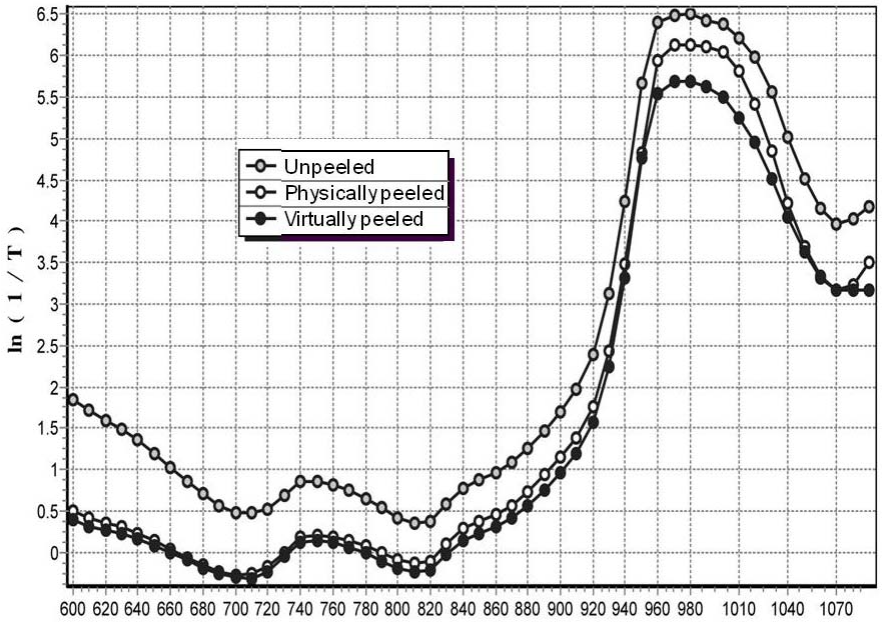
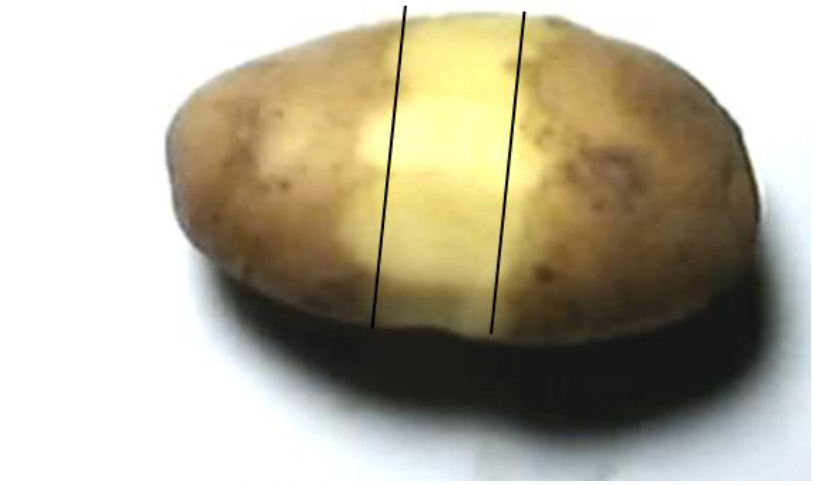


Figure 1.
Transmittance spectra of unpeeled, physically and virtually peeled potato tuber (variety BINTJE) with a skin injured by scab (Sample 257)

If only the optical density of the unpeeled tubers is used, i.e. if the STL-technology is not applied, is received quite inaccurate information for the internal optical density and from here for the internal quality of the tubers too.

If the difference between the two graphics of unpeeled and virtually tuber is bigger the quality and/or thickness of the peel (skin) is worse.
The bad quality of the peel stands (masks) the discovery of defects, especially of smaller internal defects.

Potato 146



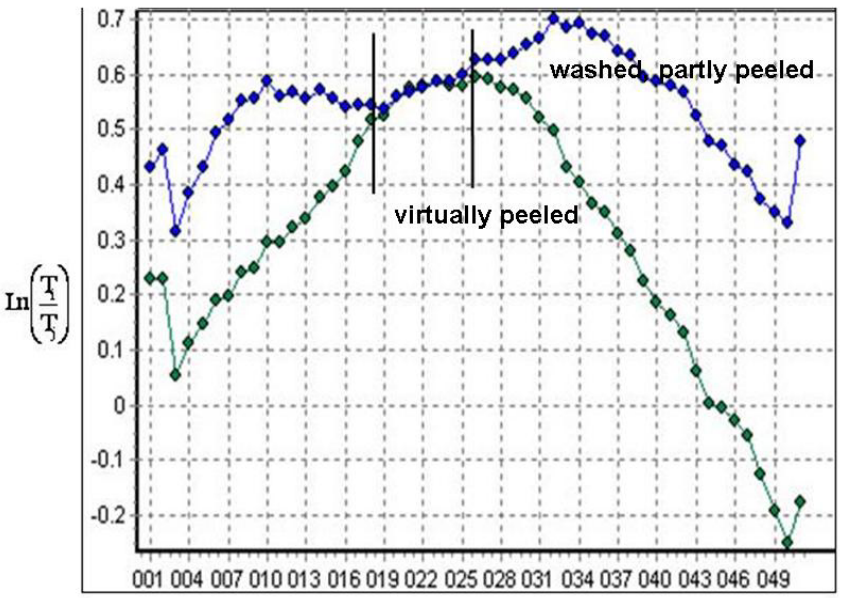


Figure 2.
Partly peeled potato tuber

The figure fig. 2 - Potato146 shows tuber with imperfect peel, caused by scabies but with flawless internal flesh. In the middle the tuber is physically peeled. Exactly on this place the graphics of the unpeeled and virtually peeled tuber coincide. This shows that the virtual peeling of the tuber with STL-technology is perfect and completely corresponds on the physical peeling in the middle of the tuber.

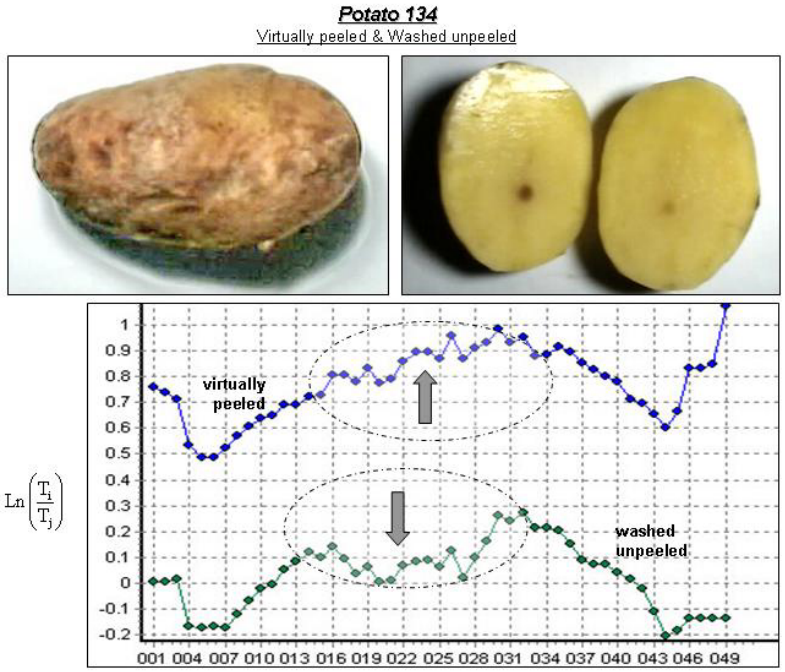


Figure 3.
The strongly defective peel masks the small internal defect.

The figure 134 shows that the strongly defective peel masks the small internal defect. Without STL-method its discovery is practically impossible with the well known Optical Sorters, working on the principal of reflectance and on the principal of transmittance. The great difference between the graphics of the virtually peeled tuber and of unpeeled tuber shows the possibility by STL-technology to be recognized the quality and the thickness of the peel: as the graphics are more near to each other so the peel is of high quality and thin; as the graphics are more far (remote)

so the peel is more damaged (presence of scabies, rezoctonium, wrinkled and etc.) and more thick.

CONCLUSION

For the first time the STL-method has been experimentally verified in on-line system for sorting of agricultural produce – potatoes.

Feasibility for on-line sorting of potato tubers

The spatial scanning is effected in DEMO-machine NIQAT01LAB in the process of tubers free falling with an initial speed 1.2 m/s and a slope of 15° toward horizontal line. This is a simple and efficient way to be secured the minimum admissible distance (≈10 mm) between the tubers at a velocity ≈ 2 m/s in the inspection zone. This operation mode ensures maximum rate of **sorting up to 40 tubers per second** or average capacity of **3.6 t/h**. Those parameters are fully corresponding to industrial practice. NIQAT01 makes classification into 6 classes - 3 by defectiveness + 1 waste and 2 – by the extent of greening. The software allows for combination between the separate classes, so that the number of sorted fraction should be from 2 to 6. Thus, the system NIQAT can easily be adapted to modern technologies for on-line sorting of potatoes intended to storage, retail trade, processing and sowing.

Perspectives

The experience from the created industrial prototype of the new machine and its commercialization would create possibility for the development of this machine for more precise sorting and of other agricultural products. These are products for which the peel does not allow important parameters of their internal quality to be reliably determined (the quality of the flesh), as example: the degree of maturity, internal color, chemical composition, internal structure and etc.

Potential objects for STL method application and the new technology are: mature onion, kiwi , avocado, citrus fruits, mellows, some apple varieties, peaches, pears and others.

Sorting of fruits and vegetables by using the new STL sorting system will bring to clear classifying by quality and a full correspondence between the price and the quality of the product. With these and other priorities the system NIQAT is competitive on the world market.

ACKNOWLEDGMENTS

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- The leadership of Agricultural Academy and CANRI-Plovdiv for prolonging the investigations with NIQAT01LAB and for its the popularization at domestic and international forums.

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NON - DESTRUCTIVE PHOTOSYNTHETIC DETERMINATION OF BEAN PLANTS (PHASEOLUS VULGARIS L.) RESPONSE TO SALT STRESS

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ABSTRACT

Using of non-destructive methods of chlorophyll fluorescence characteristics of photo system II (PSII) to determine the beginning of the salt stress in plants, were studied. The bean plants (cultivars Lody, Gina and Tara) were grown in pots as hydroponics cultures in a half-strength Hoagland nutrient solution. The plants were treated for 7 days with NaCl and Na_2SO_4 (concentration of 100 mM). It was found that salt stress increases initial (F_0) fluorescence and decreases maximal (F_m), and variable (F_v) fluorescence, as well as F_v/F_m parameter in dark adapted leaves. In light adapted leaves a significant decrease in quantum yield

(Y), photochemical quenching (qP) and electron transport rate (ETR) of PSII was occurred. In conclusion, it is considered that cv. Tara is more tolerant and cv. Lody is more sensitive to salt stress. The applied Na_2SO_4 caused stronger inhibition for all cultivars than those treated with NaCl .

INTRODUCTION

The application of non-destructive methods for the purpose of photosynthesis study is of particular importance as they provide information for the functional activity of the photosynthesis apparatus in native condition. The luminescence methods, including the fluorescence induction and the delayed fluorescence of FS2, possess the necessary characteristics to be applied for the purpose of photosynthesis study *in vivo*. They are non-destructive methods based on independent chlorophylls emission, and are characterized by high sensitivity and in formativeness with respect to the photosynthetic reactions. Besides that, they are easy to apply, fast, reliable, put into effect both in laboratory and natural conditions, and not lastly, they are considerably cost-effective.

Due to the functional connection of PS2 with the remaining components of the photosynthesis apparatus, the chlorophyll fluorescence is considered to be an indirect index for the condition of the integral photosynthetic processes in plants (Krause, 1991). At physiological temperatures the fluorescence, mainly, is being emitted by chlorophyll *a* of PS2 and reflects the photosynthesis primary processes (Zlatev et al., 2005).

Chlorophyll fluorescence measurements have been widely used to determine the plant response behaviour to environmental stress conditions (water, temperature and salinity stress, heavy metal stress etc.) (Glynn, 2003). Chlorophyll fluorescence yield, such as minimal chlorophyll fluorescence (Fo) and variable chlorophyll fluorescence Fv can be used for evidencing stress and damage of the photosynthetic apparatus, and characterizing the environment where plants grow in (Papageorgiou et al.,2005; Zlatev et al., 2005).

The salinity of soil is a widespread environmental problem and an important factor in limiting agricultural productivity. The high salinity levels of soil and irrigation water are known to affect many physiological and metabolic processes, leading to cell growth reduction (Gamma et al.,2007). The effect of excess salinity markedly decrease the growth and

transpiration rates (Massai et al., 2004), photosynthesis and pigment contents (Stoeva and Kaymakanova, 2007), affect seed germination, water deficit, cause ion imbalance of the cellular ions resulting in ion toxicity and osmotic stress (Khan et al.,2008).
The aim of the study was to determine the effects of salt stress on chlorophyll fluorescence parameters in intact leaves of three common bean cultivars – cv. Lody, cv. Gina and cv. Tara.

MATERIAL AND METHODS

Bean plants (*Phaseolus vulgaris* L.,) cultivars Lody, Gina and Tara, were grown in pots filled by half-strength Hoagland nutrient solution and grown in a growth chamber under controlled environmental conditions. The conditions, maintained during the experiments, were the following: light duration – 14 hours, light intensity (PAR) 250 μmol m^{-2} s^{-1}, temperature – 22 ± 2 ^{o}C and relative air humidity – 60 ± 5%. At the appearance of the first trifoliate leaf, an experimental design with three treatments was arranged for every cultivars:1- control – plants, supplied by ½ of Hoagland solution; 2-plants, supplied by ½ of Hoagland solution enriched with 100 mM NaCl; 3-plants, supplied by ½ of Hoagland solution enriched with 100 mM Na_2SO_4. The treatment of plants with salts continued for 7 days.
Chlorophyl fluorescence parameters were measured using a pulse amplitude modulation chlorophyll fluoremeter MINI-PAM (Walz, Effeltrich, Germany). Minimal fluorescence, Fo was measured in 60 min dark-adapted leaves using weak modulated light of < 0.15 μmol $m^{-2}s^{-1}$ and maximal fluorescence, Fm, was measured after 0.8 s saturating white light pulse (>5500 μmol $m^{2}s^{-1}$) in the same leaves. Maximal variable fluorescence (Fv=Fm-Fo) and the photochemical efficiency of FS2 (Fv/Fm) for dark adapted leaves were calculated. In light adapted leaves steady fluorescence yield (Y= (Fm' – F)/Fm'), maximal fluorescence (F'm) and minimal fluorescence (F'o) measured when actinic light was turned off, were determined. Photochemical (qP = (Fm' – F)/(Fm'-F0);) and non-photochemical (qN= (Fm – Fm')/(Fm – F0);) quenching parameters were calculated according to Schreiber et al., (1986), using the nomenclature of van Kooten and Snel (1990). The efficiency of electron transport as a measure of the total photochemical efficiency of PS2 (Y)

and rate of- electron transport (ETR) were calculated according to Genty et al. (1989).

Statistical analysis

Values are the mean ± SE from three consecutive experiments, each including at least five replication of each variant. In this study the results of second experiment was present. The Student's *t*-test was used to evaluate the differences between control and stressed plants.

RESULTS AND DISCUSSION

In order to characterize the functional activity of photosystem 2, the following types of fluorescence were determined - initial (zero) – Fo, maximum – Fm, and variable – Fv .

The results in Table 1 show that salt stress induced an increase in Fo in dark-adapted leaves of the studied cultivars between 20-30% above the control plants. Cultivar Tara was less affected. Fo reflects the condition of the antenna chlorophyll *a* and the energy loss in the PS2 reaction centres. Fo is changed in the case of structural changes in the pigment apparatus. Fm and Fv may indicate the increase in a non-photochemical quenching process at or close to the reaction centre (Baker and Horton, 1987).

Table 1

Parameters of chlorophyll fluorescence in dark adapted leaves of control and salt stressed bean plants.

		Fo	**Fm**
cv. Lody	Control	549±24	2115±47
	NaCl	623±21** (123)	1720±58 (81)
	Na_2SO_4	715±31** (130)	1620±66 * (76)
cv. Gina	Control	577±25	2185±51
	NaCl	625±19 (108)	1813±47 (82)
	Na_2SO_4	690±21* (119)	1618±62**(77)
cv. Tara	Control	560±28	2080±22
	NaCl	585±22 (105)	1872±48 (90)
	Na_2SO_4	620±19 (111)	1830±31 (88)

		Fv	Fv/Fm
cv. Lody	Control	1566±48	0.740±0.028
	NaCl	1117±29* (65)	0.0.645±0.031 (87)
	Na_2SO_4	975±51** (62)	0.601±0.030 (81)
cv. Gina	Control	1608±44	0.735±0.029
	NaCl	1188±22* (72)	0.655±0.032 (89)
	Na_2SO_4	992±51** (64)	0.589±0.019* (80)
cv. Tara	Control	1525±71	0.733±0.033
	NaCl	1287±44* (84)	0.687±0.040 (93)
	Na_2SO_4	1210±61 *(79)	0.661±0.037 (90)

* P<0.5; ** P<0.1;

The results in the same table show that with respect to NaCl the Fm values were reduced to a lesser degree, in contrast to Fv - 5-10% compared to the control. This reduction was more considerable in case of the second salt treatment (Na_2SO_4), where those values were from 20 % (cv. Gina), to 38% (cv. Tara). The value Fv, which is an index for the electron transport rate through the reactive center(RC) of PS2 is a sensitive indicator of changes in the ultrastructure of the thylakoide membranes, was characterized by a considerable decrease in the salt-treated plants.

The established during the study increase of Fo and decrease of Fm caused a reduction in the correlation Fv/Fm (Tbale 1) in the plants from the studied cultivars. The Fv/Fm ratio, which characterizes the maximum quantum yield of the primary photochemical reactions in dark-adapted leaves is an indicator for the potential functional activity and quantum effectiveness of the primary photochemical reactions of PS2 in the dark-adapted leaves (Ranjbarfordoei et al.,2006). The results in the table show that those reactions were inhibited to a greater extent in the leaves of the plants from cv. Lody and Tara, treated with Na_2SO_4 – 19-20%. The results from our study differed to a certain extent from those of Lu et al., 2003, conducted with halophyte *Suaeda salsa*. They suggest that PS2 activity is very resistance to salt stress in these plants, but in the sensitive rice cultivars the PS2 activity is sensitive.

That seems to indicate, to some extent, the occurrence of chronic photoinhibition due to photoinactivation of PS2 centers, possibly attributable to protein damage (Campos, 1998).
Fv/Fm reflects the maximal efficiency of excitation energy capture by "open" PS2 reaction centers. A decrease in this parameter indicates down regulation of photosynthesis or photoinhibition (Oquist et al., 1992). First trifoliate leaves showed a slight decrease in this parameter (Table 1). This is a result of a large proportion of absorbed light energy not being used by the plants in the photosynthesis process, as shown by the increase in qN (Table 2). Saline stress increased qN in plants and supporting the protective role of the non-photochemical quenching against photoinchibition (Tezara et al., 2003).
In all of the cultivars the occurrence of photoinchibition was further highlighted by the significant decline of quantum yield of electron transport (Y), which is a measure of the total photochemical efficiency of PS2 under photosynthetic steady-state conditions. Photochemical quenching (qP) presented a similar behavior to Y (Table 2). The results in table 2 show that Y was decreased to the greatest extent in the plants from cv. Gina, treated with Na_2SO_4 –52%. Despite the decreases in the photochemical efficiency of PS2, cv.

Table 2
Parameters of chlorophyll fluorescence in light adapted leaves of control and salt stressed bean plants.

		Y	**qP**
cv. Lody	Control	0.508±0.022	0.650±0.031
	NaCl	0.279±0.018** (55)	0.390±0.029 (60)
	Na_2SO_4	0.254±0.031** (50)	0.357±0.022**(50)
cv. Gina	Control	0.539±0.022	0.620±0.041
	NaCl	0.300±0.028** (56)	0.446±0.029 *(72)
	Na_2SO_4	0.258±0.033***(48)	0.421±0.021**(68)
cv. Tara	Control	0.489±0.025	0.635±0.044
	NaCl	0.332±0.021* (68)	0.539±0.022 (85)
	Na_2SO_4	0.293±0.019**(60)	0.476±0.028 **(75)

		qN	ETR
cv. Lody	Control	0.450±0.023	124.9±6.8
	NaCl	0.540±0.017* (120)	68.6±4.4**(55)
	Na_2SO_4	0.585±0.021**(130)	62.5±5.9**(50)
cv. Gina	Control	0.548±0.027	132.5±5.5
	NaCl	0.685±0.019* (125)	73.8±6.1 * (55)
	Na_2SO_4	0.723±0.011** (132)	63.5±4.4**(48)
cv. Tara	Control	0.575±0.021	120.0±4.8
	NaCl	0.661±0.028 (115)	81.9±4.2* (67)
	Na_2SO_4	0.678±0.033* (118)	72.0±5.0* (61)

* P<0.5; ** P<0.1; *** P<0.01

Tara presented highest qP and Y, as well as the lowest energy dissipation (qN) value. Cv. Lody showed stronger decrease in photosynthetic capacity under salt stress. In several cases qN was a much more sensitive indicator of stress response than qP (Ranjbarfordoei et al., 2006). In our study we presume that qP, as an indicator of salt stress, is much more sensitive than qN.
The reduction of Y is connected with the increase of the quenching of the rising energy in PS2 and usually is regarded as an index for negative metabolic regulation of the electron transport (ETR**)**. The considerable reduction of Y is an indicator of either of damages in the reaction centers of PS2 or of a slowly recovering quenching.

On the basis of the conducted studies the following conclusions can be drawn:

1. The photochemical activity of PS2 in young bean plants, subjected to salt stress, was reduced considerably compared to the controls in all of the three studied cultivars. Among the chlorophyll fluorescence parameters most informative of the functional condition of the plants' photosynthesis apparatus were the maximal photochemical activity of PS2 (Fv/Fm), the quantum yield of the electron transport (Y), and the electron transport rate (ETR).

2. The plants from cv. Tara proved to be more tolerant to the applied salt stress, in which Fv/Fm, Y and qP were reduced to a lesser extent.
3. The salt stress effect in bean plants can be assessed by means of the functional condition of the photosynthesis apparatus. In this respect, the following indices of chlorophyll fluorescence can be recommended: maximal photochemical activity of PS2 (Fv/Fm), actual quantum yield in light-adapted leaves (Y), and photochemical quenching (qP).
4. This investigation is significant in the aspect that photosynthesis is a good indicator of adaptation of plants to the environment. Since the measurement is a non-destructive, fast and reliable method, this makes chlorophyll fluorescence an attractive tool for environmental research purposes.

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CULTIVAR DIFFERENCES IN CD, ZN AND PB ACCUMULATION AND PRODUCTIVITY OF DURUM WHEAT PLANTS GROWN IN METAL CONTAMINATED SOILS

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ABSTRACT

A pot experiment has been conducted in "Poushkarov" Institute of Soil Science during 2007 to study the effects of metal contaminated soil on Cd, Zn and Pb plant accumulation, plant performance and grain productivity of three durum wheat cultivars. The contaminated soil was taken from the vicinity of the non-ferrous metal producing smelter near Plovdiv, while the control, noncontaminated soil having similar physical-chemical properties - from the experimental field of the Agricultural University of Plovdiv. Plant samples were taken and measured for heavy metal content in root, leaf, stem and grain during the vegetation period. Both chlorophyll content and leaf gas exchange parameters were determined in the flag leaf at the stage of grain filling. The grain productivity was established at harvest. The results showed that the wheat plants from all cultivars grew weaker in metal contaminated soil than the respective controls during the whole vegetation period. This corresponded to their decreased net photosynthetic rate, final shoot dry biomass accumulation as well as lower grain productivity. Grain Cd content was over the permissible limits in all durum wheat cultivars.

INTRODUCTION

Soil metal contamination is one of the most serious ecological problems all over the world. Metal contaminated land in Bulgaria is over 19 500 ha and the basic sources of contamination are mining and metal smelting industry (Grancharov and Popova, 2003). Generally, the risk associated

with metal contaminated soils is linked to (1) possible entrance of some heavy metals (Cd, Pb) into food chain, where they may provoke toxic effects on humans, animals, microorganisms and plants and (2) an ability of excess heavy metals to induce chronic phytotoxicity leading finally to a loss of crop productivity.

Among the problematic heavy metals, Cd attracts the highest concern as it can accumulate in human body over time from ingestion of food containing Cd leading to a risk of chronic toxicity with excessive intake (Grant et al., 2008). Cadmium rarely is a single soil pollutant; more often it accompanies Zn and Pb in soils contaminated by smelting industry. It is easily accumulated in plants due to its high mobility in soil-plant system, but hardly may induce phytotoxicity alone. Lead has limited solubility in soils and its availability for plants is minimal due to different processes involving complexation, sorption and precipitation. Nevertheless, Pb can negatively affect root physiology and therefore may contribute to development of chronic phytotoxicity (Sharma and Dubey, 2005). Zinc is essential element, readily taken up by plants. Grain Zn deficiency rather than grain Zn accumulation is an environmental problem, thus, Zn significance as a heavy metal is mostly related to its phytotoxicity at high tissue levels (Weckx and Clijsters, 1997).

The ability of different species to uptake heavy metals depends on many soil and plant factors. For example, the interactions between different heavy metals in the soil may enhance or limit plant uptake of a specific metal ion. Well known are Cd-Zn interactions during uptake, translocation and accumulation in plants (Siedleska, 1995). General consensus exists that some crops have ability to uptake higher levels of trace elements than others, although there is no correlation between tissue metal concentration and plant tolerance. Crop species and cultivars, for example, differ widely in their ability to absorb, accumulate and tolerate Cd. It is well accepted that durum wheat, sunflower, rice and some other crops have ability to accumulate Cd, frequently containing more than 0.1 mg Cd kg^{-1} dry grain matter (Harris and Taylor, 2001). This value was proposed by the international food standards organisation, Codex Alimentarius Commission, as an upper limit for cereals, pulses and legumes. Therefore, a significant effort is now in place towards different approaches to control food Cd levels, including (1) screening for low Cd-accumulating genotypes, (2) monitoring of Cd levels in crops grown near

metal contaminated soils as well as (3) searching for realistic solutions for soil Cd decontamination.
The heavy metal contamination is well recognized problem in Bulgaria. Different research programs aimed to contribute to the sustainable management of metal contaminated soils are underway. Our research program is focused on physiology of Cd uptake and performance of durum wheat plants at combined soil metal contamination. Here, we report our results on Cd, Zn and Pb accumulation and plant performance of 3 durum wheat cultivars grown in contaminated soil collected near the non-ferrous metal producing smelter in Plovdiv.

MATERIAL AND METHOD

Pot experiment has been conducted in "Poushcarov" Institute of Soil Science - Sofia during 2007 with 3 durum wheat cultivars grown in both noncontaminated and metal contaminated soils. The contaminated (Cd, Zn, Pb) soil was collected from the vicinity of the non-ferrous metal producing smelter near Plovdiv and noncontaminated soil with similar physical-chemical properties - from the experimental field of the Agricultural University of Plovdiv. Both soils had average organic matter content, high carbonate content and pH (H_20) between 7.5 and 7.8. The total content of Cd, Zn and Pb in the contaminated soil was – 9.5, 400 and 283 mg kg^{-1} soil, respectively. The experimental design included 6 treatments (3 cultivars X 2 soils) in 3 replications (pots, each filled with 5 kg of soil). After the emergence the number of plants per pot was reduced by thinning to 10. Plants were irrigated with tap water to constant pot weight during whole vegetation period. Leaf gas exchange of the flag leaf was measured by LCA-4 portable photosynthetic system at the stage of grain filling. During the same period leaf samples were taken and photosynthetic pigments content was measured spectrophotometrically. Plants were harvested at full maturity. Grain yield and Cd, Zn and Pb content in plant organs were measured by ICP spectrophotometry.

Table 1
Heavy metal content in plant organs of durum wheat cultivars grown in metal contaminated soil

Treatments		Heavy metal content in plant organs (mg kg^{-1} dry mass)		
Cultivars	Soil status	Heavy metal	Roots	Leaf
Vazhod	noncontaminated (control)	Cd	0.37	0.23
		Zn	46.0	19.6
		Pb	3.7	6.0
	contaminated	Cd	15.3	4.5
		Zn	305.0	171.5
		Pb	26.7	9.9
Yavor	noncontaminated (control)	Cd	0.36	0.20
		Zn	83.2	18.2
		Pb	3.9	5.8
	contaminated	Cd	12.4	5.3
		Zn	326.1	229.2
		Pb	22.4	9.27
Saturn 1	noncontaminated (control)	Cd	0.40	0.27
		Zn	73.3	27.3
		Pb	3.7	6.6
	contaminated	Cd	15.5	5.05
		Zn	338.0	242.1
		Pb	23.8	11.6

Treatments		Heavy metal content in plant organs (mg kg^{-1} dry mass)		
Cultivars	Soil status	Heavy metal	Stem	Grain
Vazhod	noncontaminated (control)	Cd	0.22	0.09
		Zn	7.7	21.9
		Pb	3.3	1.9
	contaminated	Cd	3.8	1.39
		Zn	308.2	64.5
		Pb	6.6	3.4
Yavor	noncontaminated (control)	Cd	0.18	0.07
		Zn	7.3	40.8
		Pb	3.3	0.9
	contaminated	Cd	3.5	2.21
		Zn	396.1	76.5
		Pb	5.9	1.5
Saturn 1	noncontaminated (control)	Cd	0.26	0.11
		Zn	4.0	38.7
		Pb	9.7	1.3
	contaminated	Cd	5.6	1.72
		Zn	308.3	61.5
		Pb	7.2	1.5

RESULTS AND DISCUSSION

According to the current Bulgarian regulation for soil quality, the contaminated soil used in our experiment exceeded the standard trigger values for Cd, Zn and Pb, which were respectively – 3.0, 300 and 120 mg kg^{-1} soil at pH > 7.4 (Atanassov, 2008).

Table 2
Photosynthetic rate, total chlorophyll content, plant dry biomass and grain productivity of durum wheat cultivars grown in metal contaminated soil. In brackets - % from the control.

Treatments		Parameters / measures	
Cultivars	Soil status	Photosynthetic rate (μmol CO_2 m^{-2} s^{-1})	Chlorophyll content (mg g^{-1} FW)
Vazhod	noncontaminated (control)	12.80 (100)	3.68 (100)
	Contaminated	10.78 (84)*	3.98 (108)
Yavor	noncontaminated (control)	12.04 (100)	4.14 (100)
	Contaminated	10.53 (87)*	3.85 (93)
Saturn 1	noncontaminated (control)	11.53 (100)	3.56 (100)
	Contaminated	10.40 (90)*	3.55 (100)

Treatments		Parameters / measures	
Cultivars	Soil status	Plant dry biomass (g)	Grain productivity (g pot^{-1})
Vazhod	noncontaminated (control)	3.17 (100)	11.12 (100)
	Contaminated	2.44 (77)*	6.62 (60)*
Yavor	noncontaminated (control)	3.00 (100)	9.59 (100)
	Contaminated	2.64 (87)*	5.80 (60)*
Saturn 1	noncontaminated (control)	2.83 (100)	10.35 (100)
	Contaminated	2.49 (88)*	7.09 (69)*

* - significant difference at P = 0.05.

The content of Cd in the soil was more than 3 and that of Pb more than 2-fold of the respective trigger values. Therefore, it was not a surprise that grain Cd content of wheat plants grown in the contaminated soil was more than the established upper limit for cereals – 0.1 mg Cd kg^{-1} (Table 1).

In fact, the plants from all used durum wheat cultivars contained near critical Cd grain level even in noncontaminated (control) soil. Cd content in the grain at contaminated soil was many times over the limit and varied from 1.39 mg Cd kg^{-1} for Vazhod to 2.21 for Yavor. As might be expected, the maximum Cd (12 – 15 mg Cd kg^{-1}) was observed in the roots, which first come in contact with the heavy metals. In plants Cd content gradually decreased from roots to leaves to stem to grain. Concerning Pb, it followed the same trend in all cultivars grown in the contaminated soils, except for the controls, where its root content was lower than that in leaves, probably due to some aerosol influx. Zn content in the grain of wheat plants grown in the noncontaminated soil varied between 21.9 and 40.8 mg Cd kg^{-1}, whereas at contaminated it was about twice higher. Leaf Zn content was close to the low range for cereals of 15 mg kg^{-1} in noncontaminated (Storey, 2007), but raised dramatically at contaminated treatments reaching values that might be phytotoxic (305 – 396 mg kg^{-1}).

Pb grain content was minimal and it could not possibly create a risk, but the detected high Cd level in the grain does not allow this grain to be used as a foodstuff. Concerning Zn, its high grain content is even preferable due to its generally low content in the grain in some crops. Unfortunately, in this case it was linked to high grain Cd content.

Bulgarian Ministry of Agriculture recommends an “adaptable agriculture” as a strategy for sustainable management of metal contaminated soils. This means that technical non food crops or cereals should be used for seed production. Our previous results showed that the Cd contamination in the grain did not affect seeding qualities, thus resulting in good yields with normal Cd levels when sowed in noncontaminated soil (Vassilev, 2002). Nevertheless, this option seems problematic for contaminated soils due to the induced chronic phytotoxicity problems. The wheat plants from all cultivars grew weaker in the metal contaminated soil than the respective controls during the whole vegetation period. The plants produced less tillers and had shorter height and spike length as well (data not shown). This corresponded to their disturbed photosynthetic

performance. The data in Table 2 showed that the plants grown at contaminated soils had their net photosynthetic rate diminished by 10 to 16%. It was not due to decrease in total chlorophyll content as reported in other studies (Vassilev et al., 1998a). Most probably the observed negative effect on photosynthesis is related to stomata limitation as a result of water relations disorders. The multiplication of the metal-induced negative effects on the cardinal physiological processes during the vegetation period resulted finally in a smaller dry biomass accumulation as was observed earlier in barley plants grown in Cd-contaminated soil (Vassilev et al., 1998b). Both shoot weight of treated plants and the grain productivity were significantly less - by 12 to 23% and by 31 to 40% from the respective controls.

In conclusion, the results obtained in this study demonstrated that durum wheat had an ability to accumulate Cd in unacceptable grain levels. Variability between the cultivars has been observed but at that Cd contamination level it was not significant in terms of potential human health risk. Combined contamination by Cd, Zn and Pb resulted in chronic phytotoxicity, most probably due to excess Zn accumulation. It was manifested by retarded plant growth and development and decreased net photosynthetic rate ultimately resulting in lower yield.

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INFLUENCE OF DIFFERENT INCUBATION TEMPERATURES ON GRAININESS AND ROUGHNESS OF STIRRED YOGHURT

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ABSTRACT

The aim of this work was to study how the incubation temperature and the storage time can affect the physicochemical characteristics, including graininess and roughness, of stirred yoghurt. Yoghurts were incubated at 37, 42 or 45°C. Physicochemical properties of stirred yoghurt were determined during storage at 4°C for 15 days. Visual roughness, number of grains, perimeter of grains, syneresis, storage modulus, and yield stress decreased, when the incubation temperature was decreased. The storage

time did not affect any of the physicochemical properties of yoghurt, except for the pH. For practical applications, incubation temperature can be optimized to improve quality or modified to create fermented milk products with different physical properties.

INTRODUCTION

Yoghurt represents a very significant dairy product around the world (Chandan, 2006). Although there is great interest in the healthy-promoting properties of yoghurt, texture of stirred yoghurt plays an important quality and consumer acceptance (Lee & Lucey, 2004; Lucey, 2004). Texture, one of the most essential components of the stirred yoghurt quality, represents all the rheological and structural attributes perceptible by means of mechanical, tactile and visual receptors (Sodini et al., 2004).

Textural defects of stirred yoghurt like graininess and roughness are objectionable as consumers expect smooth, uniform and fine-bodied products (Lucey et al., 1998). Graininess which sometimes occurs during the manufacture of stirred yoghurt is the appearance of non-dispersible particles (Tamime & Robinson, 1999). Roughness, irregular conformation of the surface structure of food, is an important physical property of solid food influencing sensory attributes (Pedreschi & Aguilera, 2000). The use of a high incubation temperature is often associated with these types of defects (Lucey & Singh, 1998; Lucey, 2004). Sodini et al. (2004) reported that the graininess of stirred yoghurt decreased, when yoghurt milk was incubated at 38°C instead of at 42°C. The objective of this research was to study the effect of incubation temperature on the physicochemical properties, including graininess and roughness, of stirred yoghurt.

MATERIAL AND METHODS

1. Milk processing and yoghurt preparation

Skim milk [13% (w/w) total solids] was prepared with low-heat skim milk powder [36.1% (w/w) total protein, BY 409 EG, Bayerische Milchindustrie eG, Landshut, Germany] dissolved in distilled water. The milk was left to hydrate for 2 h at ambient temperature while being continuously stirred. The standardized milk was heated at 95°C for 5 min

and, then, subsequently cooled to 37, 42 or 45°C in the tubular heating equipment (200 L h^{-1}) of the Dairy for Research and Training Department at the University of Hohenheim (ASEPTO-Therm UHT-Pilotanlage, Asepto GmbH, Dinkelscherben, Germany). After cooling, 0.1 g L^{-1} of frozen pellets (starter culture Yo-Mix 621, Danisco A/S, Denmark) was added, and the yoghurt milk was incubated at 37, 42 or 45°C until the pH had decreased to 4.60. Fermentation was stopped by rapidly cooling to 4°C in an ice-water bath. At the beginning of the cooling in an ice-water bath the yoghurt was manually stirred with a stainless-steel bored disk by up and down movements for almost 60 s. After setting the stirred product into 100 mL cups, the stirred yoghurt samples were stored at 4°C for 15 days. The physicochemical characteristics of the samples were analysed at days 1, and 15 of storage.

2. Physicochemical property measurements

2.1. pH and syneresis

The pH was determined by a Knick 765 pH meter (Knick Elektronische Messgeräte GmbH & Co., Germany), and syneresis of the yoghurt samples was measured using centrifugation method (Bhullar, Uddin & Shah, 2002).

2.2. Graininess and visual roughness

Graininess was measured by image analysis using the protocol described by Kücükcetin (2008). Image analysis was performed to determine the number of grains and boundary length (perimeter) of the grains as a measure for graininess. The number of grains indicating a perimeter greater than 1.0 mm per 3 ml of yoghurt and the mean perimeter of grains (PG) were evaluated. The mean absolute intensity deviation of each pixel from a median smoothed intensity of the picture was defined as visual roughness (R_{vis}) of the yoghurt sample. The measurement of R_{vis} was based on that described by Kücükcetin (2008).

2.3. Texture measurement

The textural properties of the samples were analyzed after 1-day and 14-day storage. Storage modulus (G') and yield stress were determined according to Baravian et al. (2002) and Steffe (1996), respectively.

3. Statistical evaluation

All statistical calculations were performed using SAS Statistical Software (release for Windows, SAS Institute Inc., USA).

RESULTS AND DISCUSSION

The pH at the end of the incubation period was similar for the different yoghurts with an average pH of 4.6, regardless of the incubation temperature. The pH values had decreased significantly ($p<0.05$) in each of the yoghurt samples after having been stored at 4°C for 15 days.
Table 1 shows the syneresis, number of grains, perimeter of grains, visual roughness, storage modulus and yield stress of the samples. At day 1, average syneresis of the yoghurt samples incubated at 45, 42 and 37°C was measured to be 76.2±0.9, 73.9±0.8 and 71.0±225%, respectively. Syneresis in yoghurt samples decreased as the incubation temperature decreased, which is in agreement with previous studies. Lucey, Munro, and Singh (1998) and Lee and Lucey (2004) reported that yoghurt produced at a lower incubation temperature showed lower syneresis.
The number of grains and the mean perimeter of grains of the day 1 yoghurt varied from 21 to 183 per 3 mL of the sample and from 2.1 to 3.4 mm, respectively, according to the incubation temperature. The number of grains in the yoghurt incubated at 45°C was higher than that of the yoghurt incubated at 37 or 42°C. The use of high incubation temperatures in cultured products promotes the formation of grains (Lucey, 2004). At a low fermentation temperature, the aggregation of proteins occurs more slowly, and a large number of protein-protein interactions between the casein particles takes place. Thereby, less rearrangement of the particles during gel formation occurs. This forces the formation of a more continuous network and contributes to an increase in the rigidity of the network. This may also explain why products obtained at low temperatures are smoother, as a lower extent of particle rearrangement would imply less graininess (Sodini et al., 2004). The visual roughness was influenced by the incubation temperature. The effect of storage was not significant ($P>0.05$). The visual roughness decreased significantly ($p<0.05$) as the incubation temperature was decreased.

Table 1
Physicochemical properties of stirred skim milk yoghurt

	Storage time (day)	37°C	42°C	45°C
Syneresis (%) (w/w)	1	71.0±2.2	73.9±0.8	76.2±0.9
	15	71.4±0.6	73.6±0.6	76.1±0.5
Number of grains 3 mL^{-1}	1	21±4	50±13	183±8
	15	22±2	54±6	179±13
PG[c] (mm)	1	2.1±0.1	2.9±0.1	3.3±0.3
	15	2.1±0.1	2.9±0.2	3.4±0.2
R_{vis} (unit of AID_m)[d]	1	1.4±0.1	2.6±0.1	5.4±0.3
	15	1.4±0.1	2.7±0.2	6.4±0.2
G' (Pa)	1	230±13	331±12	400±15
	15	225±8	330±21	406±8
Yield stress (Pa)	1	32±7	47±5	68±2
	15	34±8	45±5	69±5

[a] Values (means ± S.D.) of the rheological properties of stirred yoghurt

The storage modulus and yield stress are presented as rheological parameters of the stirred yoghurt samples in Table 1. The G' and the yield stress of the yoghurt varied from about 225 to 406 Pa and from 32 to about 69 Pa, respectively, according to the technological conditions. The G' values of the samples increased significantly (p<0.05) with incubation temperature; a similar trend was reported by Lankes, Ozer, and Robinson (1998), who showed that the number and distribution of the strong bonds increased with incubation temperature. These authors assumed that the stronger protein bonds contributed to the elastic character of viscoelastic gels. The yield stress of yoghurt samples incubated at 37°C was lower than those incubated at 42 or 45°C. The yield stress significantly (p<0.05) decreased as the incubation temperature decreased.

Conclusions

This study has shown that incubation temperature affects the physicochemical properties of stirred yoghurt. As the incubation temperature was decreased, the number of grains, perimeter of grains, visual roughness, syneresis, G' and yield stress decreased. For practical applications, incubation temperature can be optimized to improve quality

or modified to create fermented milk products with different physicochemical properties.

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COMPARISON OF DIFFERENT SEA BUCKTHORN BERRY VARIETIES ON THE BASIS OF PHYSICAL PROPERTIES

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ABSTRACT

Sea buckthorn (*Hippophae rhamnoides* L., *Elaeagnaceae*) as an edible berry has a long history of application as a food both in Asia and in Europe.
To describe the berries for different kind of purposes (transportation, processing) some chemical and physical analyses (the moisture content, dimensions and size distribution of the berries and also puncture resistance) were carried out.

INTRODUCTION

Sea buckthorn is a bush with berries from yellow to red in colour which has been used for centuries. These berries contain a large variety of substances especially those that are biologically active and have antioxidant properties.
In ancient Greece, sea buckthorn leaves added to horse fodder were well reputed to result in weight gain and shiny hair; thus, the Latin name "*Hippophae*" meaning shining horse.
Sea buckthorn occurs as a native plant distributed widely throughout temperate zones between 27° and 69° N latitude and 7° W and 122° E longitude including China, Mongolia, Russia, Great Britain, France, Denmark, Netherlands, Germany, Poland, Finland, Sweden, and Norway (Li and Schroeder, 1996).
During the last 10 years the cultivation of sea buckthorn in Estonia is turned more popular – there is over 500 ha sea buckthorn plantations. There are two research institutions in Estonia – the Experimental Station at Rõhu (experiments with sea buckthorn since 1998) and Polli Horticultural Research Centre in Estonian University of Life Sciences – who study the different growing conditions and crop yield. Mostly are growing in Estonia the sea buckthorn varieties, which are developed

under leading professor Trofimov in institute near the Botanic Garden of Moscow University.
The study on physical properties, such as size, weight and crushing strength of berries are required for the development of the grading system for berries.
The objective of this work was to give an overview and to compare different sea buckthorn varieties on the assumption of their physical characteristics in years 2005-2007.

MATERIALS AND METHODS

Materials

Sea buckthorn berries were harvested in seasons 2005, 2006 and 2007 from different cultivars grown in Estonia. The varieties with Russian origin were marked as AVR – Avgustinka, BOA – Botanicheskaja Aromatnaja, BOL – Botanicheskaja Ljubitelskaja, BOR – Botanicheskaja, HPR – Gibrid Pertchika, OTR – Otradnaja, PSR – Podarok Sadu, TRR - Trofimovskaja, VOR – Vorobjevskaja; and varieties with German origin were marked as ASK – Askola, DOR – Dorana, HER – Hergo and SIR – Sirola.

Methods

The moisture content was characterized using halogen moisture analyser HR83 (Mettler Toledo, Switzerland).
The geometric mean diameter (D_g) and the degree of sphericity (□) of the fruits were calculated according to Mohsenin (1970).
The mass of the berries was weighed by a chemical balance AB204 (Mettler Toledo, Switzerland).
The puncture resistance of sea buckthorn berries was characterized using a texture analyzer TA-XT2i (Stable Micro Systems, UK).
Some chemical analyses were also carried out. All the chemical and physical experiments were described by Lõugas (2006).

RESULTS AND DISCUSSION

The experiments were carried out with 13 sea buckthorn berry varieties, but unfortunately not with all varieties in all years. The first crop from German varieties was obtained in 2006, next year two varieties failed

according to hard winter. And also some Russian varieties were not covered for all the years.
The moisture content was measured in all species of sea buckthorn berries. The average values are reported and are presented in Table 1. The values for Russian varieties were in the range of 81.3-85.8 %, 79.7-83.0 % and 81.3-87.3 % in 2005, 2006 and 2007, respectively; 79.5-82.0 % and 81.0-82.4 % in 2006 and 2007, respectively for German varieties.

Table 1.
The moisture content (%) and the mass (g) of the berries

Berry variety	Moisture content, %			Mass, g		
	2005	**2006**	**2007**	**2005**	**2006**	**2007**
AVR	85.8	80.7	87.3	0.60	0.49	0.67
BOA			83.4			0.58
BOL	83.7	80.6	84.5	0.76	0.57	0.58
BOR		83.0	85.3		0.55	0.73
HPR		82.5	81.3		0.57	0.73
OTR	82.4	81.8	82.3	0.65	0.50	0.49
PSR	81.3	81.0	82.0	0.49	0.42	0.53
TRR	83.5	79.7	81.5	0.85	0.55	0.67
VOR	83.8	82.4	83.2	0.76	0.77	0.65
ASK		*79.5*			*0.26*	
DOR		*81.5*			*0.23*	
HER		*82.0*	*81.0*		*0.29*	*0.30*
SIR		*81.0*	*82.4*		*0.46*	*0.48*

Three principal dimensions, namely length, width and thickness, were measured using a micrometer. For Russian varieties the length of the berries was between 10.64-13.71 mm in 2005, 10.74-13.34 mm in 2006 and 9.88-12.46 mm in 2007; and the width of the berries was in the range of 7.79-9.03 mm in 2005, 8.10-9.70 mm in 2006 and 7.74-9.38 mm in 2007. The smallest berries belonged to variety PSR. For German varieties the length of the berries was 9.31-10.72 mm in 2006 and 8.45-10.51 mm in 2007; and the width of the berries was in the range of 6.45-8.44 mm in 2006 and 6.53-7.80 mm in 2007. The smallest and the biggest berries were in variety HER and SIR, respectively.

According to the formulas (Mohsenin, 1970) were calculated geometric mean diameter (Dg) and sphericity (Φ). With these parameters it is possible to evaluate the shape of the berries. The certain values are given in Table 2. It could be pointed out, that the berries with German origin were smaller than the berries with Russian origin.
Most spherical berries for Russian varieties in 2005 were in AVR variety; simultaneously most oval berries were in variety BOL, in 2006 also BOR and TRR and in 2007 BOR and BOL, respectively. As we can see from Table 2 the sphericity for two German varieties (HER and SIR) is quite average, but the rest are oval berries.

Table 2.
The geometric mean diameter (Dg, mm) and sphericity (Φ, %) of the berries

Berry variety	Dg,mm			Sphericity, %		
	2005	**2006**	**2007**	**2005**	**2006**	**2007**
AVR	9.33	9.46	9.21	86.7	81.2	79.4
BOA			8.80			81.5
BOL	10.05	9.74	9.29	73.3	80.4	76.6
BOR		9.86	10.17		87.1	85.0
HPR		9.90	9.95		81.3	80.0
OTR	9.59	9.45	8.74	79.0	81.3	82.5
PSR	8.64	8.88	8.42	81.2	82.7	85.3
TRR	10.36	9.63	9.61	76.0	79.4	80.1
VOR	9.65	10.77	8.83	77.1	80.8	76.8
ASK		*7.63*			*78.9*	
DOR		*7.32*			*77.4*	
HER		*7.84*	*7.11*		*84.3*	*84.3*
SIR		*9.13*	*8.61*		*85.3*	*82.1*

Also the mass of the berries is given in Table 1. In 2005 the mass of the berries is between 0.49-0.85 g, in 2006 0.42-0.77 g and in 2007 0.49-0.73 g. The German varieties had lower values of mass – for ASK, DOR and HER 0.23-0.30 g and for SIR 0.46-0.48 g, respectively.

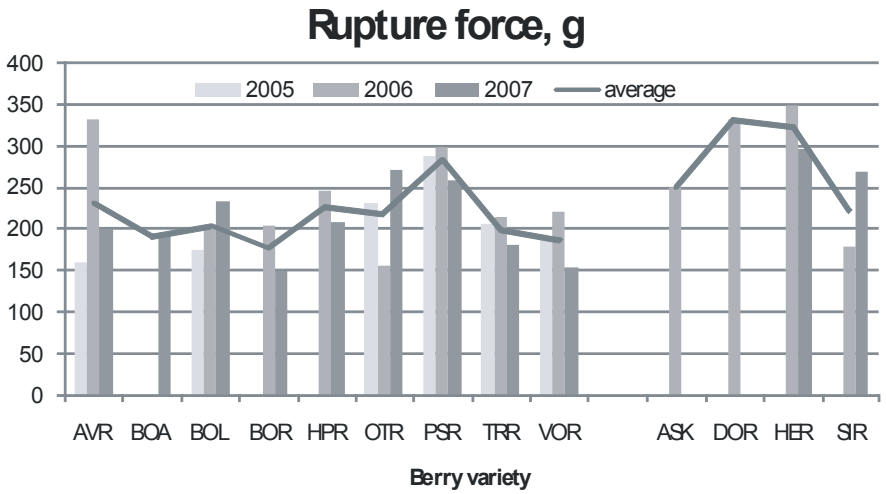

Figure 1.
The rupture force of the berries

The rupture force of sea buckthorn berry varieties is shown in Figure 1. Previous studies have been shown that the puncture resistance of the berries is influenced by freezing and defrosting (Lõugas, 2006). As all the characteristics of the berries are influenced by climatic and other conditions, we can see that the puncture resistance is not the exception. The summer 2006 was sunny and warm – the moisture content of the berries was little bit lower compared to other years, and the rupture force had due to that higher values. And also could be pointed out that smaller berries (PSR and German varieties) had higher rupture force.

Physical properties of berries vary with the species and in some extension also with years. As a result of present work we could not point out considerable differences between berry varieties – there were some tendencies, but it was not enough to make final conclusions. More experiments are needed for variety selection.

Among some chemical analyses we have been determined the content of vitamin C, β-carotene, reducing sugars, titratable acidity. Also all these values varied among the varieties and years. These results are presented in Figure 2.

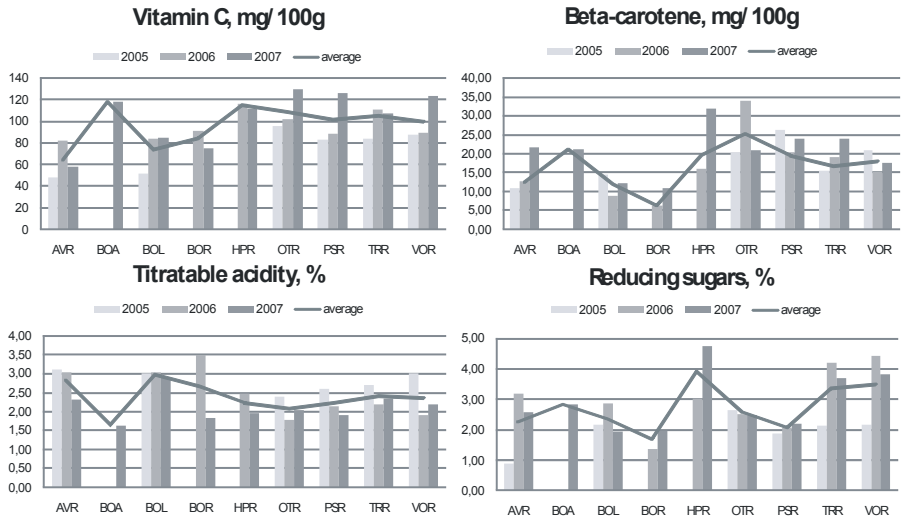

Figure 2.
Vitamin C, β-carotene, titratable acidity (expressed as malic acid) and reducing sugars content in sea buckthorn berry varieties.

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RHEOLOGICAL MEASUREMENTS FOR STANDARDIZATION OF VISCOSITY OF TEST BOLUS AND FOODS FOR PATIENTS SUFFERING FROM DYSPHAGIA

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ABSTRACT

Dysphagia is defined as difficulty in swallowing of food and liquids, caused by abnormalities of function of muscles and nerves as well as by damage of structures of the oral cavity, pharynx, larynx and oesophagus. Patients suffering from dysphagia often are unable to swallow food or liquids of certain physical/rheological properties. Viscosity is a fundamental rheological property both of the foods used for feeding, and the for test foods applied in endoscopic and X-ray swallowing studies. The aim of this study was to determine the viscosities of the test foods as well as those of foods used for feeding in order to compare and standardize them. We prepared an increasingly concentrated series of the test foods (jelly, pudding, puree, mush) by adding thickening substance (Resource Thicken Up® (Novartis) made of cornstarch) to water, and then determined the viscosities. We also measured the viscosities of commercially available foods, self-prepared foods of different thickness, and foods with known formulas. From these results we could identify the food formula that corresponded to the test bolus that could be swallowed by the patient without aspiration in the course of a video-endoscopic or X-ray swallowing study. The measurements were taken with a dynamic shear rheometer (UDS200).

INTRODUCTION

The oropharyngeal dysphagia is a disorder of the oral food preparation and its oral, pharyngeal and oesophageal transport. Various neurological and oncological diseases play role in its development. The most dangerous consequence of dysphagia is the aspiration, when the food/liquid gets into the trachea and pneumonia induced by the frequent aspiration. The object of dietetics is to set the adequate rheological properties, temperature, flavour and energy content of the food to be swallowed down safely (4,5,6). There are two methods used to follow up the process of swallowing and the path of the food bolus: Video-endoscopic swallowing studies, which can be used to determine with what swallowing technique, in what posture, and what consistency and amount of bolus can be swallowed by the patient without aspiration, plays an important role in the diagnostics of dysphagia (1,2). Similarly, in X-ray swallowing studies, we track the path of contrast materials of different consistencies and determine which consistency patient can swallow without difficulty (1). The oral feeding is only possible after test swallowing without aspiration.
In the clinical practice, the consistency of the food to be prescribed for the feeding of the patient was determined by such swallowing tests. The food jelly, pudding, puree or mush prepared individually and many times by different recipes always deviated from the test bolus causing uncertainties in the judgement of the results of the swallowing tests. For this reason, the standardisation of the viscosities of the test bolus, X-ray contrast medium and the foods themselves seemed to be necessary. When preparing the foods, the energy content, the presence of nutritive and the liquid content were considered.

MATERIALS AND METHODS

Steps of the procedure:
1. Standardization of the test food series or X-ray contrast medium according to their viscosity.
2. Assigning commonly consumed foods to these standard test food/contrast material series on the basis of matching viscosities.
3. Determining the nutrient, energy, and fluid contents of the foods for which we specified the viscosity, and including these data in the food

formulas to allow for their preparation either at the hospital or at home to specific standards.

From the list of rheological parameters of foods we chose the determination of viscosity since it best describes the property flow characteristic of materials concerning the swallowing process (3). Viscosity, or internal friction, describes an internal movement in flowing bodies (media), effected by the shear force (parallel to the direction of movement), and is a material constant depending on temperature and pressure. Its units are described as Pa.s. [1 centiPoise (1 cP)=1mPas] and it can be measured by a viscometer.

The measurements were performed using a dynamic shear rheometer (UDS200) and a PP50 plate/plate measurement system (Physica / Anton Paar GmbH., Graz, Austria). We considered it important to determine the viscosity of foods served both warm and cold, thus measurements were taken at 40^{o}C and at 20^{o}C or room temperature. In the course of the studies, 30 values were obtained for each material. Measurements were repeated three times (90 values). The duration of each measurement was the same (10 sec). The viscosity was plotted against the shear rate (flow curve). Test foods administered in video-endoscopic swallowing studies should be used immediately after preparation since the viscosity values may change over time (we observed this phenomenon in our measurements). This also applies to contrast media (Telebrix®) thickened with Resource Chicken Up® and used in X-ray swallowing studies. We prepared an increasingly concentrated series of the test foods (jelly, pudding, puree, mush in several variations of viscosity /3,3 g/ 100cm^3-5 g/ 100 cm^3 -6.6 g/ 100cm^3-7.5 g/ 100cm^3-10 g/100 cm^3-15 g/ 100cm^3-20 g/ 100cm^3/ by adding Resource Thicken Up® (Novartis) made of cornstarch to water.

Corresponding to everyday foods, we chose foods of gel, pudding, puree and mush consistency from hospital menus. Viscosities of a total of 5 cold and 15 warm foods were measured. The foods were prepared by qualified dieticians according to accurately specified (g, cm^3) food formulas.

Flow curves (rheograms) were prepared from the viscosity readings of the test foods, the X-ray contrast media, and the real foods. The viscosity was plotted against the shear rate on a decimal logarithmic graph. We matched the rheograms of the test food or X-ray contrast medium of various consistencies with the rheogram of the food with the closest values**.** The rheograms were created by the Microsoft Office Excel 2007 software.

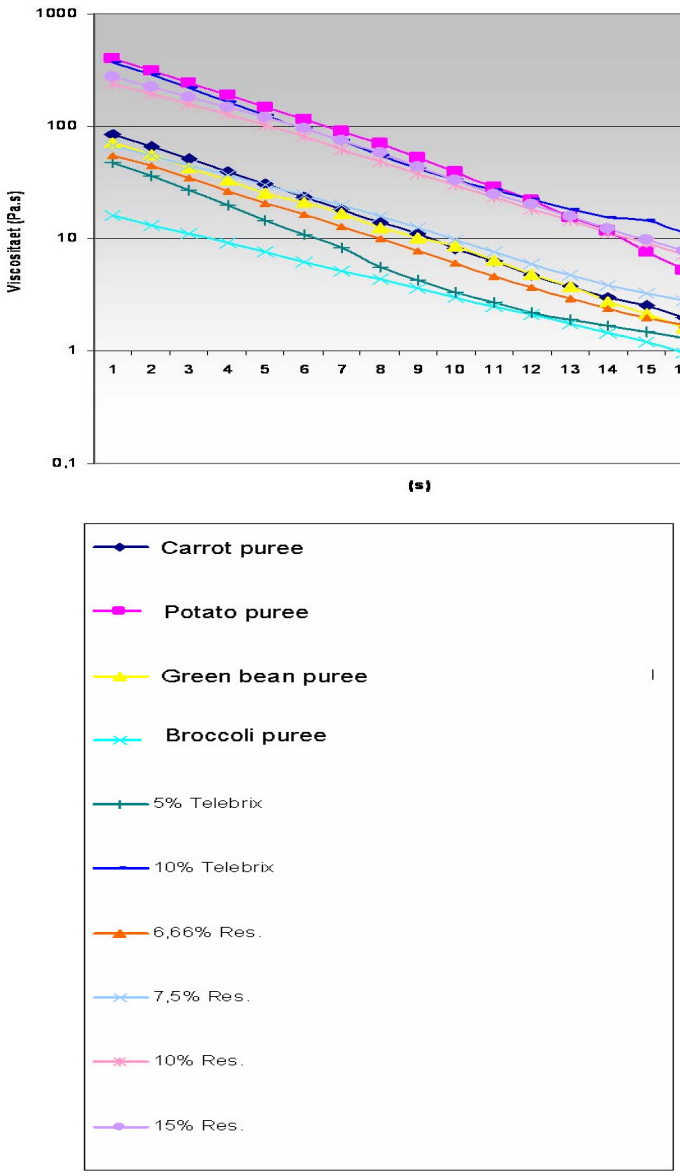

Figure 1
Rheograms (flow curves) of test food (6.6 g/ 100cm^3 -15 g/ 100cm^3), contrast medium (5 g/ 100cm^3-10 g/ 100cm^{3}s Telebrix) and vegetable puree (carrot, potato, green bean, broccoli)

Analysis and matching of the rheograms were performed by determining the minimal distance of each plot of the graphs to those of the other graphs using the comprehensive R Archive Network statistical software version 2.4.0.

RESULTS AND DISCUSSION

Based on comparative viscosity measurements, the foods used to the swallowing tests (endoscopy, swallowing X-ray test), the standardised bolus and the usual cold and warm foods were determined and assigned to each other.
Hereafter, the individual results of the measurements will not be detailed; instead, the viscosity curves of the test food Resource Thicken Up® (6.6 g/ 100cm^3 -15 g/ 100cm^3), Telebrix (5 g/ 100cm^3-10 g/ 100cm^3) and the vegetable purées of comparable viscosity will be shown.
In the left column of Table 1 the test foods (1a) and contrast medium (1b) are indicated. In the right side of the Table 1 are the real foods in the same row which correspond to the test foods and contrast medium.

CONCLUSION

Determining a diet, which beyond the appropriate fluid, energy and nutrient content, also involves such rheological properties that allow for oral feeding without aspiration, is a key element in the management of oropharyngeal dysphagia.
We also deal with food properties in a phase of swallowing therapy in order to find out what types of foods can be swallowed without aspiration, and contain the appropriate amount of energy, nutrients and fluid to provide an optimal continuous oral feeding regimen for the patient.
We specified and matched the standardized boluses used in swallowing studies with everyday cold and warm foods on the basis of comparative measurements of their viscosities. Using the same method of measurement, by the identity or very close similarity of the curves obtained, the real food to be assigned to the test food was specified.

Table 1
Comparison of the test foods and real foods (1a) and contrast medium and real foods (1b) on the basis of their viscosity

1a

Test food % Test food g/ 100cm^3 (20°C)	**Foods**
3.3 g/ 100cm^3	peach puree(40°C)
	Danone® kephir warm(40°C)
	Danone® kephir cold(20°C)
5 g/ 100 cm^3	broccoli puree, warm and cold(40°C and 20°C)
	fruit jelly warm Dini(40°C)
6.6 g/ 100cm^3	vegetable soup(40°C)
	green pea puree(40°C)
	peach jelly(20°C)
	Danette® pudding cold(20°C)
7.5 g/ 100cm^3	carrot puree (40°C), cold vanilla pudding(20°C)
10 g/100 cm^3	potato flakes+water+Nutridrink®+broccoli püre(40°C)
15 g/ 100cm^3	potato flakes+water(40°C)
	potato flakes+water+Nutridrink®(40°C)
	potato flakes+milk(40°C)
20 g/ 100cm^3	corn mush(40°C)
	potato purée+water(40°C)
	potato purée+milk(40°C)

1b

Contrast medium and Resource® g/ cm^3(20°C)	**Foods**
10 g/100 cm^3 Telebrix®	potato purée(40°C)
5 g/100 cm^3 Telebrix®	Danette® vanilla pudding warm, (40°C)Resource® 6,6 g/ 100cm^3 (20°C)

Our foregoing clinical experience shows a successful switch to oral feeding by serving foods prepared in this way with a known viscosity as well as caloric, nutrient and fluid content. Food formulas will be made available to hospital kitchen staffs and patients in order to widely ensure patients a proper oral feeding both at the hospital and at home. We intend to assess the results of feeding at hospitals and at home by conducting a questionnaire survey in the future. (The known caloric, nutrient and fluid content of formulas available upon request).

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ADVANCE METHODOLOGY FOR CONTROL OF CHEMICAL CONTAMINANTS IN FOOD

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In order to ensure the safety of food, it is necessary to consider all aspects of the food chain from the primary production through the harvesting and

storage to the processing and sales and supply of food to the consumer. The main contemporary tendencies in fast screening of food contaminants and residues are discussed. The main steps of laboratory analysis are mentioned. Besides laboratory available methods for precise and relevant analysis, some practical approaches are presented for early detection of contaminants as immunoassay in different formats. Their advantages and disadvantages comparing to the traditional instrumental methods are outlined.

Sources of chemical contamination of plant foodstuffs

Food is an essential ingredient to life, and access to food is often limiting factor in the size of a given population. Many substances are used to grow the quantity and quality of food needed the human population. Many of the agrochemicals are pesticides (e.g. herbicides, insecticides, fungicides, acaricides, fumigants) that may appear as residues in the food. Other type of agrochemicals that may appear as residues in animal-derived foods are veterinary drugs (e.g. antibiotics, growth promotants, and hormones). Different types of environmental contaminants (e.g. polyhalogenated hydrocarbons, polycyclic aromatic hydrocarbons, organometallics) can appear in food through their unintentional exposure to the food through air, soil, or water. Food may also be contaminated by toxins from various microorganisms, such as bacteria or fungi (e.g. mycotoxins) or natural toxins already present in the food or that arise from spoilage. Packing components (e.g. styrenes, phthalates) can also leach into food unintentionally. In addition, chemical preservatives and synthetic antioxidants may be added after harvest or during processing of the food to extend storage time or shelf-life of food products. Other chemical additives (such as dyes, emulsifiers, sweeteners, synthetic flavor compounds, and taste enhancer) may be added to the food to make it appear better to the consumer or to alter its taste or texture.

All these types of additives and contaminants are regulated by government agencies worldwide. The only way to know which chemicals and how much of them are in food is through chemical analysis. Without doubt, more than millions analyses of food contaminants and additives are conducted worldwide per year by industry, government, academic, and contract laboratories. It is necessary to increase their throughput by development of fast and reliable analytical methods.

Main aspects of analysis of chemical contaminants

Sample preparation for analysis of chemical contaminants in foodstuffs and foods consists of homogenization, extraction and clean up steps. Homogenization of sample is extremely important in some cases when contaminant is unequal distributed. A common example is mycotoxin analysis due to irregular distribution of fungi infection on the crops. This special feature requires strictly homogenization of high amount of sample and analysis of random portion of it.

The extraction procedure consists of separation of the analytes from the matrix and presentation of the material in a form that can be easily analyzed. The type of extraction step that is used for a particular matrix depends on the nature of the matrix and analytes. In some cases it is possible to extract many analytes by one solvent or solvent mixture (e.g. pesticides, phthalates), but in another case it is necessary to apply different extraction systems for analytes, because if their different structure and behavior (e.g. mycotoxins).

Currently, the common analytical approaches used for detection of chemicals in foods involve gas or liquid chromatography coupled to selective detectors (Hu et al., 2004; Stajnbaher D. et al., 2003). Due to co-extraction of many matrix components which lead to overlapping or masking of signal for analytes, matrix-matched standards should be used and confirmation of positive results is needed for official control purposes.

Nowadays, analytical methods in control laboratories are multi-class, multiresidue methods in order to detect a great number and wide range of contaminants (Anastassiades et al., 2003). In case of veterinary drugs, multiresidue methods are lacking and it is not unusual for single-methods to be used in monitoring programs. Many of common methods are time-consuming, laborious, and expensive, require careful safety precautions, generate hazardous waste, use a lot of glassware and lab space and lack of degree of sensitivity needed for some applications.

Due to necessity of regular analysis of high number of residues or early detection of contaminants, some screening tests are very promising as preliminary test. In case of positive result, it is necessary to perform additional analysis by conventional more precise analytical technique. Using this first step, the number of samples for analysis could be reduced and hazardous solvent waste as well.

Screening techniques for analysis of residues and contaminants

An immunoassay is a biochemical test that measures the concentration of a substance in a sample or sample extract, using the reaction of an antibody or antibodies to its antigen. In case of pesticide residues. immunoassay is particularly suited for polar, water-soluble pesticides and their degradation products that are generally difficult to analyze using conventional analytical methods. Comparisons of quantitative immunoassay with conventional single residue methods using gas or liquid chromatography to analyze specific pesticide/food commodities show that immunoassay can analyze four to five times as many sample in a given time period (Newsome et al., 1981; Newsome, 1985; Newsome, 1987). In addition, immunoassay can be simpler to use than conventional techniques, require less skilled personnel, minimum instrumentation time and comparatively inexpensive equipment. Some notable applications of ELISA give detection of particular herbicides in water and soil samples (Gabaldon et al., 1999; Wright et al., 1999). Nowadays, tests for pesticides are available mostly for herbicides belonging to triazine group - atrazine, cyanazine, but also test kits are developed for carbamates (carbofuran, aldicarb), acidic amides (alachlor, metolachlor), phenoxy acids (2,4-D), aldrin, paraquat ect.

Much more popular are immunoassay tests in mycotoxin analysis because usually different compounds are analyzed by single methods. Using immunoassays, it is possible to reduce time of analysis and to improve laboratory throughput. There are commercial test kits in different format - ELISA kits, strip kits, caps, immunoaffinity columns, available for all regulated mycotoxins. Some practical applications of immunoassays in mycotoxins analysis with limit of detection or cut-off characteristics are presented in Table 1.

Analysis of animal samples is facilitating by ELISA kits available for some veterinary drugs as chloramphenicol, nitrofurans, corticosteroids and hormones. Usually monitoring of human exposures to polycyclic aromatic hydrocarbons is performing by monitoring of its metabolite benzo[a]pyrene in blood and urine (biomarker). Polyclonal and monoclonal antibodies against its DNA adducts have been developed and used in radioimmunoassays or competitive ELISA assays in order to monitor human exposure to benzo[a]pyrene (Santanella, 1988; Santanella, 1990).

Table 1. Test kits availability and limit of detection for some regulated mycotoxins for screening

Mycotoxin	Immuno-affinity columns	Screening kits / cut-off	ELISA kits / limit of detection
Aflatoxins-total	+	+ / 4 ppb*	+ / 1 ppb
Aflatoxin B1	+	+ / 2 ppb	+ / 1 ppb
Aflatoxin M1	+	-	+ / 5 ppt**
Ochratoxin A	+	+ / 4 ppb	+ / 2 ppb
Deoxynivalenol	+	+ / 0.2 ppm***	+ / 0.2 ppm
Fumonisins total	+	-	+ / 0.2 ppm
T-2 toxin	+	-	+ / 35 ppb
Zearalenone	+	-	+ / 10ppb

* ppb – part per billion, ng/g; ** ppt – part per trillion, pg/g; *** ppm – part per million, μg/g

Despite above mentioned advantages, immunoassays may not be as sensitive for some compounds as conventional methods, and they can have lower levels of reproducibility. Because immunoassays are compound-specific they are not suitable for multi-residue analysis. In some cases food matrix requires considerable cleanup work, therefore immunoassay may be no faster than conventional techniques. In addition, for some analytes with small molecules or having non-rigid structure it may not possible to develop antibodies. There are still a lot of discussions regarding cross-reactivity of specific antibodies with other chemicals present in food.

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MULTICOMPONENT CHROMATOGRAPHIC METHODS FOR DETERMINATION OF PESTICIDE RESIDUES IN FOOD

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ABSTRACT

Different types of pesticides are widely applied to protect plants from disease, weeds and insect damages during plant growing and storage. Many of these compounds can remain as residues in foodstuffs after their application and therefore they can pose serious risk for the consumers. Hence adequate pesticide residue control intended to ensure the safety of foodstuffs is needed. A brief overview concerning the analysis of pesticide residues by chromatographic methods in samples of plant origin is presented. The basic principles and recent developments in the sample preparation (extraction and clean-up), detection and quantification are discussed. Comparison between traditional solid phase extraction techniques and so-called QuEChERS (quick, easy, cheap, effective, rugged, safe) approach is emphasized. Possibilities and limitations of single quadrupole mass spectrometer for quantitative determination are also discussed.

INTRODUCTION

Pesticides are used to protect crops before and after harvest from infestation by pests and plant diseases. Most of them are toxic compounds which pose hazard to human health and the environment. In order to protect consumers from exposure to unacceptable levels of pesticides European Commission has established maximum residue levels (MRLs) in food and feed (Regulation (EC) No 396/2005). Adequate control is necessary to ensure that residues found in foodstuffs are within the set limits. With a view to this requirement, a number of methods have been developed and applied routinely (Luke et al., 1975; Specht et al., 1995; Hernando et al., 2001; Vidal et al., 2006). The most efficient approach to

pesticide analysis involves the use of multiclass, multiresidue methods (MRMs) that allow simultaneous determination of wide range of compounds such as organochlorine, organophosphorus, carbamates, pyrethroids, triazoles and dicarboximides in one run. The varied physical and chemical properties of pesticides and the diversity of matrices in which the residues must be monitored make the development of MRMs a very complex task.

The determination of trace residues and contaminants in complex matrices, such as food, often requires extensive sample extraction and preparation prior to instrumental analysis (Tekel et al., 1996). Liquid-liquid extraction (LLE), solid phase extraction (SPE), solid phase microextraction (SPME), liquid phase microextraction (LPME), matrix solid phase disperstion (MSPD) and gel permeation chromatography (GPC) are some of the frequently used techniques for sample preparation. Many approaches are involved in this process but the purpose is one - effective extraction of the analytes and isolation of the residues from matrix components to reduce undesirable effects of co-extracted compounds such as lipids, waxes, pigments, sugars and carboxylic acids during chromatographic determination. Nowadays trend in the field of sample preparation methods for plant matrix is research for finding faster, less laborious, less harmful, cost-effective and rugged procedures, which preserve high recoveries and good precision, and are applicable for as wide as possible range of compounds. As a product of such research an important new extraction technique have been introduced by Anastassiades et al. in 2003 with the acronymic name QuEChERS, that reflects its major features and advantages (quick, easy, cheap, effective, rugged, safe) (Anastassiades et al., 2003).

Gas chromatography (GC), fast GC and high performance liquid chromatography (HPLC) combined with mass spectrometric (MS) or tandem mass spectrometric detector (MS/MS) are the current leading approaches in pesticide residue analysis today (Alder et al, 2006). Single quadrupole GC-MS provides less sensitivity and smaller number of analytes that can be determined at the same time than tandem detection systems but it is still the most available and within the financial capacity of the laboratories around the world.

This work describes validation of two methods for simultaneous determination by GC-MS of forty fungicides and insecticides that belong to various chemical classes (organochlorine, organophosphorus,

carbamates, pyrethroids, triazoles, dicarboximides, strobilurins etc.). The methods are based on different sample preparation techniques – SPE on polymeric sorbent and QuEChERS. The two approaches are compared by their performance characteristics and economic effectiveness.

MATERIALS AND METHODS

Reagents and materials

a) Solvents - Acetone, acetonitrile, ethyl acetate and methanol (gas chromatography grade) were purchased from Merck KGaA (Darmstadt, Germany).

b) Standards - Analytical standards of the pesticides with certified purity were obtained from Dr. Ehrenstorfer (Augsburg, Germany) and Riedel-de Haen (Seelze, Germany). Stock solutions of each pesticide were prepared in ethyl acetate or acetone at concentration ranged between 960-1420 µg/ml and were stored in a freezer at -18°C. Mixed standard solution of all target pesticide was prepared in acetone at concentration 10 µg/ml and was used for the preparation of working standards and fortification. Ethion (100 µg/ml in acetone) and Triphenylphosphate (20 µg/ml in acetonitrile) were used as internal standards for SPE and QuEChERS techniques, respectively.

c) SPE columns and bulk sorbent - Polymer-based sorbent LiChrolut® EN with surface area 1200 m^2/g and particle size 40–120 µm, 500 mg prepacked in 6ml cartridges was purchased from Merck KGaA (Darmstadt, Germany). Bulk primary secondary amine sorbent – Bondesil PSA, with particle size 40 µm was obtained from Varian (Varian Incorporated, Harbor City, USA)

d) Salts - Magnesium sulfate (anhydrous, grit) and disodium hydrogencitrate sesquihydrate obtained from Fluka, trisodium citrate dihydrate Riedel-de Haen (Seelze, Germany) and sodium chloride (Merck, Germany), all of p.a. grade.

Instrumentation

A Thermo Finnigan Trace GC Ultra (Thermo Finnigan, Milan, Italy) coupled to Trace DSQ MSD (Thermo Finnigan, Austin, Texas, USA), equipped with split/splitless injector and Varian FactorFour Capillary Column capillary column VF 5ms, 30 m × 0.25 mm ID × 0.25 µm film thickness was applied. A Thermo Finnigan AI3000 autoinjector (Thermo

Finnigan, Milan, Italy) was used to perform 1µl injections. Xcalibur 1.3 data system software was used for data acquisition and processing. The carrier gas He was of purity 99.9995% (SIAD, Bulgaria). Vortex mixer V-1 plus (Boeco, Germany) and centrifuge Rotofix 32A (Hettich GmbH&Co.KG, Germany) were used in the sample preparation process. Vacuum manifold Vac-Elut (Varian Inc., The Netherlands) coupled to vacuum pump 2012C (Welch Rietschle Thomas, USA) was used for SPE.

Extraction procedure

For SPE technique a previously described method was used (Stajnbaher et al., 2003) as well as for the QuEChERS (Anastassiades et al., 2003). Some changes were made in order to adjust and optimize both methodologies for the purpose of the present study. A full scheme of the procedures is shown in Figure 1.

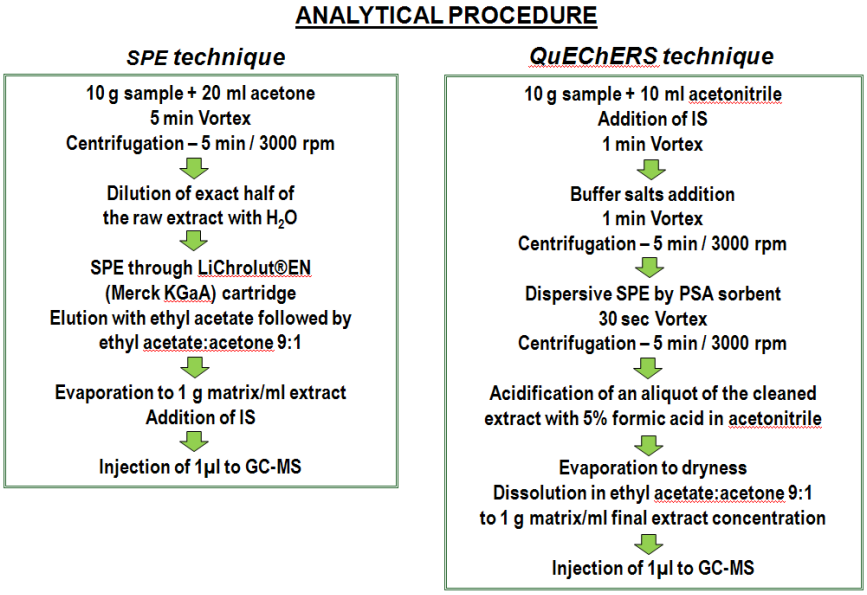

Figure 1.
Sample preparation procedure for the two techniques

RESULTS AND DISCUSSION

Method performance

Linearity was studied in the range 10-1000 μg/kg with five calibration points (10, 50, 100, 250 and 1000 μg/kg) by matrix-matched standards prepared by adding appropriate amounts of mixed standard solution of pesticides to blank peach extract. Correlation coefficient (R^2) was higher than 0.98 for both techniques and all the tested compounds. Limits of detection (LOD) and quantification (LOQ) were determined as the lowest injected concentration of the pesticides (in spiked peach extracts) that yield signal-to-noise ratios 3 and 10, respectively. For SPE technique calculated LOQ values ranged between 0.9 and 15.2 mg/kg and for the QuEChERS – 1.9 and 35.7 mg/kg. Blank peach samples were fortified with mix standard of target pesticides at three concentration levels – 10, 100 and 200 μg/kg in order to obtain recovery and precision data. Recoveries ranged between 79.5 and 104.0 mg/kg with RSDs from 4.5 to 23.1 % for SPE based method whereas for the QuEChERS method the recovery range was 56.0-109.8 mg/kg with RSDs from 3.5 to 25.3 %. Summarized results are presented in Table 1.

Table 1. Average recoveries and LOQs for the analyzed compounds obtained by two methods

Pesticide group	Recovery range %		LOQ [mg/kg]	
	SPE	QuEChERS	SPE	QuEChERS
Organophosphates	88.3 ÷ 96.0	82.0 ÷ 109.8	0.9 ÷ 10.7	2.9 ÷ 35.7
Organochlorine	79.5 ÷ 94.3	75.2 ÷ 93.1	6.7 ÷ 9.1	9.9 ÷ 14.8
Pyretroids	84.1 ÷ 104.0	56.0 ÷ 103.9	2.8 ÷ 14.0	1.9 ÷ 34.3
Triazoles	90.3 ÷ 101.5	94.7 ÷ 107.5	2.4 ÷ 15.2	3.7 ÷ 15.1
Carbamates	89.1 ÷ 93.7	85.2 ÷ 103.3	3.4 ÷ 7.1	4.0 ÷ 9.6
Dicarboximides	89.4 ÷ 95.3	67.4 ÷ 104.6	4.9 ÷ 8.3	6.1 ÷ 30.5
Strobilurins	92.2 ÷ 97	84.9 ÷ 106.7	3.3 ÷ 5.8	4.2 ÷ 7.6

Validation parameters were satisfactory for all target compounds when SPE technique was used. Some problematic pesticides could not be analyzed (captan, folpet and chlorothalonil) or were insufficiently recovered (deltamethrin) by QuEChERS but for the rest of the analytes this method provided good analytical results.

Economic effectiveness

Economic effectiveness of both procedures was assessed by the calculated price for individual sample taking into account putted consumables, time requirements for overall analysis and laboratory equipment needed. Traditional SPE produced more clean final extracts compared with the QuEChERS ones which is a key factor defying the frequency of system maintenance. Using SPE approach maximum 4 samples could be analyzed during 8-hour working day by single analyst at present laboratory conditions. The price of the consumables needed for individual sample was about 6 euros. By using QuEChERS procedure it was possible to analyze twice sample more and the costs per sample were 3 times less. It needs less expensive and simplified equipment. Therefore QuEChERS showed clear excellence with a view to economic effectiveness. Due to sufficient analytical performance and low cost QuEChERS method presents an attractive approach for routine applications in control laboratories performing monitoring programs.

ACKNOWLEDGEMENT

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EXPERIMENTAL AND NUMERICAL STUDY OF THE HENS EGG BEHAVIOR AT THE IMPACT

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ABSTRACT

Hens eggshell behavior at the impact by a circular rod is studied. The instrumentation of the rod enables to obtain time history of the force at the point of the bar impact. The velocity of the rod is gradually increased up to some critical value at which the eggshell failure starts. At the same time the surface displacement of the eggshell is also recorded. The numerical simulation of the egg behavior under this impact has also been performed. LS DYNA 3D finite element code has been used for the evaluation of the force and surface displacement at the points of their experimental detecting. The experimental results well agree with numerical ones. The elaborated computational procedure has been future used for the numerical simulation of the Hens eggshell behavior at the impact on a rigid plate. Qualitative features of the numerical simulation agree with results recorded using of the high speed camera.

INTRODUCTION

Eggs can be regarded as naturally packaged food. When examining the quality of the packaging, one primarily considers the strength of the eggshell. The eggs are exposed to many different kinds of loading occurring during their collection, within the sorting equipment, and during transport. There exist several techniques to determine the material strength of an eggshell see e.g. (Kemps et al., 2006).These experimental methods can be defined as quasi – static and dynamic. Most of works performed up to now have been performed under static loading. Owing to the fact that practice loads on the eggshell have a dynamic nature and so the dynamic strength of an eggshell could relate better to conditions experienced during handling and transportation of the eggs. As the measure of the shell strength the dynamic stiffness K_d was introduced

(Coucke, 1998).To determine K_d, the egg is excited by a small impact, and the vibration behavior is registered. Subsequently from the resonant frequency and the mass of the egg, the K_d is calculated.

In the given paper the method of the measurement of the dynamic strength of the eggshell is described. This met odd enables to measure not only vibration behavior but also the time history of the loading force. Preliminary experimental results together with the results of a numerical simulation are presented.

MATERIAL AND METHODS

For the experiments eggs of Rhode Islands hens co to bylo za vejce have been used. The eggs have been loaded by the impact of a free-falling cylindrical bar(6 mm in diameter, 200 mm in height – made from aluminium alloy) – see Fig. 1. At the point at the egg equator surface displacement as well as the surface velocity have been measured using of the laser-vibrometer. Eggs have been loaded by the bar impact on the pole (sharp or blunt) and on the equator. The height of the bar, *h*, has been changed up to the value at which eggshell fracture starts

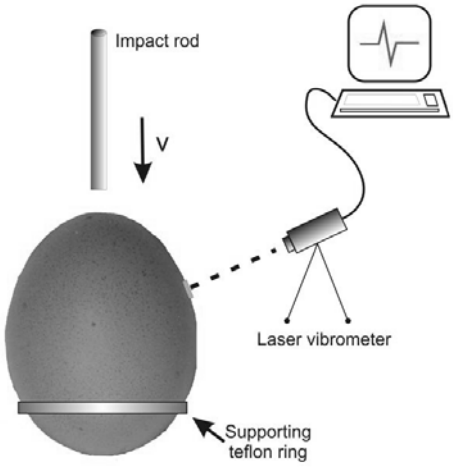

Figure 1.
Schematic of the experimental method.

RESULTS AND DISCUSSION

In the Fig. 2 the experimental record of the forces at the point of contact between bar and egg is presented. One can see that there is a deviation from the shape of this function at the moment of the fracture origin.

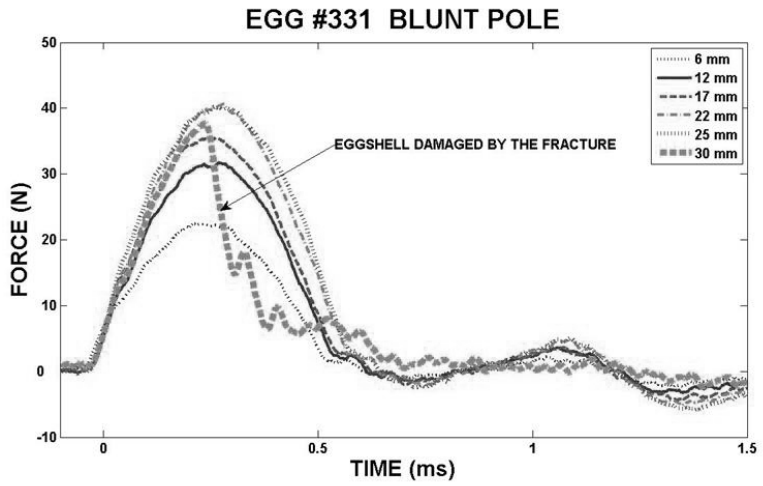

Figure 2.
Experimental records of the time history of the force at the bar impact. The different values of heights h are given in the upper right corner.

For each type of the dynamic loading (i.e. impact on the blunt pole, sharp pole and on the equator).The value of the force F_{max} at which the eggshell breaks has been evaluated. Results are shown in the Fig. 3.

Even if the number of the tested eggs is limited one can see that the values of the force at which the fracture starts is very closed to that obtained at the quasi-static loading e.g. by a compression between two plates. The effect of the loading orientation is also the same like at the quasi static loading. This conclusion support a hypothesis that the mechanical properties of the eggshell are independent on the loading rate and/or on the strain rate, respectively. This is different result from the conclusion of the paper, where this dependence has been reported. The next research and much more number of tests are needed for the explanation of the loading rate influence.

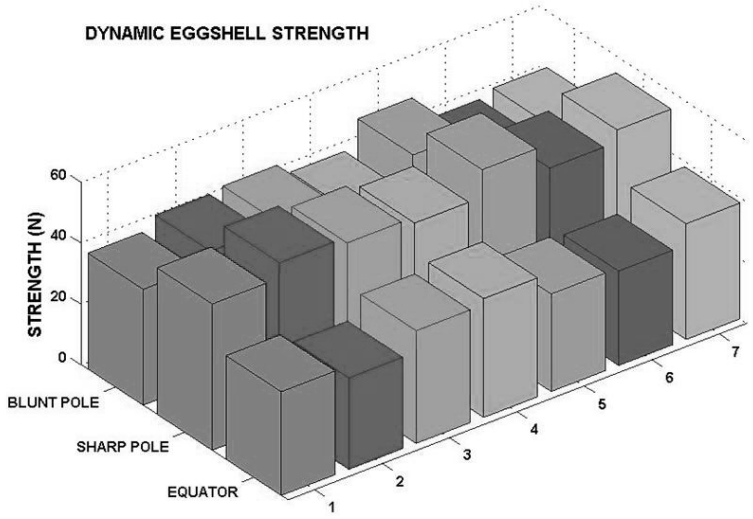

Figure 3.
Eggshell strength under dynamic loading by the falling bar.

Numerical Simulation.

Similarly as in our previous works the eggshell is considered as linear isotropic elastic material. Its behavior is than described by the Young modulus E and by the Poisson constant ν. The same description is used for the eggs membrane. Eggs liquids are considered as compressible. No other rheological model can be implemented into the LS DYNA software. In the Fig. 4 the comparison between the experimental recorded and computed force is displayed. The agreement is very good.

Numerical procedure has been used for the simulation of the egg falling on the rigid plate with the striking velocity 1.4 m/s. This experiment has been recorded by the high speed camera and than it was simulated using of the LS DYNA finite element code. The numerical and experimental records of the eggshell fracture development exhibited a good agreement. Numerical simulation can not describe the real flow of the eggs liquids after the eggshell break. This is a consequence of the neglecting of the real behavior of the eggs liquids.

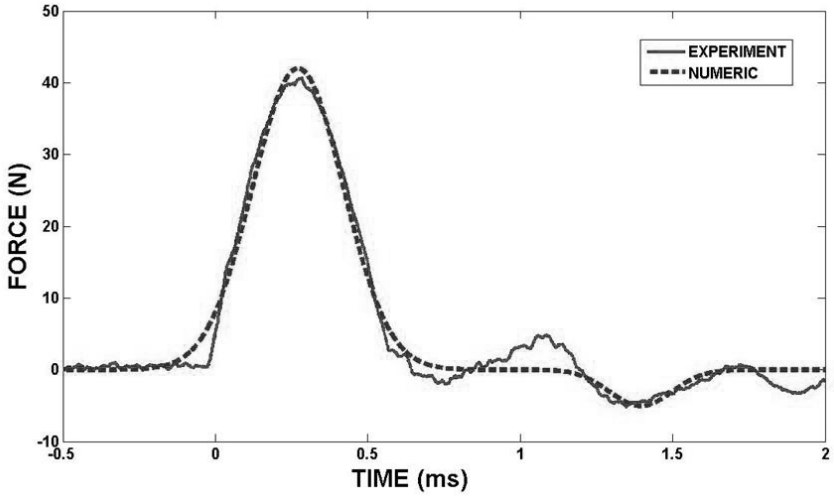

Figure 4.
Experimental and computational impact force.
(Impact on the blunt pole. Height h = 22 mm)

Concluding remarks.

In the given paper the experimental method of the eggshell dynamic strength evaluation has been described. This procedure extends the up to now used method of the evaluation of the dynamic behavior of the eggshell. Preliminary results show that the mechanical properties of the eggshell can be strain rate independent. The experimental method has been numerically simulated. The agreement between experimental and numerical results promise the evaluation of the stress state at the moment of the fracture origin. From the numerical computation the stress at which the eggshell fracture occurs can be obtained. This stress represents the eggshell strength. This strength is independent on the eggs shape as well as on the eggshell thickness. It seems that this strength is an intrinsic material parameter which may be affected by the eggshell microstructure, by its chemical composition and by some elements distribution.

ACKNOWLEDGEMENTS

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INVESTIGATION OF THE STABILITY OF COW BUTTER BY USING A LASER REFRACTOMETRY

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ABSTRACT

An investigation of the change in the refractive index of “Fibella” cow butter was carried out over time by using a laser refractometer. Samples

were stored at $(t = -18 \pm 2)^{\circ}C$ and were taken from butter on days 5, 30, 60, 120 and 150. Nonlinear regression models from type

$$n = n_0 + A.\exp\left(\frac{-t}{B}\right)$$

were obtained.

INTRODUCTION

The investigation of the stability of organic substances such as cow butter and margarine is a topical issue in that this factor is used in assessing the capacity of products to resist undesired changes (oxidation, color change, and other physical and chemical parameters) (Ivanov et al, 1984). Researches have reported that packing material and storage temperatures also have significant effects on the storage stability of butter (Bakirci et al, 2002). It has been established that long before the appearance of organoleptic symptoms of decay, the nutrient value of milk fat begins to deteriorate, because the first effect of oxygen affects their bioactive complex (linolic, linoleic, and archidonic fatty acids, liposoluble vitamins A, D, E, K, carotenoids, steroles, etc.) (Voznesonskiy and Levitskiy, 1979) Oxidative rancidity is the result of oxidation of free unsaturated fatty acids to hydroperoxidex, aldehydes, ketones, acids. The effects are caused by oxygen, heat, moisture and some elements (Tekinsen, 2000). In the process of storage of fats, along with these compounds, polymerization products are formed, too (Inichov, 1951). The issue of assessing the quality of butter is important because some of the substances formed during butter storage can prove to be harmful for the human health. Identifying such components is usually done chemically by slow and costly methods, such as liquid chromatography. Oxidation processes with all undesirable effects inevitably take place during warehouse storage of milk fats.

The aim of this work is to offer a fast, quick, efficient refractometric method for the assessment of the stability and freshness of milk fats without the use of chemical reactants.

MATERIALS AND METHODS

The refractive indices (RI) of "Fibella" cow butter were measured by using a laser refractometer. The product had a fat content 75 %, moisture content 16 % and it was produced by OMK, Plovdiv, Bulgaria. Samples were stored at temperature $t = (-18 \pm 2)^{\circ}C$ for five months.

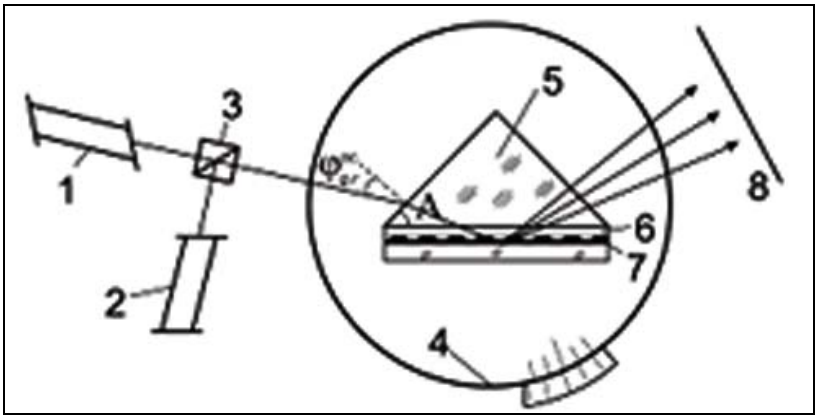
In order to measure the refractive index of cow butter without the involvement of chemical reactants, it is necessary to use a specially designed diffraction grating – a temperature indicator allowing simultaneous determination of the critical angle ($\varphi_{g,r}$), of the total internal reflection and the temperature of the sample. The diagram, the method of obtaining and action of the diffraction grating are set out in detail (Nikolova et al., 2007). It should also be emphasized that the liquid chromatography and some chemical methods are traditionally used for estimating the stability of vegetable and animal fats over time (Xanthopoulos et al, 1994, Duncan et al, 1991, Precht et al, 2001).

In this work we suggest a simpler refractometric method for assessing the freshness of "Fibella" cow butter by investigation of the dependence on the refractive index on storage time. For this purpose, a two-wavelength laser microrefractometer was constructed. The device is schematically shown in Figure 1. It has several main advantages: mechanical imperfections are minimized; temperature control during the measurements is avoided.

As a laser sources a He-Ne "Spectra-Physics" 0.5mW and 0.1mW pointer are used, generating at 632.8 nm and 532 nm, respectively. To get a clear diffraction picture we must make a very thin layer $(5\mu m)$ of butter or margarine. The thickness measurements are performed using a "Mitutoyo Digimatic Micrometer" with uncertainty $\pm 1\ \mu m$. The corresponding RI is calculated by the following relation (Sainov, 1991):

$$n_{g,r} = N_{g,r} \sin\left[A \pm \sin^{-1}\left(\frac{\sin \varphi_{g,r}}{N_{g,r}} \right) \right], \qquad (1)$$

where symbols "g" and "r" are for 532 nm and 632.8 nm wavelength, respectively; A is the prism (5) refracting angle, in our case – 65deg. Prism's RI are N_g = 1.748 and N_r = 1.735, made by heavy flint-glass TF-4.



1 – He-Ne laser; 2-Laser pointer; 3-beam splitter; 4- goniometer;
5- heavy flint-glass prism; 6-sample; 7- metal grating; 8-screen.
Figure 1
Principle scheme of laser refractometer

The experimental uncertainty mainly depends on the angular resolution $\Delta\varphi$ of used goniometric table - "Microcontrole" TR 80 : $\Delta\varphi^o$ = 1arcmin ~ 3.10^{-4}. After differentiation of the relation (1), it easy to show that for small critical angles in the air (that is the case in the present work), we have as a good approximation: $\Delta n_{g,r} \approx \cos A.\Delta\varphi \leq \cos 60^o.\Delta\varphi = 2.10^{-4}$.

Statistical Analysis:
Each refractive index value is the mean of eight different measurements. The mean square error was calculated for the average result at confidence probability p=0.95.
For each temperature value 25°C, 30°C, 32°C, 35°C for the "Fibella" cow butter, nonlinear regression models were obtained describing the dependence of the refraction index of the butter under study on storage time. The parameters of the models were obtained and estimated by using the Origin 6.0 software. The suitability of the models was evaluated and compared using the coefficient of determination (R^2), room mean square error (RMSE), reduced chi-square (χ^2)). A model was considered to be good when the coefficient of determination (R^2) was high, RMSE was low

(Doymaz, 2004), and the reduced chi- square $\left(\chi^2\right)$ was also low (Doymaz, 2004).

RESULTS AND DISCUSSION

This work investigated the behaviour of the RI of the "Fibella" cow butter over a period of 5 months. The samples were taken from a newly-opened product packed in an aluminum foil on days 5, 30, 60, 120 and 150. Three parallel measurings of each value were performed, and the average results are presented in Table 1.

RI of the investigated cow butter diminished with time. This showed that a change began in the product structure as a result of oxidation processes occurring in it. The fact that the RI did not grow in the said period confirmed Inihov's findings (Inichov, 1951) that no aldehides or ketones were present in the cow butter under study. Therefore, the measurements of the RI of cow butter at certain time periods can be used as a criterion to assess its freshness. For that purpose, based on the data in Table 1, mathematical models were obtained describing the dependence of the RI of "Fibella" cow butter on storage time for two wavelengths $\left(\lambda_1 = 532nm, \lambda_2 = 632.8nm\right)$ using the Origin 6.0 software.

Table 1
Refractometric data for "Fibella" cow butter

Refractive index -n					
5 days		30 days		60 days	
532nm	632.8nm	532nm	632.8nm	532nm	632.8nm
1.4679	1.4619	1.4650	1.4586	1.4629	1.4552
1.4603	1.4570	1.4592	1.4532	1.4582	1.4503
1.4582	1.4555	1.4570	1.4520	1.4562	1.4492
1.4560	1.4492	1.4549	1.4473	1.4535	1.4460
1.4544	1.4397	1.4534	1.4393	1.4522	1.4389

Refractive index -n			
120 days		150 days	
532nm	632.8nm	532nm	632.8nm
1.4601	1.4531	1.4589	1.4525
1.4564	1.4488	1.4557	1.4486
1.4546	1.4461	1.4542	1.4458
1.4523	1.4446	1.4520	1.4444
1.4501	1.4381	1.4492	1.4377

A nonlinear regression model of the type $n = n_0 + A.\exp\left(\frac{-t}{B}\right)$ is not mentioned at 40^0C, because it has a relatively low significance factor. The latter can be attributed to the fact that a temperature of 40^0C is relatively hard to maintain to within 0.2^0C. Hence the larger mean standard deviations in Table 1 in measuring the RI at $t = 40^\circ C$.
The models were fitted to the obtained experimental data and the estimated parameters of the models are listed in Table 2.
From Table 2 it is evident that, high R^2, low RMSE and χ^2 for the models at 30°C and 35°C for green and red wavelengths, respectively, lead to their acceptance. The presented models can be used for assessing the freshness of the "Fibella " cow butter if the refractive indices are measured at one of the indicated wavelengths $\left(\lambda_1 = 532\,\text{nm}, \lambda_2 = 632.8\text{nm}\right)$ and temperatures of 30^0C and 35 ^{0}C, respectively.

CONCLUSIONS

A refractometric method is proposed allowing the assessment of the freshness and stability of milk fat in a prompt, easy way without the use of chemical reactants. The cow butter under study, stored at low

temperatures, changed its RI by the expiry date relatively slowly due to the oxidation processes taking place. No increase in the RI was observed over the that period which showed that despite the oxidation processes taking place, there were no substances harmful for the human health, such as aldehydes, ketones, and free fatty acids.This method can be improved in the future and can be applied in the assessment of plant oils and lard.

ACKNOWLEDGEMENTS

The authors express their thankfulness to the Ministry of Education and Science for the financial support by Contract № У- АН- 06/04

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Table 2
Estimated parameters and criteria for the comparison of the presented models for four temperatures

Models	$\lambda = 532nm$				$\lambda = 632.8nm$			
	(25±0.2)°C	(30±0.2)°C	(32±0.2)°C	(35±0.2)°C	(25±0.2)°C	(30±0.2)°C	(32±0.2)°C	(35±0.2)°C
Coefficients								
A	**0.0117** $\pm 7\times10^{-4}$	**0.0094** $\pm 9\times10^{-4}$	**0.0060** $\pm 8\times10^{-4}$	**0.0052** $\pm 4\times10^{-4}$	**0.0112** $\pm 5\times10^{-4}$	**0.0099** $\pm 3\times10^{-4}$	**0.0119** $\pm 4\times10^{-4}$	**0.0057** $\pm 9\times10^{-5}$
B	**92.2** ±14.2	**209.5** ±28.5	**124** ±30.8	**79.3** ±15.7	**55.1** ±8.1	**40.1** ±3.5	**63.8** ±5.8	**57** ±9
n_0	**1.4567** $\pm 8\times10^{-4}$	**1.4511** $\pm 9\times10^{-4}$	**1.4524** $\pm 8\times10^{-4}$	**1.4512** $\pm 4\times10^{-4}$	**1.4518** $\pm 5\times10^{-4}$	**1.4483** $\pm 2\times10^{-4}$	**1.4445** $\pm 4\times10^{-4}$	**1.4440** $\pm 1\times10^{-4}$
Comparison criteria								
R^2	**0.998**	**0.9999**	**0.997**	**0.996**	**0.996**	**0.998**	**0.999**	**0.9999**
RMSE	$\pm 1.4\times10^{-4}$	$\pm 6\times10^{-5}$	$\pm 7.7\times10^{-5}$	$\pm 1.5\times10^{-4}$	$\pm 2.3\times10^{-4}$	$\pm 1.7\times10^{-4}$	$\pm 1.3\times10^{-4}$	$\pm 6\times10^{-5}$
χ^2	5×10^{-8}	1×10^{-8}	1.5×10^{-8}	5.5×10^{-8}	1.4×10^{-7}	7.5×10^{-8}	4×10^{-8}	1×10^{-8}

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THE TECHNIQUES USED FOR INFORMATION AND EDUCATION OF CONSUMERS SUFFERING FROM FOOD ALLERGY AND INTOLERANCE IN HUNGARY

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ABSTRACT

In Hungary, there are different informational and educational techniques for sensitive consumers, especially; the information channels “from product to patient” are various. The current labelling laws are not sufficient for consumers suffering from food allergy and food intolerance, so we have to provide the information in other ways about “free from” products.
The aim of our study was to evaluate existing management strategies of food allergy in Hungary, especially, the function of the current allergen labelling and the Hungarian Food Allergy and Food Intolerance Databank. The further aim of the research is essential to the patients suffering from the food allergy/intolerance best service opportunities, methods, finding intervention points or strategies on Hungary judged from more viewpoints for best one concerned. We analysed the function

of the Hungarian Food Allergy and Food Intolerance Databank and used a special interview method.
The Databank completes the labelling laws. It is an alternative way for consumers suffering from food allergy and food intolerance to know the information about “free from” product. The Databank has published the ”free from” product list since the year 2000. This consists of an electronic data basis containing 9 categories.
The food safety includes the allergen information as well. The allergen information as consumers' information on the labelling are not sufficient and they do not replace the dietician counselling and patient management, despite this very important the full and reliable labelling, that is on the tag character the reliability of an allergen statement.

INTRODUCTION

The prevalence of food allergy/intolerance has been estimated to be around 1-3% in adults and 4-6% in children in Europe (EFSA, 2004). Nowadays in Hungary the prevalence of food allergy is 1% in adults and 2-9% in 0-3 years old children (Hídvégi, 2000).
The representatives of the medical science put efforts into getting the allergy under control, the treatment of the symptoms joining forces with other specialists and the governments' representatives, but the illness cannot be healed definitively.
The treatment of the food allergy/intolerance is the lifelong allergen elimination diet that should be feasible only with appropriate information about allergen content and dietetic management (Pálfi, 2006).
The adequately safe allergen free nutrition makes it difficult, that the allergen, and the appearance of the allergic symptoms most diverse (Ortolani et al., 1999).
It belongs to food safety the patients suffering food allergy/intolerance allergen to information the fulfilment of his jute claim.
Although it is allergic/intolerance information intended as consumers' information on the labelling not enough and the dietician counsel, patient management are not replaced, very important the full and reliable claim of the foods, that is the reliability of the labelling.
The European Parliament and Council 2003/89 (2000/13 modification) a governing principle being about a labelling lifts some from among the allergen in connection with the indication of the food components, but

this not sole information device for the allergic ones. Lifted allergens the cow's milk (milk protein and lactose), Soybean, peanut and nuts, egg, gluten and other cereals, fish and crustaceans, celery, the additives the sulphides, azocolours and benzoic.
The European Union the labelling of the allergen is regulated in the national laws, orders without. In Hungary the 19/2004. Band order, and its modification (40/2008) from the nomination of the foods grants it in detail, the labelling of what kind of allergen components obligatory.
In the full process of the food production may become contamination the food with the allergen. It is wrote on the packaging in single cases that a product with nuts content is manufactured, for example on the production line in the firm. This may be help as much for the consumer suffering allergy, than disadvantage.
The manufacturer gives an over caution in single cases information of allergen, and it tightens it unnecessarily hereby his allergic diet. We may face the labelling concerning the food intolerance, for example “lactose free milk”.

MATERIAL AND METHOD

The aim of our study was to evaluate existing management strategies of food allergy in Hungary, especially, the function of the current allergen labelling and the Hungarian Food Allergy and Food Intolerance Databank. The further aim of the research is essential to the patients suffering from the food allergy/intolerance best service opportunities, methods, finding intervention points or strategies on Hungary judged from more viewpoints for best one concerned.
We analysed data from the registered food companies, products and the patients suffering from food allergy/intolerance in the Hungarian Databank and we used a special interview technique.
The interview the aspects of a method differing for the treatment of the food allergy and food intolerance the single specialities (e.g. public health, food industry, consumer protection, patient organisations) from his viewpoint more survey it comprehensively based on a criterion (Millstone, 2007). The interview subjects from the next areas we selected it: food industry regulation, food industry, food trade, consumer protection, public catering, public health, patient organisations, and media. The interview consists of three parts being built each other, that

the successors: the definition of the options and the identification of the criteria, the score, the weights assigning. The interview ahead defined statements, so-called options and the viewpoint of their evaluation, that is implies criteria. The stakeholder in writing about the particular options, the form of commentaries, concerned score-system forms an opinion on his basis. The stakeholder may refine it with weighting the developed opinion.

RESULTS AND DISCUSSION

Currently the food law and the free from products' databank do not concern the public catering and the catering industry.
The current labelling laws are not sufficient for consumers suffering from food allergy/intolerance, so we have to provide the information in other ways about "free from" products. In Hungary, there are different informational and educational techniques for sensitive consumers, especially; the information channels "from product to patient" are various.

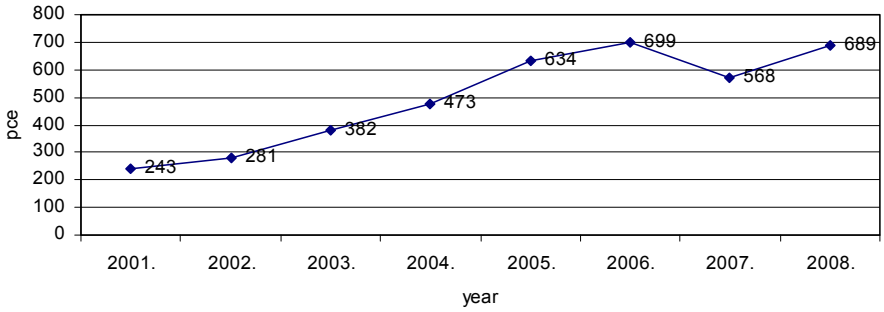

Figure 1
The number on the registered food
in Hungarian Food Allergy and Food Intolerance Databank
(pce/year)

The Hungarian Food Allergy and Food Intolerance Databank has published the "free from" product list since the year 2000. We found that the number of registered products increased between 2001-2008 (1. Figure). In addition in 2008 we published approximately 5000 pieces

to the patients who suffered from food allergy and we had approximately 5000 website-download. The Databank consists of an electronic data basis containing 9 categories (2. Figure). We hold continuous and regular nutritional counselling.
The stakeholders of the different areas find the Databank function necessary unambiguously because he complements the present legal regulation, and it facilitates allergic consumers' orientation (3. Figure).

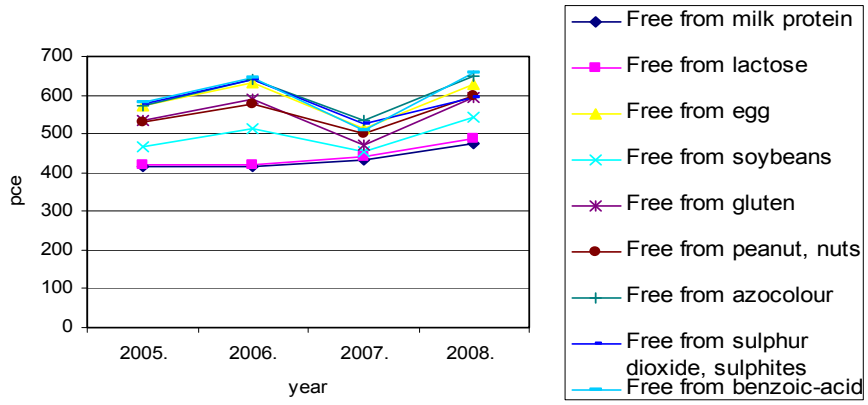

Figure 2
Product division according to free from categories
between 2005-2008

For the most important one the consumer protection and the policy makers keep it. In terms of the economicalness not the best intervention points or strategies, but the interviewers consider it as one which can be accomplished the most easily after all (4. Figure).

Discussion

The patients suffering allergy are very conscious consumers. They those who take it into consideration the most sincerely the labelling legible information.
The Hungarian Databank function side by side the labelling laws. It is an accessory and indispensable way for consumers suffering from food allergy/intolerance to know the information about “free from” product.

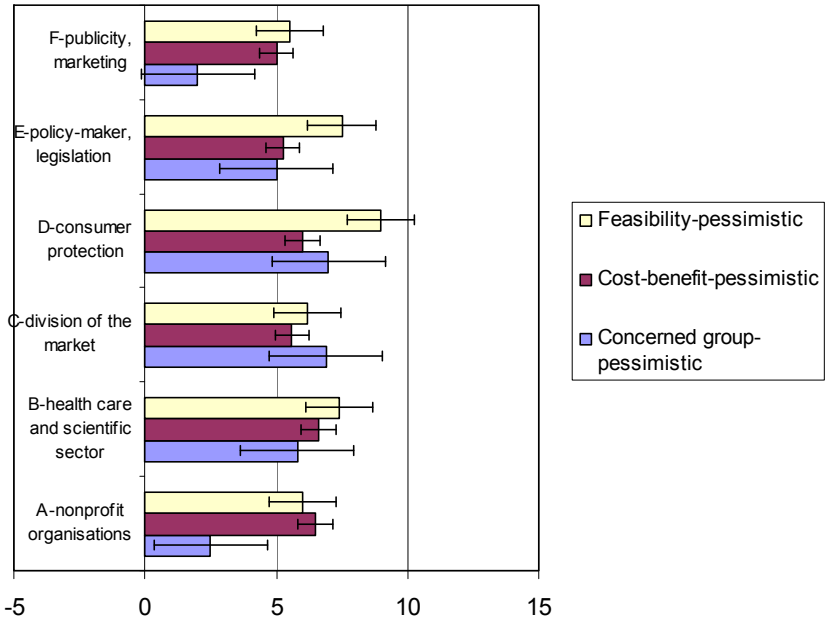

Figure 3
The necessity of the Food Allergy and Intolerance Databank based on the different stakeholder groups' opinion different criteria

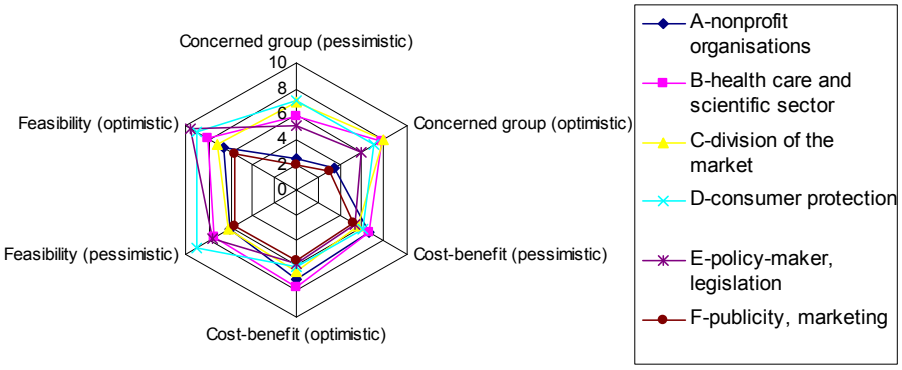

Figure 4
The necessity of the Food Allergy and Intolerance Databank based on the different stakeholder groups' opinion

The food safety includes the allergen information as well. The allergen information as consumers' information on the labelling are not sufficient and they do not replace the dietician counselling and patient management, despite this very important the full and reliable labelling, that is on the tag character the reliability of an allergen statement.

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LASER ABLATION OF FRUIT AND VEGETABLES

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ABSTRACT

This work considers the possibilities for peeling fruits and vegetables by treatment with a CO_2 laser. It was found that for the fruits under study the laser ablation provided an efficient peeling of the materials while preserving the organoleptic properties, such as freshness, naturalness, and texture. As a result of the treatment, a number of aroma substances were released, as well as plant cells, which could be used in the production of edible food coatings - an alternative to emulsion produced biopolymer films.
After laser treatment of citrus fruit (oranges and lemons), the fruits were peeled and pectin was extracted from the peels by means of a classical type of extraction in HCl water solution. The pectins obtained were studied for yield, degree of esterification, polyuronic content, molecular weight, gel strength and other physico-chemical prop-erties. It was found that in all samples the laser pretreatment of the materials led to an increase in the pectins yield, gel strength and purity, at an insignificant reduction of its molecular weight and degree of esterification.

INTRODUCTION

The specific interaction between the laser emissions and the biological structures made possible the wide use of IR and UV lasers in medicine and biology immediately upon their invention [1]. Recently, there has been a growing interest in the application of laser ablation of the raw materials in order to intensify the chemical and biological processes which are directly related to food technology. [2,3,4].
The aim of this work is to study the potential of laser ablation of fruit and vegetables as a new physical method for intensification of processes in the food technologies.

MATERIALS AND METHODS

The fruit and vegetables in the experiments were purchased on the local market. They were randomly assigned to either the control sample or the ablated sample at equal initial weight. The treatment of the fruit surface by means of laser irradiation was done using a CO_2- laser operating at

10.6 μm wavelength. Upon treatment of the fruit, the peels were removed and dried together with those from the control samples to a constant weight at room temperature. Pectin was extracted from the air-dried peels by means of a classical extraction [5] in an aqueous solution of 0,5M HCl at a temperature $t^0 = 85^0$ C, hydromodule 1:20 and time τ=45 min. The polyuronic content (PUC) of the materials was determined by the method of Gee [6]. The degree of esterification (DE) and the purity of the obtained pectins were assessed by the neutralization method of Owens et al. [7]. The IR spectra of the pectic preparations were taken on spectrophotometer Carry-5 using KBr tablets. The gel strength (GS) of the pectins was determined by measuring the strength of the reference 65% sucrose gels by the method of Tar-Baker [8]. The intrinsic viscosity[η] of the pectic aqueous solutions with 0,1 M NaCl was determined by the methods set out in [9] using Huggins' equation.
The molecular (meanviscosimetric) weight of the pectic macromolecules was calculated from the equation of Mark-Houwink

$$[\eta] = K.M^{\alpha}$$ as both the constants and $K = 9{,}55.10^{-2}$

$$\alpha = 0{,}73$$

were taken from Anger [10].

Theoretical rationale

Biological tissue can be viewed crudely as a material consisting of cells that reside in and attach to an extra cellular matrix. By mass, most biological tissue are dominated by water (more 60% to 99%), cellulose, pectin substances and other polysaccharides. Experimental results reported in the literature reveal that the energy density required to initiate ablation of biological tissue with nanosecond laser pulses is 10-fold less than that required for vaporization [4]. When the laser pulse duration is shorter than the characteristic time, the material is "inertially confined", i.e. it does not have time to expand and heating takes place at a constant time. Most materials are weaker in tension than in compression and it will fail wherever the induced tensile stresses exceed the tensile strength. When a biological object absorbs and is heated by laser energy, the resulting nonuniform temperature distribution causes internal forces which lead to thermoelastic deformation.

Water, cellulose and pectin are most often the main chromophores for pulsed IR or UV ablation, respectively.
Baranov et all [11] elaborates on the use of CO_2-laser operating at λ=10.43μm as a source of laser irradiation for modifying biological objects. It is shown that the adsorption of the electromagnetic radiation at λ=10.43μm was much larger compared with that at λ=1.06μm The calculations by the formulae of Frenel showed that the irradiation reflection off the surface of biological objects was insignificant and the laser energy was absorbed in a thin surface layer as deamplification was according to the law of Lambert-Buger where $k = e^{-\alpha . L_{abs}}$ $\alpha = \frac{4\pi\chi}{\lambda}$ is the linear absorption factor, χ- absorption index ($\chi_{H_2O} = 0{,}091$). For $k = 0{,}001$ the absorption penetration depth of CO_2 laser irradiation was $L_{abs.} = 64\mu m$,as of the energy was absorbed in the surface layer with a thickness ≈ 20 μm. The absorption energy was spent on heating and evaporation of the water contained in the biological object. The destruction of the plant tissue was caused by the hydrodynamic effect of laser irradiaiton. As a result of the intensive vapour formation in the capillary-porous structure of the plant tissue, an immense pressure was generated destroying the and bridges connecting the protopectin in the plant cell. The pectolytic enzymes responsible for the depolymerizaiton and deesterificaiton of the pectic macromolecules were inactivated. Rapid heating of tissue by pulsed laser radiation also leads to the generation and propagation of thermoelastic stresses as the heated tissue volume reconfigures to its new equilibrium state. The peak thermoelastic .ablative cutting or material removal re-quires the fracture of chemical bonds. The breakage of bonds leads either to the removal of molecules, molecular fragments and molecular clusters or to the formation of voids within the bulk of material. Bubble or crack formation results in the ejection of non-decomposed material fragments upon mechanical failure of the material. Vaporization, molecular fragmentation and void formation are all phase transitions and can be accomplished via photothermal, photomechanical mechanisms. $\approx 90\% Ca^{+2} Mg^{+2}$

fig 1

fig 2

fig 3

fig 4

fig 5

fig 6

fig 7

fig 8

Figures 1, 2, 3, 4, 5, 6, 7, 8
Non treated and CO_2 ablated surfaces of fruits and vegetables.

RESULTS AND DISCUSSION

Table 1
Experimental results on the yield and physicochemical properties of pectin from oranges and lemons

Sample	Treatment	Pectin vield %	Purity %	DE %	$[\eta]$ dlg^{-}	M_{η}	GS^{0}_{TB}
Thin-skinned lemons	Control Laser ablated	8,9 10,8	86,2 90,1	61,4 59,2	2.62 2.62	93 000 93 000	240 255
Thick-skinned lemons	Control Laser ablated	13,0 16,2	88,8 91,2	64,7 61,2	3.32 2.31	133000 78 000	270 280
Oranges 1	Control Laser ablated	9,0 12,0	77,4 80,1	58,4 56,2	1.16 1.39	31 000 39 000	225 230
Oranges 2	Control Laser ablated	9,1 11,6	78,4 81,3	59,0 56,8	1.17 2.32	32 000 79 000	240 250
Oranges 3	Control Laser ablated	13,1 16,5	77,9 80,2	65,2 65,0	4.33 4.52	186000 197000	270 285

It can be seen from the photos that the thickness of the removed surface layer was very small (up to several μm). It could vary with changes in the power of the laser beam and the time of treatment. That was optimized for each type of treated material. In all fruits and vegetables under study it was fount that as a result of the laser treatment the organoleptic properties, such as freshness, hardness, taste, and color were preserved which confirmed the apriori established fact that temperature changes were limited in depth to μm. Laser ablation has a substantial advantage compared with the classical thermal peeling in that it peels fruits and vegetables without significantly changing their organoleptic properties. A

follow-up study is needed to conduct biological research into changes in the enzymes and vitamins of the treated fruits and vegetables.

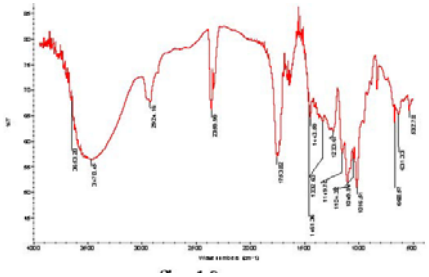

fig 9

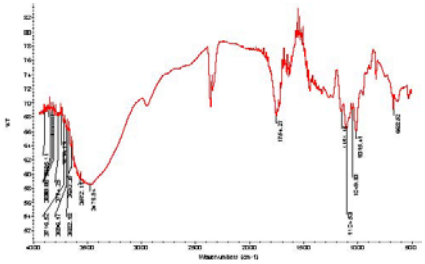

fig 10

Figures 9 and 10
Control pectin and of pectin obtained from laser ablated materials.

Table 1 presents a summary of the data from our experiments conducted with oranges and lemons concerning the yield and physicochemical properties of the pectin extracted from them. It can be seen from the table that for all cases the pre-treatment of fruits by laser irradiation led to an increase in the pectin yield compared with the controls, increase in the purity of the pectin obtained at an insignifican reduction of its degree of esterification. The molecular mass was lower only in the thick-skinned lemons, while in the other samples it was higher or unchanged. Figs 9 and 10 present spectra of control pectin and of pectin obtained from laser ablated materials. Both spectra distinctly show the absorption bands typical of the two pectins: 2500-3000 cm^{-1}, 1550-2500 cm^{-1} from 850-1500 cm^{-1} and from 400 to 850 cm^{-1}. The intensive narrow band of the valency fluctuations of the carbonyl  group from the ester  n the 1753-1754 cm^{-1} ranges, but they were better distinguished for pectin obtained as a result of laser ablation of the materials due to the higher purity of the pectic sample.

The gel strength of the reference 65% sucrose gels pepared with the pectin obtained from laser ablation as an indirect indicator of pectin quality, was higher in comparison with the values for the controls. During the preliminary laser ablation of the fruits it was found that there was an

intensive release of aroma substances. This fact gives reasons to carry out separate experiments aimed at extracting aroma substances from materials for which the traditional extraction processes are not efficient enough.

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RHEOLOGICAL PROPERTIES OF A GLUCOMANNAN OBTAINED FROM A PSYCHROPHYLIC YEAST STRAIN *SPOROBOLOMYCES SALMONICOLOR* AL_1

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ABSTRACT

Rheological properties of glucomannan were studied, such as its intrinsic viscosity (6.90 ± 0.22) dl.g-^{1}. The rheological profile of aqueous solutions of glucomannan was described by the power law $\tau = K\gamma^{n}$ as the value $0.33 \leq n \leq 0.60$ was not affected in a statistically noticeable manner by variations of concentration $0.25 \leq C \leq 2.0\%$ or temperature $25 \leq T \leq 82^{0}C$, which confirmed the pseudoplastic behaviour of glucomannan solutions as non-Newton liquids. It was shown that the structuring of the concentrated aqueous solutions of glucomannan was due to the formation of hydrogen linkages between the relevant macromolecules.

INTRODUCTION

Konjac glucomannan is a neutral polysaccharide isolated from the tuber of *Amorphophallus konjac* c.Koch. It consists of β-1,4- linked D- D-glucosyl and D-mannosyl units at a ratio 1:1.6, with about 1 in 19 units being acetylated (Zhang et al. 2004). Konjac glucomannan is mainly used in food industry as a gelling agent, thickener, film former, stabilizer, emulsifier and soluble fiber source in bakery products, confectionary, dairy products, noodles, pasta, health foods, functional foods, frozen foods and ice cream, canned, minced meat and fish, salad dressing and sauces, pet foods, edible films and capsules (Nishinari et al. 1992; Ratcliffe et al. 2005). Polysaccharides containing D-mannose and D-glucose in similar proportions were isolated from plants and produced from yeasts (BeMiller et al. 1993; Adami and Cavazzoni, 1990; Chiura et al. 1982; Vorotynskaya et al. 1992; Pavlova et al. 2004). Yeast glucomannan is not marketed as a dietary supplement.
The aim of this paper is to present the study of rheological properties of aqueous solutions of glucomannan produced by the psychrophilic strain *Sp. salmonicolor* AL_1.

MATERIALS AND METHODS

Yeast strain *Sporobolomices salmonicolor* AL_1 was isolated from soil lichen taken from the region of the Bulgarian base on Livingston Island, Antarctica and selected as a suitable producer of exoglucomannan (Pavlova et al. 2004). It was registered in the National Bank for Industrial

Microorganisms and Cell Cultures, Bulgaria, under № 08290. Exopolysaccharide production, isolation of crude exopolysaccharide and it chemical analysis was published in a previous article of ours (Pavlova et all 2004).

Intrinsic viscosity $[\eta]$ of glucomannan solutions was determined by measurements with capillary viscosimeter VPG 2 type Ubelode at temperature 25^0C. Experimental data processing was done by a computer program written in FORTRAN 77 on the grounds of an algorithm for finding a linear equation in accordance with Huggins equation (Morawetz 1967). The rheological profile of the aqueous glucomannan solutions was measured by means of a Rheotest – 2 Rheoviscometer, Germany, using measuring cylinder of the device N. The concentrated solutions of glucomannan under study were within the 0.50 – 2.50% range. The measurements were conducted at different temperatures in the $25 \div 82^0 C$. range. The Oswald-de Waale power law model (Holdsworth 1993) was used to describe the flow behaviour of glucomannan solutions

$$\tau = K.\gamma^n \qquad (1)$$

where τ is the shear stress (Pa), and γ is the shear rate (s^{-1}) K is the consistency index ($Pa.s^n$), n is the flow behaviour index (dimensionless)

The numerical values of the shear rate γ and the shear stress τ were processed using REOTEST computer program written in FORTRAN 77. During test data processing, the methods of the least squares were applied to find equations of simple and plural linear regression (Draper 1981). Numerical procedures implementation was performed by computer subroutines compiled by (Johnson 1980).

RESULTS AND DISCUSSIONS

The parameters established during the batch fermentation of the strain indicated that a temperature of 22 °C, aeration at 0.75 v/v/m and agitation at 500 rpm proved to be the most suitable conditions for polysaccharide synthesis. During the biosynthetic process, the apparent viscosity of the culture broth increased to the maximum value of 15.37 mPa.s, and the polysaccharide yield was 5.63 g/l on a culture medium containing 4.00%

of sucrose and 0.25% of ammonium sulfate, at a temperature of 22°C for 120 hours.

The following results were obtained from the experiments carried out with capillary viscometer VPG 2 type Ubelode at temperature 25^0C from Huggins equation

$$[\eta] = (6{,}90 \pm 0{,}22) dl.g^{-1} \qquad K''_H = 0{,}58 \pm 0{,}14$$

The rheological profile of glucomannan water solutions was determined at a shear rate γ at $1{,}5 \div 1312 s^{-1}$. The n values obtained during our studies from equation (1) for all solutions of glucomannan fell within the $0{,}33 \le n \le 0{,}60$ range, which showed that glucomannan behaved as pseudoplastic non-Newtonian liquids.

The quest for dependence of $n = f(C\%)$ and $n = f(T)$ did not yield satisfactory results. For all regression dependences the correlation coefficient was R<0.63. This was to show that n was not affected in a statistically noticeable manner by the variation of concentration and temperature.

The magnitude of the consistency index K grew with increase of concentration of solutions. Both exponential (Eq.2) and power type (Eq.3) relations were found to be suitable for the description of the relationship between the concentration and the consistency index.

$$K = 1{,}064.\exp(0{,}9876C) \qquad (2)$$

the correlation coefficien R=0.932

$$K = 3{,}781.C^{0{,}883} \qquad (3)$$

the correlation coefficien R=0.976

The joint effect of the concentration C and temperature T on the K values was studied by means of a multiplicative model of multiple regression of the kind (4) :

$$K = \alpha.C^{\beta}.\exp\left(\frac{\Delta E_a}{RT}\right) \qquad (4)$$

where α and β are constants, R – gas constant kJ/mol.K, ΔE_a – activation energy (kJ/mol)

Processing the experimental data was implemented using the MLRG software based on the least squares method for finding multiple regression within the range $0{,}25 \le C \le 2\%$ and changes in T within the $25 \le T \le 82^0 C$ range.The values obtained for the constants in equation (4) at a multiple correlation coefficient R_n=0.969 were:

$$\alpha = 4{,}75410^{-5}$$

$$\beta = 0{,}7579$$

$$\Delta E_a = 28{,}55 \pm 1{,}24 \frac{kJ}{mol}$$

According to (Cottreli 1954) the energy of the hydrogen bond is $\Delta E_a \approx 23{,}4 \frac{kJ}{mol}$

The value we obtained for the activation energy showed that, the hydrogen bonds established between the separate macromolecule chains had a dominant importance in the formation of structures in the aqueous solutions of glucomannan. Their ruptures when the velocity gradient γ was changed and the following recovery determined the pseudoplastic nature of glucomannan. The value we obtained for the activation energy $\Delta E_a = 28{,}55 \frac{kJ}{mol}$ was very close to the value of the activation energy $\Delta E_a = 27{,}63 \frac{kJ}{mol}$

Obtained by Maekaji (Nishinari et al. 1992) on konjac mannan obtained from the tuber of the Amorphophallus konjac plant c. Koch.

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COMPARISON OF THE ANTIOXIDANT ACTIVITY IN BERRIES AND FRUIT PRODUCTS MADE FROM BERRIES

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ABSTRACT

Berries (raspberry, blueberry, blackberry, black and red currant, gooseberry, etc.) are one of the popular group of fruits. They contain pigments in a big quantity. These pigments belong to the group of the anthocyanidins and are mostly flavonoids. These compounds are very important in the healthy nutrition. Our aim was to examine the berry species known on Hungary and the prepared foods from them. We wished to collect data with the examinations the fruit's antioxidant characteristics, and to find contexts concerned with the measured antocianidin pigment content with C-vitamin content.
Highest (more than 200 mg/100 g) C-vitamin content was measured in the elder, black currant, blueberry and in the blackberry. Very high (50 mg/100g) anthocyanidin quantity was measured in the elder and the blackberry. High pigment content (10-20 mg/g) was in the black currant, blueberry and in the josta. Anthocyanidins and C-vitamin are responsible for the antioxidant activity together.
The quantity of antioxidant activity in fruit-foods differ from that of the fresh fruits with an order in generally. We found very big differences in measured nutritional value because of different quality of jams and frozen fruits.

INTRODUCTION AND TARGET

As it is known the berries contain big qunatity of pigments, which mainly are flavonoids which belongs to the anthocyanidin group. Firstly its related to this group the big antioxidant and free radical inhibition capacity. These properties in the last period were overvalue in the healthy diet. Our target was to test the antioxidant capacity of those berries species and those groceries made from berries, which are mostly consumed in Hungary. We wanted to collect data with these examinations about the fruits antioxidant properties and to relate our measures with the anthocyanidin pigment and the C-vitamin content. Lastly we studied, how fruits keep their antioxidant capacity during processing.

MATERIAL AND METHOD

We got the raw samples from Hungarian Central Agricultural Office, Fruit Variety Trial Station Pöloske, and we bought the fruit food products like juice, jam, dried fruits, canned and frozen fruits in groceries. Our working team used traditional methods to determine the antioxidant activity and pigment content of fruits and fruit products. We used spectrophotometer for the measure.

Antioxidant activity

We used the classical DPPH method. We gave the sample activity, named H-donor activity with the I_{50} value (50% inhibition) which shows among standard circumstances how much sample quantity cause 50 % colour reduction of the free radical komplex (2,2,-diphenyl-1-picryl-hidrazil-hidrate) added to the sample.

Flavonoids - anthocyanidins

We solved from the samples the pigment colour with sulfuric-acid-etanol-mix. After direct measurement of absorbance we counted the whole quantity of flavonoids with the equivalence of the main pigment component (cyanidin-3-glucosid).

C-vitamin

After watery solution we added ferri-chloride and 2,2,-dipiridil reagent to determine the quantity of vitamin with measuring colour intensity of dipiridil-ferro-ascorbate komplex (altered Spanyár-method).

RESULTS AND DISCUSSION

RAW FRUIT SAMPLES

The data of measurements are in Table 1. Very high (more than 200 mg/100 g) C-vitamin content was measured in the elder, black currant, blueberry and in the blackberry. High (more than 110 mg/100 g) C-vitamin content was measured in the red currant, in the red gooseberry and in josta (it is an species-hibrid goosberry x black currant). Medium (40 mg/100 g) C-vitamin content was measured in the raspberry and the blue grape, 30 mg/100g in the red cranberry and the white currant.

Table 1
Analytical results: vitamin C, anthocyanidin and antioxidant activity in raw fruits

BERRY (Latin name)	Variety	Harvest time	C vitamin mg/100g	Antho-cyanidin mg/g	Anti-oxidant activity I50, mg
Elder (Sambucus nigra)	Haschberg	Aug 30	240,0	53,85	0,165
Black currant (Ribes niger)	Titania	July 05	238,2	21,10	0,119
Blueberry (Vaccinium myrtillis)	Heidii	July 05	213,8	12,18	0,423
Blackberry (Rubus fruticosus)	Loch Ness	July 05	203,4	46,80	0,090
Red currant (Ribes rubrum)	Jonkeer v Tets	July 05	114,1	7,50	0,552
Josta (Ribes uva-crispa x Ribes nigra)		July 05	110,4	11,85	0,428
Gooseberry red (Ribes uva-crispa)	Rokula	July 05	79,0	8,36	0,484
Raspberry (Rubus idaeus)	Rumilova	July 05	45,4	5,40	0,457
Grape red (Vitis vinifera)	Hamburgi muskotály	Sept 24	45,1	7,49	0,312
Raspberry yellow (Rubus idaeus)	Golden Bliss	July 05	33,6	0,37	0,336
Cranberry (Vaccinium macrocorpon)	Koralle, Pilgrim	Sept 11	28,1	3,49	0,724
White currant (Ribes rubrum)	Blanka	July 05	24,4	2,43	0,522
Rose hip (Rosa canina)		Sept 30	127,2	1,615	0,011

Very high (50 mg/100g) anthocyanidin quantity was measured in the elder and the blackberry
High pigment content (10-20 mg/g) was measured in the black currant, blueberry and in the josta. Anthocyanidins and C-vitamin are responsible for the antioxidant activity together.

PROCESSED FRUIT SAMPLES
The data of measurements are in Table 2.

Jams

It is evident, that the C-vitamin content in jams is much less compared to the fresh fruits since they are prepared by heat treatment. The C-vitamin content in the majority of jam samples is about 20-40 mg / 100 g exception of elder and rose-hip where we found more than 100 mg C-vitamin.

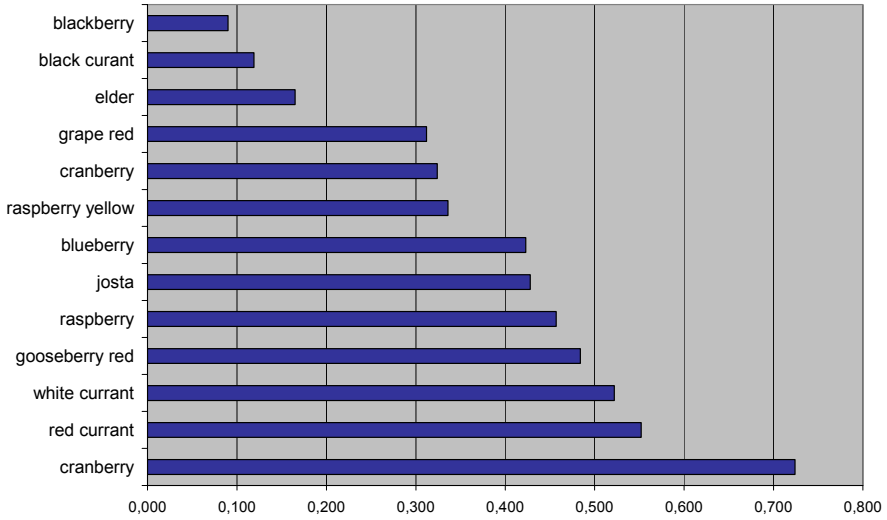

Figure 1.
Antioxidant activity of fresh fruits (I_{50} mg)
mg sample necessary to 50% inhibition in colour of DPPH reagent

The anthocyanidin content decreased to third or fifth parts compared to the fresh fruits.

The quantity of antioxidant activity in jams differ from that of the fresh fruits with an order in generally. We found very big differences in measured nutritional value because of different quality of jams. The C vitamin contents changed 20-70 mg / 100 g in different rose-hip jams.

Canned fruits

A few samples were examined, we took out only the fruit as a sample from the canned fruits.

The C-vitamin and anthocyanidin content are very high in canned blueberry, medium level in raspberry and strawberry.

Table 2
Analytical results: vitamin C, anthocyanidin and antioxidant activity in fruit-foods

BERRY Food type	**C-vitamin mg/100g**	**Antho-cyanidin mg/g**	**Anti-oxidant activity I50, mg**	**BERRY Food type**	**C-vitamin mg/100g**	**Antho-cyanidin mg/g**	**Anti-oxidant activity I50, mg**
ELDER				RASPBERRY			
fresh	240	53,8	0,165	fresh	45,4	5,40	0,457
Sokoró jam	22,0	19,2	0,875	frozen	48,4	4,61	0,821
Botész jam	55,5	23,9	0,198	Pacific jam	17,2	0,10	1,265
Botész pulp	69,1	53,3	0,108				
BLACKBERRY				RED CURRANT			
fresh	203	46,8	0,09	fresh	114	7,50	0,552
frozen	39,3	12,1	0,861	frozen	48,6	5,62	0,777
canned	37,2	7,70	0,984				
BLACKCURRANT				STRAWBERRY			
fresh	238	21,1	0,119	canned	15,8	1,69	1,036
Pacific jam	16,9	0,55	1,350	Pacific jam	15,8	0,00	2,086
Pacific mini jam	20,7	0,10	1,048	frozen	24,4	7,08	0,908
Blueberry				rose hip			
fresh	214	12,2	0,423	fresh	127	1,61	0,011
Herbária jan	17,7	2,24	0,508	Botész jam	69,1	1,86	0,376
St Dolfour jam	18,8	3,49	0,996	Vácrátót jam	34,6	1,06	0,809
Pacific jam	16,1	0,10	1,330	Herbária jam	18,5	1,43	1,296
				Pacific jam	17,4	0,54	0,953

Frozen fruits
In several frozen fruit samples we measured significantly lower vitamin and pigment content than in the raw fruits. The loss of vitamin and pigment arose in the course of the transport and preparation of fruits, or long storage of frozen products.
We measured high vitamin C in an very good quality frozen raspberry.
Juices
The antioxidant activity of juices shows a tight connection with the fruit content. Between the grape juices with 100% fruit content there is not an

essential difference considering the manufacturers. We measured the biggest antioxidant activity in the juice made from elder and black currant together. (Figure 2)

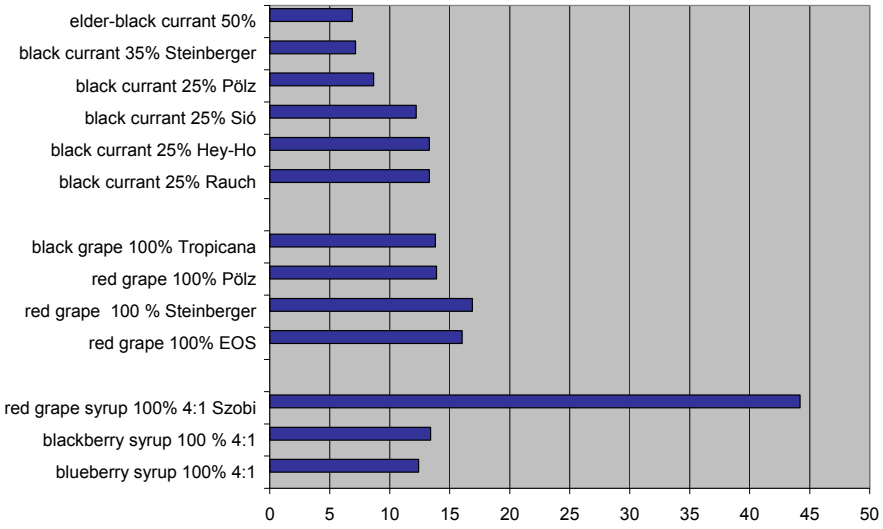

Figure 2
Antioxidant activity of fruit juices (I_{50} ml):
ml sample necessary to 50% inhibition in colour of DPPH reagent

Conclusion

The results gave us a numerical comparison of the different berries related with the nutrition value. Among the examined fruits the ones which shown the biggest antioxidant activity were: black elderberry (Sambucus nigra), black currant (Ribes nigra) and blackberry (Rubus sp). We gave importance to the measured data of the middle field since those berries like the raspberry or the strawberry are consumed the most in Hungary. The data we got from the fruit products were very diverse since the fruit content and the heat treatment influence the got results. It is important to consider the raw material maturity and quality as well. Remarkable differences were detected in C-vitamin content in different rose hip jams, and in anthocyanidin content in different elderberry jams as well. To conclude, on the base of results we can recommend these fruits to the people who are interested in continue a healthy diet.

EFFECTS OF SOME EXTRUSION PARAMETERS ON THE HARDNESS OF EXTRUDED LENTILS

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ABSTRACT

The objective of this research was to study the effects of moisture content (18, 20, and 22%), and screw speed (100, 150, and 200 rpm) on the hardness of extruded lentils. The lentil semolina was extruded with a single screw extruder (Brabender 20 DN) at constant barrel temperature (160ºC), screw compression ratio (2:1), and die diameter 4 mm. The hardness of the extrudates was measured with a TA.XT Plus Texture Analyser, Stable Micro Systems. The textural profiles of the extrudates showed that feed moisture had the highest effect on the hardness.

INTRODUCTION

Lentil (*Lens culinaris*) is one of the oldest known and one of the most important pulse crops. It is a rich source of protein (22-35%), complex carbohydrates (50-58%), dietary fiber, minerals (potassium, calcium, copper, iron, magnesium, phosphorus, and zinc), vitamins (folic acid and other B vitamins), and polyunsaturated free fatty acids (linolenic, linoleic, and oleic acids). It is also high in leucine, isoleucine, lysine, histidine, tyrosine and other amino acids. Lentil is deficient in two essential amino acids, methionine and cystine (Wang et al., 2006).
Lentil is an excellent supplement to cereal grain diets because of its good protein/carbohydrate content. It is used in soups, stews, casseroles and salad dishes. Sometimes they are difficult to cook because of the hard seed coat that result from excessively dry production conditions.
Extrusion technology can be used to inactivate antinutrients, reduce the cooking time and the concentration of complex carbohydrates, and to

improve the nutritional, textural, and sensory characteristics of dry bean extrudates (Berrios, 2006).
Texture is an important attribute in that it affects processing and handling, influences food habits, and affects shelf-life and consumer acceptance of foods. Food texture is commonly characterized by means of sensory and/or instrumental methods.
The hardness is very important textural characteristic from a processing viewpoint. Extruded lentils needs to be dried to an appropriate moisture level and desired texture to facilitate milling, blending, mixing, or further processing (Yanniotis et al., 2007).
The aim of this investigation was to study the effects of some extrusion parameters on the hardness of extruded lentil.

MATERIALS AND METHODS

1. Lentil semolina

Lentil seeds were ground using a hammer mill and passed through standard sieves. Prepared particle size of lentil semolina was about 1 mm with an approximate composition of: moisture 10.2%, protein 28.1%, carbohydrates 57.1%, and fat 1.0%. Lentil semolina was mixed with distilled water to be obtained various moisture contents 18, 20, and 22%. Samples moisture was equalized in a refrigerator at 5 ºC for 24 h.

2. Extrusion

Lentil semolina was extruded in a laboratory single screw extruder (Brabender 20 DN, Germany) with compression ratio of 2:1, screw diameter 20 mm, and die diameter 4 mm. The feed screw speed was fixed at 50 rpm. The temperatures in the barrel from the inlet to the die were kept constant at 140-150-160ºC. The screw speed was variable (100, 150, and 200 rpm).

3. Hardness

Hardness of the extrudates was measured in tenfold with a TA.XT Plus Texture Analyser (Stable Micro Systems Ltd., England) using a 50 kg load cell and a 2-bladed Kramer shear cell. The test settings were as follows: Test speed 1.0 mm/s, Distance 10 mm.

4. Data analysis and presentation of the results

It was carried out two-factor experiment with three levels of variance. The experimental results were analyzed using Analysis of variance method (ANOVA). The ANOVA decomposed the variability of the measured force into the effects due to moisture content and screw speed. The effect of each factor was assessed having removed the effect of the other factor. The interaction between moisture content and screw speed was also considered. The results are presented in the diagrams as mean values with the 95.0% confidence interval.

	Levels		
Moisture content (%)	**18**	**20**	**22**
Screw speed (rpm)	**100**	**150**	**200**

RESULTS AND DISCUSSION

In this investigation the hardness was determined as the maximum force required for fracture of the extrudates with the TA.XT Plus Texture Analyser. Typical plot produced from testing of extruded lentils is given on Fig. 1.

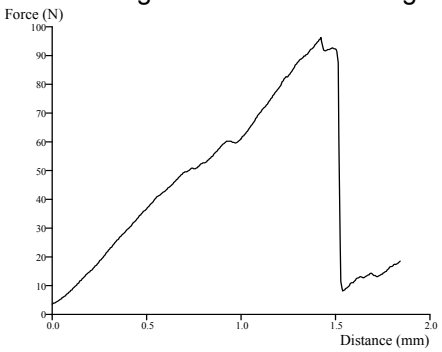
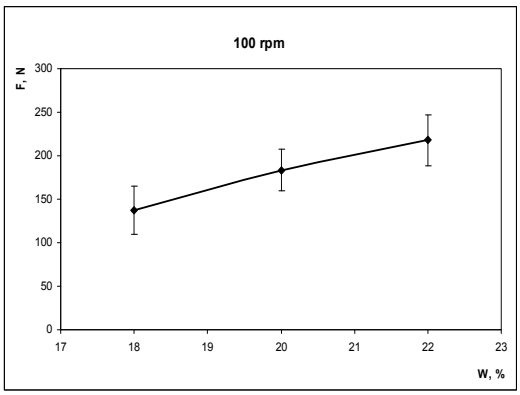
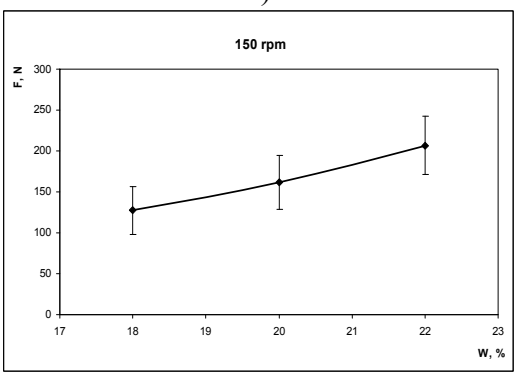


Figure. 1
Typical curve of extruded lentil tested
with TA.XT Plus Texture Analyser

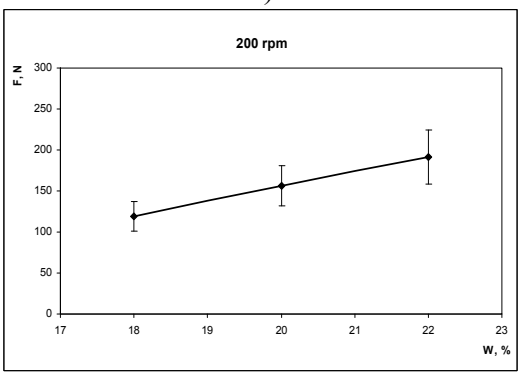
The results show a trend toward increase of the hardness with increasing the moisture content from 18 to 22% (Fig. 2). The analysis of variance shows that there is a statistically significant difference at the 95.0% confidence level in the hardness between the samples with 18 and 22% moisture content.



a)



b)



c)

Figure 2
Hardness vs. moisture content of extruded lentil at various screw speeds

Based on the results from ANOVA the following regression equation has been obtained:

F = - 221,056 + 21,25W + 0,05n, (N)
Where F – measured force, N; W – moisture content, %; n – screw speed, rpm.

The ANOVA table partitions the variability in the measured force into separate pieces for each of the effects due to moisture content and screw speed as well the interaction between them at the 95.0% confidence interval (Fig. 3).

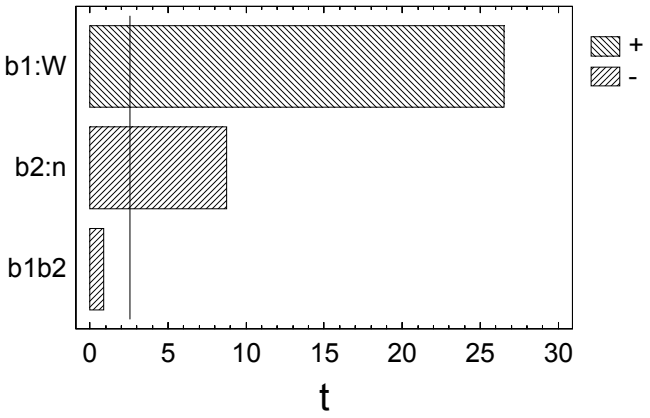


Figure 3
Standardized estimated effects of regression model coefficients on the hardness
(R-squared = 99.3619% Standard Error of Est. = 3.51347)

The linear effect due to the moisture content of lentil semolina (W, %) had mostly influence on the hardness followed from the effect due to the screw speed of the extruder (n, rpm) at this extrusion condition (compression ratio of 2:1; die diameter 4 mm; barrel temperature 160ºC). The residual quantity distribution for the regression model of the hardness is uniformly distributed around zero and no values exceed two times the standard error (Fig. 4).

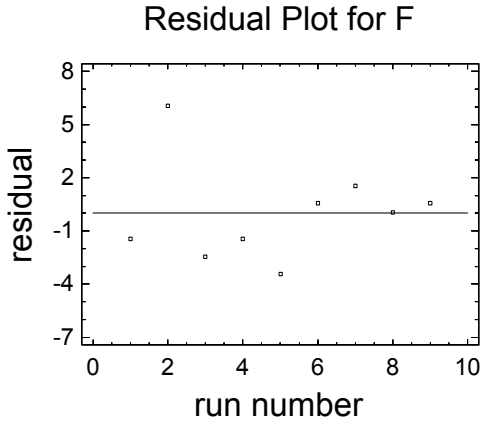
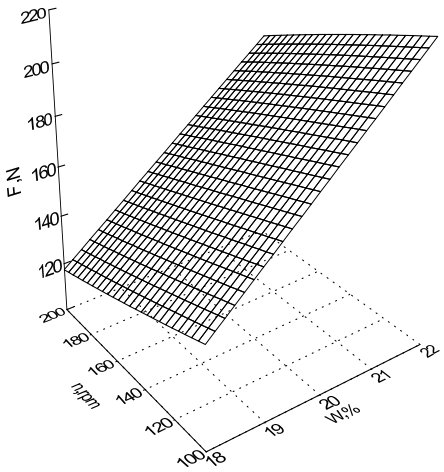


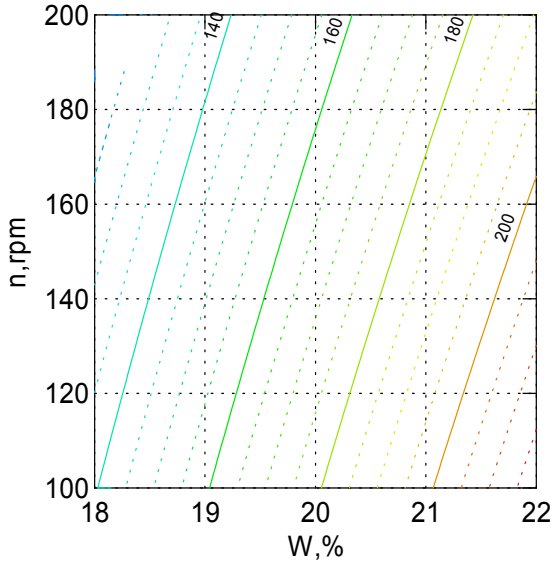
Figure 4
Residual distribution diagram for the regression model
of the hardness
(R-squared = 99.3619% Standard Error of Est. = 3.51347)

The hardness of extruded lentils increases by 38% with raising the moisture content from 18 to 22% for each of the extruder screw speeds (100, 150, and 200 rpm). The hardness decreases by about 7% with increasing the screw speeds from 100 to 150 rpm, and from 150 to 200 rpm at one and the same moisture content (Fig. 5).

These results correspond with established from Winnikowa (1991), Balandran-Quintana et al. (1998), and Avin et al. (1992). Winnikowa has investigated the effects of extrusion on the hardness of corn meal extrudates. She has established that the moisture content has the highest influence on the hardness of extrudates at moistures varying from 14 to 20%, die temperatures from 150 to 200ºC, and screw speeds from 6.64 to 7.74 s^{-1}. Balandran-Quintana et al. have extruded pinto bean flours using a Brabender single screw extruder at three different die temperatures (140, 160, and 180ºC), feed moisture content (18, 20 and 22%), and screw speeds (150, 200, and 250 rpm). They have reported that screw speed had no effect on any depending variable. Avin et al. have extruded red bean flours containing either low (15 %) or high (25 %) moisture, in a Brabender laboratory single screw extruder at temperatures of 90, 110, and 124ºC and screw speeds of 80, 120, and 160 rpm.



a) Three-dimensional coordinate system



b) Two-dimensional coordinate system

Figure 5
Hardness changes (F, N) depending on moisture content of lentil semolina (W, %) and extruder screw speed (n, rpm)

They have reported that screw speed only influenced extrusion yield. The highest value for hardness (218 N) of extruded lentils was obtained in samples at 22% moisture and 100 rpm. The lowest value for hardness (117 N) was obtained in samples at 18% moisture and 200 rpm.

CONCLUSIONS

The effect of moisture content of lentil semolina and screw speed of the extruder on hardness of extruded lentil was studied. The moisture content of the lentil semolina affects mostly the hardness of the extrudates at the present extrusion condition. The screw speed of the extruder has a slight influence on the hardness. The hardness increases with raising the moisture content from 18 to 22%. It decreases with increasing the screw speed at one and the same moisture content. The highest value for hardness of extruded lentils was obtained in the samples at 22% moisture and 100 rpm. The lowest value for hardness was obtained in the samples at 18% moisture and 200 rpm.

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RESEARCH ON THE ECOLOGICAL CONDITION OF THE AGRICULTURAL OBJECTS AFTER THE IMPACT OF POTENTIAL CONTAMINATION

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ABSTRACT

The appearance of biological, chemical and radioactive polluters in foods creates a potential risk for the health of the consumers. The polluters in foods can have direct effect and future health effect. Potentially risky substances enter in foods in a consequence of the pollution of the environment due to the transport and industry as well as the violation of the good agricultural and production practice.
The radiation pollution on the territory of Europe after the Chernobil accident poses a new problem – late consequences due to the components of the radioactive tail, included in the composition of the agricultural products and feed raw materials.
The nuclear power station "Kozlodui" turns up a potential source of radioactive impact upon the population and the environment in case of an accident. The main technological equipment and systems in case of an accident may provoke radioactive emissions on the territory of the nuclear power station and the environment around it.
These circumstances define the priority of the radiation monitoring in the North-West of Bulgaria. During the period 2004 – 2007 we carried out researches on the content of radionuclides in soil, cow milk, fish, game and other agricultural objects.
The obtained results on the pollution level of the examined samples show that it is considerably below our national and European standards.

INTRODUCTION

The pollution of the environment and the food products of vegetal and animal origin with radionuclides, heavy metals and other toxic elements emerge as a dominating problem in the ecology. The fast development during the last decades of the past century of the nuclear energetic,

ferrous and non-ferrous metallurgy leaded up to the entry of number of technogene polluters in the environment on the territory of Bulgaria.
The monitoring of the radiation background in every point of the terrestrial surface is important characteristic of the radiation status of the environment.
It is well-known that on the way of the food chain the radionuclides, the heavy metals and other toxic elements, through the soil, water and plants enter the animal constitution, food raw materials and products of animal origin and from there – the human body.
That is why the food raw materials and products, obtained in region of the country of a risk regarding the pollution with technogene radionuclides (radionuclides and heavy metals) can be sources of their remaining quantities.
The purpose of the present investigation is the following: to define the level of the pollution of the workable soils, vegetal and animal raw materials and products from north-west of Bulgaria with technogene polluters i.e. with radionuclides and heavy metals.

MATERIALS AND METHODS

The object of the carried out researches during 2004 - 2007 were 358 samples from workable soils, 5 samples from green crop, 114 samples from cow milk and milk products, 13 samples from freshwater fish and 66 bone samples from game (hind, deer and wild-boar), mainly from previously determined control points of the monitoring net from the regions of Vidin, Montana, Kozlodui I Vratza. (Annual Report on the environment status in R. Bulgaria 1999; Bulletin, 2000)
The samples of cow milk and milk products were taken by the employees of the State veterinary sanitary control from private producers and companies and the rest of the samples are gathered in expeditions.
The preparation of the samples of animal raw materials and products for analysis of heavy metals and other toxic elements was made by methods, described in the developed by us research project (Monov,G. and collaborators, 1996), and of the soil samples through the receipt of acid extract.
The quantity assessing of heavy metals was done by the method of nuclear emission spectrometry with apparatuses Spectroflame. The quantity assessing of technogene radionuclides was done by non-

destructive gamma-spectrometric analysis on γ-spectrometric system ORTEK with 25% HPGe detector, with time of measuring 60000 seconds in container type MARINELI with volume 0.5 l and through radio-chemical analyses according "Modified complex methodic for radio-chemical assessment of low beta-activities from ^{90}Sr and ^{137}Cs in biological objects" and "Methodic for radio-chemical assessment of ^{90}Sr in soils and water samples" (Naidenov, M. and collaborators, 2001).

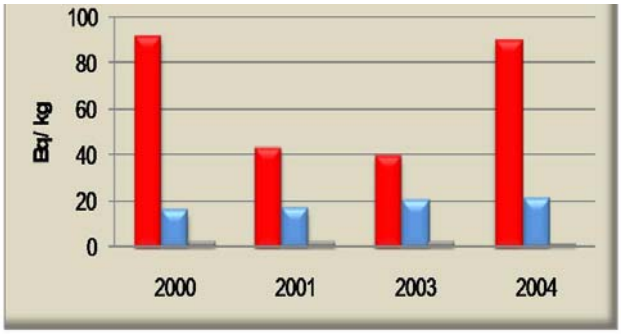

Figure 1
Contents of Cesium-137 in Soil Samples
from North-West Bulgaria (Bq/kg)
(min, an average value and max)

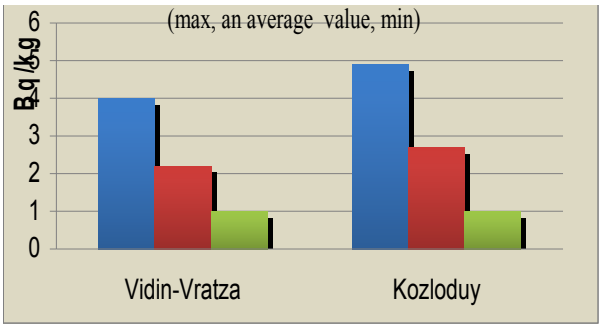

Figure 2
Contents of Strontium-90 in Soil Samples
from North-West Bulgaria (Bq/kg) 2003-2004
(max, an average value, min)

The received results from the tested samples are statistically processed by the method of Student-Fisher and other program products (Student-Fischer, 1976).

RESULTS AND DISCUSSION

The obtained results from the researches we generalized and presented in 6 graphic figures. We grouped the data in the way that we have a concept of the presence and the quantity of technogene radionuclides and heavy metals in each link of the food chain of the population which lives in the researched area.

On fig.1 and fig.2 the content of ^{137}Cs and ^{90}Sr in Bq/kg soil is presented. We have shown the minimal, maximal and average value.

Comparing the maximal values of ^{137}Cs during the particular years it can be seen that they are in range between 90 and 40 Bq/kg soil, and the minimal values – around the minimal provable measuring value i.e. below 5 Bq/kg soil. The average values for the separate years are between 16 and 20 Bq/kg soil.

The results from the accomplished 114 gamma-spectrometric analyses of cow milk and milk products have shown values of ^{137}Cs around and below the minimal detectable activity from the gamma-spectrometer i.e. around and below 5 Bq/l milk, which didn't allow us to preset them graphically.

On fig.2 we compared the content of ^{90}Sr in the workable soils average for the whole north-west of Bulgaria and in the workable soils from the 30 km zone around NPS "Kozlodui".

The range of the measured maximal and minimal values is between 5 and 1 Bq/kg soil.

On fig.3 we presented the content of ^{90}Sr in green crops, cow milk and game tube bones. For the determination of ^{90}Sr we used radio-chemical analyses. (Grodzinskij, D.1965; Juravlev, V. 1982)

For the green crops the average values for the content of ^{90}Sr are around 2 Bq/kg, and the range between the maximal and the minimal values is from 4 to 1 Bq/kg. (Moskalev, Y., 1970)

We also presented the content of ^{90}Sr in cow milk as an end product in order to illustrate the participation of one of the most toxic radioactive elements in the human food chain.

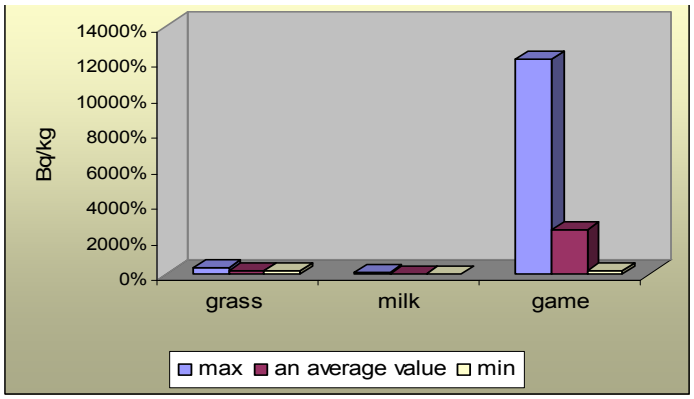

Figure 3
Contents of Strontium-90 in Biological Entities
from North-West Bulgaria (Bq/kg) 2000-2004
(max, an average value, min)

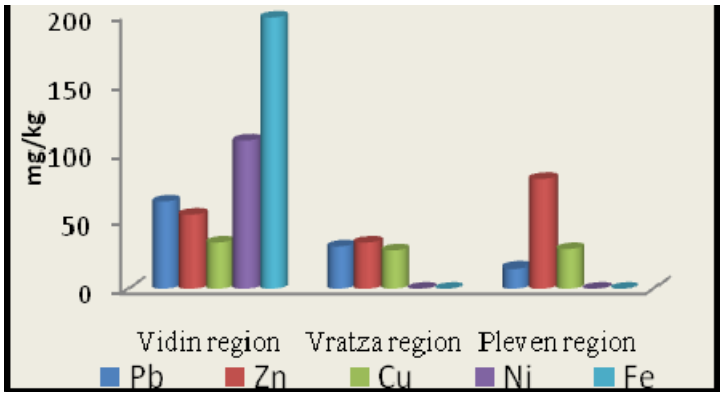

Figure 4
Contents of Heavy Metals in Soil Samples
from North-West Bulgaria (mg/kg) 2000-2004

It can be seen on the chart that the values are neglectfully low, from maximal - 0,4 Bq/l, through average – 0,24 Bq/l to minimal – 0,15 Bq/l. (”National plan for foods and feeding 2005 – 2010 ”, 2005)
Having the values of the cow milk and the green crop, the values of ^{90}Sr in the bones of the wild animals make impression.

We resumed the data received from the research on the pipe bones of deer, roes and wild boars.
These wild animals were included in our research in order to demonstrate the effect of the different feeding between wild animals and farm animals, the free way of life of the wild animals, the above sea-level habitation, etc.

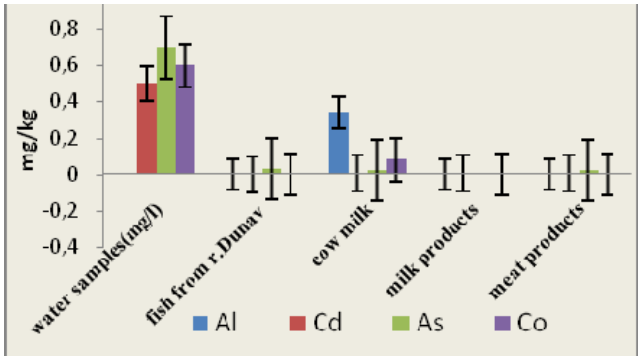

Figure 5
Contents of Heavy Metals in Samples
from North-West Bulgaria (mg/kg) 2000-2004

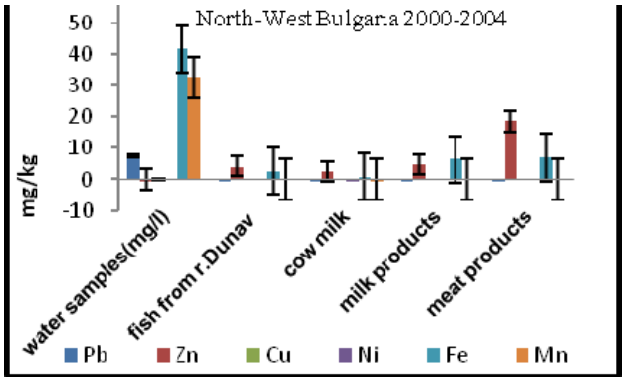

Figure 6
Contents of Heavy Metals in Samples
from North-West Bulgaria (mg/kg) 2000-2004

The highest value of ^{90}Sr – 121 Bq/kg is measured in deer from the mountain region of Berkovitza and the lowest – around 2 Bq/kg are

measured in wild boars, shot in he regions of NPS “Kozlodui” and “Belene”. The average values in these researches are around 25 Bq/kg. These data show that there is no reason for worry for including green crop, gathered from the high mountain regions or root fodder (potatoes, carrots, beetroot, etc) produced in the regions of NPS “Kozlodui” and “Belene”.

Together with the researches for remaining quantities of radioactivity, the collected sample material was examined as well for the content of heavy metals and the received results we resumed in 3 figures.

On fig. 4 we presented the content of Pu, Zn, Cu, Ni, and Fe as average values from workable soils from North West of Bulgaria, which we divided in 3 regions – of Vidin, Vratza and Pleven.

From the demonstrated data of content of these heavy metals and metalloids in the workable soils it can be seen that they are considerably below the approved norms for top acceptable concentrations, which depending on Ph of the workable soil are between 60 -120 mg/kg for Pb, 200 – 400 mg/kg for Zn, 80 - 300 mg/kg for Cu, 90 – 150 mg/kg for Ni, 1,5 – 3,0 mg/kg for Cd, 1,5 mg/kg for Hg, 20 – 40 mg/kg for Co and 25 mg/kg for As. (Regulation №3).

The data from fig.5 and fig.6 reflect the content of Pu, Zn, Cu, Ni, Fe, Mn, Al, Cd, As and Co in drinkable water which is used in the production of food products, fish from the river Danube waters, cow milk, milk and meat products.

For basis of the veterinary-sanitary assessment of the obtained results we applied Regulation № 12 / 21.21.05.2002 of MH “For norms of maximal admissible quantities of heavy metals as polluters in foods”. (Regulation №11, Official gazette 44/2002)

All the examined samples from the different food raw materials and products showed results below accepted by us hygiene norms for top admissible concentrations, which give us reason to admit that the produced in north west Bulgaria food raw materials and products for the appointed period are not of a toxicological risk for the population. (Regulation № 12, Regulation №3 / 01.08.2008; Basic standards, Official gazette 5/2001)

The results of the conducted researches give us reason to make the following conclusions:

1. The investigated workable soils are with content of Pb, Cu, Cd и As considerably lower from the accepted by us norms and quantities of technogene radionuclides, typical for the geographic region.
2. The obtained results form the researches are categorical that none of the samples of vegetal and animal products content quantities above the norms of the Pb, Zn, Cd, Cu, Ni, Fe, Mn, Al, Co and metalloid As.
3. The raw cow milk , the white cheese and the yellow cheese produced in the researched area of north-west Bulgaria are with the content of Pb, Zn, Cu, Fe, As and Cd below the accepted by us hygiene norms for top admissible concentrations.
4. The differences in the content of Pb in the muscles of different types of fish from the river of Danube are insignificant and the values of Pb, as well as As, Cd, Zn and Cu are below the accepted by us hygiene norms for top admissible concentrations of these elements.
5. The investigated samples of meat and meat products contain Pb, Zn, Cu, As, Cd and Fe below the level of the fixed hygienic norms.
6. The content of ^{137}Cs and ^{90}Sr in the investigated samples from green crop and food products from animal origin is within the framework of the background values.
7. The radioactive contamination of the workable soil for ^{137}Cs has average values around 25 Bq/kg, and for ^{90}Sr – 2,5 Bq/kg.
8. In spite of the presence of nuclear power station on the territory of northwest Bulgaria and the resent war in Serbia we may say that the territory in this part of the country is not contaminated with technogene contaminators and the produced vegetal and animal production has no risk for the consumer.

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THE FREQUENCE OF BRUCELLOSIS IN THE SOUTHERN REGION OF ALBANIA IS CAUSED BY FOOD PRODUCT

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INTRODUCTION

Brucellosis is a contagious disease, caused by the Brucellosis type bacteria. It is also known as the Mediterranean disease.

Albania has the biggest number of people being infected by it. This disease is transmitted from animals to people, causing "Malta Fever" to them.
Before 1990, our country was pronounced not to be under the risk of brucellosis. But after 1990, the political, social and economic changes in Albania brought also the improper functioning of the vet and food hygiene structures and due to this, there was also an increase of the brucellosis infection to the animals and mostly to the people.
According to the reporting health sources, actually it is one of the most contagious and dangerous diseases in Albania for the people in the rural and urban areas, alike. This fact is due to the distribution of the food products.
One of the most distributed kinds of Brucellosis in our country, just like all around the world, is Brucella Melitensis.
In this context, the South of Albania, more precisely in the District of Gjirokastra, the number of infected people reaches 30% of all the infected persons all over the country.
During 2001-2007, which will be also discussed in our thesis, the number of the infected persons is 2323, turning it into the most problematic disease.
The purpose of this thesis is to define the reasons of the spread of the brucelar infection at such levels and the measures to be taken to fight this disease of such a high social risk.

Brucellosis

Brucellosis is one of usual bacterial zoo-noses in the world, caused by organisms concerning brucellosis genre.
In Albania, brucellosis has an early origin. The first case in humans was dictated in the year 1925, in Gjirokaster.
Being that even today Gjirokaster is the most problematic case around the country concerning brucellosis infection both in animals and in humans, we took this study for the years 2001 – 2007, only for the fact that brucellosis takes first place among bacterial infectious diseases.
Brucella melitensis is the most spread from brucellas, causer of brucellosis, both in our country and abroad. Cases of the disease in humans appear after eating contaminated food with brucellosis, which

attack mainly the mouth mucus, even the mucus of the nose and the eyes, as well as little cuts and grazes of the skin.
Therefore, in order to know the present situation of brucellosis, analysing it and the general epidemiologic prospective of the brucellas, as well as the measures for protection against them, the reflection of brucellas is necessary, isolated in the Bacteriologic Laboratory of the district Gjirokaster, where brucellosis has been widespread. This disease has double healthy and economical importance in many developing countries, as well as in the developed countries.
Each year, there are about 500 000 cases with brucellosis throughout the world. Latin America, Spain, Greece, Macedonia in Europe, Iran, Iraq, Kuwait in the Middle East are defined as hyper endemic zones with more than 400 cases each year, the cause of which is generally Brucella melitensis.
Mediterranean Countries, in which our country is also included, have a yearly incidence of brucellosis in people 1- 78 cases for 100 000 habitants.
The high scale of the infection in the neighbor countries, the increase of cases with brucellosis in our country, even in the district of Gjirokaster, both in animals and in human beings, were the reasons to have the object of study the dynamics of the spread of brucellas in the Southern Region of Albania, taking a stop at the district of Gjirokaster.
But after the changes of the years 1990, there was noticed a new reappearance and quick increase in brucellosis infection, for the fact that along this period was frustrated the technique structure of the domination of this disease as:
Malfunction with efficasity of the vet service structure.
Malfunction of diagnostician measures.
Unnumbered of positive livestock heads
Absence of controlling farming products
Uncontrolled movements of the farm products, within and outside of the country, especially to Greece and Macedonia.
For these reasons, in epidemiological aspect, brucella's infection is a social warning, because the number of infected people from brucellosis is increasing more and more.
In the Southern Region of Albania in these last years (2001-2007 the years of our study) brucellosis has been one of the most problematic infectious diseases.

The purpose of this study is the representation of as much data as possible in relation with this disease analyzing:

- The spread of the brucellosis among the people in the Southern Region of Albania, stopping in the Gjirokaster district in the years 2001-2007.
- The most affected age groups.
- The spread of brucellosis in the district of Gjirokaster according to sex.
- The sanitary-hygienic measures for the elimination of brucellosis in the future.
- The seasonal character.

General characteristics of Brucellas

Morphology

- Brucellas are gram-
- Head or short scull shape
- In the sizes 1-2 micron length and 0.3-0.5 micron width
- Do not create spores
- Do not have flagella and for this reason they do not move.
- As a rule, they stay lonely, but there are cases when they are found in couples, or create short chains.

Much more the infection is spread by:

- Macerated cheese
- Milk foam, fresh butter
- Sour milk

Infected vegetables from dirty and infected animals urine
In addition, the explosions of the disease inside the family are the result of using the same infected food.

MATERIAL AND THE METHOD

For the period 2001-2007, getting to the study in South Region of Albania and especially in Gjirokaster range, are isolated and identified 2323 cases with brucellosis in all infected and carriers.

Brucellosis is identified across agglutinative proof in glass and across the R. Wright proof. Both these methods are used in the same way to bring out more correctly results.
The material for bacteriological examination attends form the patient that has fevers in wave shapes, transpiration especially in the night, headaches, and other weaknesses. From the vene of his harm is taken 5-7ml blood, which is bringing up in small sterile bottles in laboratory.
In laboratory the blood centrifugalized and is taking out its serum. From its serum is taking with automatic pipette 2 X 2.5 ml serum, which is setting down in an lama separated into two pieces. Then on the serum settled on lama is attached a drop of antigenne from Brucella melitensis or rose Bengal.
Agglutination test on test-tubes, A. Wright, is made for a precise result, defining agglutination titer on the patients' serum.
On two rows of test-tubes the serum dilution is made starting with dilution 1:40 on the first test-tube up to 1:1280 in the 9^{th} test-tube, while the 10^{th} test-tube serves fro the control of the antigen, where no diluted serum is added. 0.25 ml antigen is added in all test-tubes with diluted serum. Antigens are products of the Institute of Hygiene of Tirana. Later, the test-tubes get incubated in the thermostat in the temperature 37°C for 24 hours. Agglutination happens in cases when anticorps are present and we have to do with positive reaction.

RESULT AND DISCUSSION

In Bacteorogical Laboratory in Gjirokaster Range the 2323 isolated cases, with patients and carriers for the years 2001-2007, have resulted brucella melitensis. For the popularity of South Region of Albania, this number is to take on consideration and to estimate the disease causers have to take measures for reduction of cases with brucellosis.
Refering to the number of the infected people and to the fact that this disease is spread even to the town's habitants, including ages under 14 years old, causes fo spreading of this bacterial, present and problematic infection for the population should be appreciated.
From the spread of this disease to the people in the cities and to children in small ages, one of the principal factors in brucellas infection result to be food products, primary milk and his sub-products. After 1990 years, of the Farming denationalized, farming products sale was and is going to be

out of sanitarian hygienic standards and from responsible institutions is often unchecked.

The table below presents the dynamics of the spread of Brucellas in the Southern Region of Albania.

In table 1 is given the dynamics of brucellas for the studied years. As it can be seen from the data, the number of the patients with brucellosis has a considerable growth. Refering to the national data, it results that only in South Region of Albania, the 2323 cases with brucellas constitute almost 33% of cases in all the country.

From the table 2 and the graphic 1 we notice that the months with biggest number of brucellosis disease are: March, April, May, June and July. During these months, milk and unseasoned cheese is produced and sold from small livestock, which is produced from individuals, who in ambulatory way sell via citizens families, without completed sanitarian hygienic conditions. The spread almost in a 6 months period and for more it shows that this disease is not anymore a professional disease. In its spread, it is affected by the economical-social and cultural factors and the non-knowledge of its spread.

From the table 3 and graphic 2, a new phenomenon is noticed in the last years. The migrations of the people from the village to the towns bring with them the disease of brucellosis as a chronic disease. This change is also explained by the fact that while in the village this disease and its risk are well known, in the cities it does not happen the same. In the cities, the origin of farm products and sub products is not known, since part of such products is sold without previous control or certification. In the markets, macerated cheese is sold even in June, thus causing a high frequency of the spread of this disease almost half of the year.

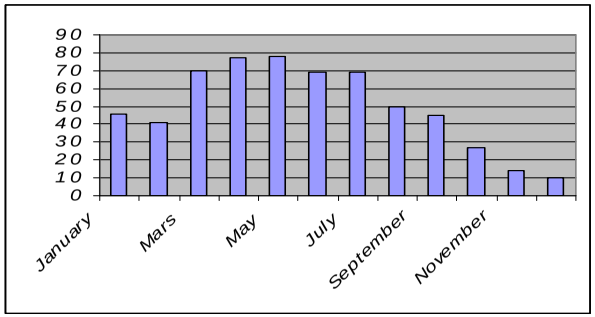
From table 5 and graphic 4, we notice that 6.20% of the infected persons are children under 14 years of age, even 2 and 3 years, which explains the fact that this disease is being spread not only as a professional disease, but also among various strata and age groups of the society. This comes as a result of nonregular higienic-sanitary measures and avoiding of the responsibility of food products contaminated with bacteria.

Table 1
Dynamic of Brucellas melitensis isolated in 2001-2007 years in South Region of Albania.

Years	January	February	Mars	April	May	June	July	August	Sept	Oct	Nov	Dec	TOTAL
2001	15	11	20	28	18	15	16	12	16	10	8	7	176
2002	35	36	40	41	43	46	47	40	34	32	20	16	430
2003	35	35	44	48	48	39	45	35	44	35	27	20	455
2004	19	19	18	41	37	46	40	37	25	25	18	15	340
2005	26	29	42	36	38	33	28	31	30	28	17	12	350
2006	15	17	18	19	28	24	21	23	19	15	11	10	220
2007	23	21	34	29	26	23	19	18	18	19	10	9	250
Total	168	168	216	242	238	226	216	196	186	164	111	89	2323

Table 2
Spread Dynamic of Brucella melitensis isolated in Region of Gjirokastra in 2001-2007 years.

YEARS	January	February	March	April	May	June	July	August	September	October	November	December	Total
2001	8	4	11	20	10	5	8	4	8	3	0	0	81
2002	7	8	12	7	11	18	19	12	6	4	6	2	112
2003	6	6	13	17	17	9	15	4	11	5	4	0	107
2004	7	7	5	11	12	12	13	11	3	3	2	2	88
2005	3	6	12	12	8	11	5	7	7	7	0	2	80
2006	2	5	4	5	13	7	6	8	6	0	2	2	60
2007	13	5	13	5	7	7	3	4	4	5	0	2	68
Total	46	41	70	77	78	69	69	50	45	27	14	10	596



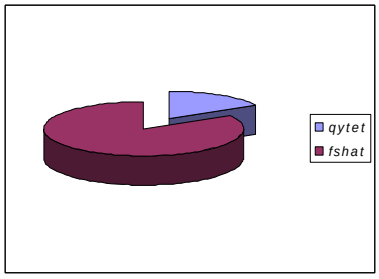
Graphic Nr. 1

Table 3.
Brucella melitensis in town and village in 2001-2007 years in range of Gjirokaster.

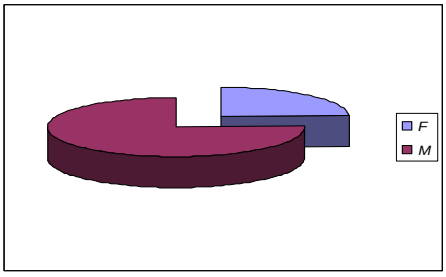
YEARS	2001	2002	2003	2004	2005	2006	2007	TOTAL	%
TOWN	17	25	15	20	13	5	12	97	16.28%
VILLAGE	64	87	92	68	67	55	56	489	83.72%
SUM	81	112	107	88	80	60	68	596	100%

Table 4.
Brucella classified on the basis of sex in the district of Gjirokaster for the years 2001-2007.

YEARS	2001	2002	2003	2004	2005	2006	2007	TOTALI	%
FEMALE	23	26	20	19	22	11	24	145	24.33%
MALE	58	86	87	69	58	49	44	451	75.67%
SUM	81	112	107	88	80	60	68	596	100%



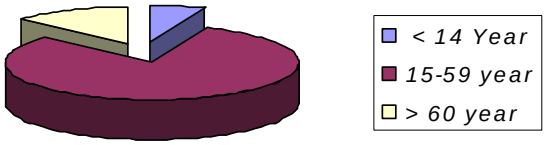
Graphic Nr. 2



Graphic Nr. 3

Table 5.
Brucella on the basis of age groups in Gjirokater.

YEARS	<14 YEARS	15-59 YEARS	60> YEARS	TOTAL
2001	4	72	5	81
2002	3	93	16	112
2003	5	82	20	107
2004	7	68	11	88
2005	6	62	12	80
2006	5	52	5	60
2007	7	52	9	68
SUM	37	481	78	596
%	6.20%	80.70%	13.10%	100%



Graphic Nr. 4

CONCLUSIONS

1. During the years 2001-2007, in the Southern Region of Albania, 2323 cases of sick persons with brucellosis and chronic patients were isolated. We notice a growth as compared to 10 years ago, which shows the

underestimation of the brucellar infection. The distribution of the cases according to the seasons reveals the predomination of the disease in spring, especially during the months, when the number of brucellas is higher, like April, May, June, July, august with 1234 cases or 54%. This is because it coincides with the period of births and abortions, particularly in the sheep, and in the lactation period.
2 As regards the age groups, the most affected from the disease is the age group of 15-60 years.

RECOMMENDATIONS

The work between the medical and veterinary institutions must be intensified and their activity must be coordinated in order to:

- prevent
- diagnose
- and treat the disease on time

For this, a control strategy for the uprooting of brucellosis is required, namely:

- The prevention of the movement of animals and the monitoring of free zones of brucellosis.
- The elimination of positive animals.
- The vaccination of herds.

While in the practical plan, in the relations with the customers, the following must be taken care of:

- The slaughter of animals in slaughter-houses under the supervision of the specialist.
- The sale of necessarily certified animal products and sub-products.
- The close cooperation between the Veterinary Service and the Hygienic-Sanitary Service.

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DEFINITION OF PHYSICOCHEMICAL PARAMETERS OF COW'S MILK

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The purpose of our job was learning of such characteristics as acidity, density of milk and lactose contents in milk. These parameters are main quality indexes of milk.
During this research analysis of crude cow's milk from different human settlements of Grodno region was made. They are Skidel, Vertelishki, Ostrovo. Milk is received from cows of personal farmsteads.
Milk is one of the most valuable foods of a person. On a nutritive value it can replace any product, but any product cannot change milk. Milk

contains all necessary substances for feeding of a person as squirrels, fates and carbohydrates. These substances are in the balanced relations and easily assimilated by an organism. Special biological value offer squirrels of milk which are the most important in biological attitude organic substances. Amino acids which forms in result of proteolysis go on constructing of cells of an organism, ferments, protective bodies, hormones etc.

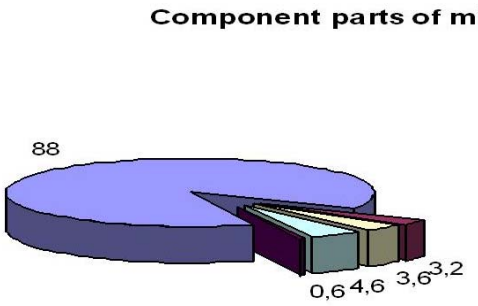
Under contents of essential amino acids squirrels of milk attribute to squirrels of high biological value. Serum milk proteins are especially rich with essential amino acids. They contain more lysine, tryptophan and some other amino acids in comparison with casein.

Great value in feeding of a person has butterfat. Presence in butter fat significant amounts of phospholipids and vitamins (A, D, E) increases its biological value. Besides, in comparison with other fats butterfat is better assimilated by an organism of a person because it has low melting temperature (27 - 34^0C). Also butterfat in milk in emulsify conditions. It means that they are in form of small fat balls.

Milk structure includes valuable carbohydrate lactose (Milk sugar) which used by an organism in the capacity of energy source.

Not less valuable are mineral components of milk. Especially is high content of calkcigerous and phosphorus which are necessary to an organism for creation of bone tissue, renewal of blood, brain activity etc.

Milk also contains macro elements as potassium, sodium, magnesium, chlorine and microelements as zinc, cobalt, manganese, copper iron, iodine which participate in constructing of ferments, hormones and vitamins.



Milk is a constant and important source almost all vitamins.

88- water; 3,2 – squirrels; 3,6 – fats; 4,6 – lactose; 0,6 – mineral substances

Physico-chemical properties of milk

Acidity of milk is primarily stipulated by presence in it of acid salts and proteins. Acidity can be titrable and conditional.

Titrable acidity expressed in standard units such as Turner's grades (^{0}T). Under Turner's grades we understand quantity of cubic centimetres 0,1н solution of a sodium hydroxide which one is extended for titrablne 100 cm3 of milk diluted twice with water. Acidity of fresh milk, on the average, compounds 16 - 180T.

Titratable acidity depends on rations of feeding, breed, age, specific features of an animal etc. Especially strong acidity of milk changed during the lactic period and at diseases of animals. Sometimes one can see rise of acidity of milk received from separate animals and even from the whole flock. It is conditioned by an insufficient quantity of salts of calcium, excess of silo and beet pulp. Rise of acidity of milk is a consequence of mineral metabolism violation in organism of an animal. Sometimes happened cases of decreasing of milk acidity (lower then 16^0T). It happened because of feeding violation of animals.

We defined titratable acidity by two methods: method of acid-base titration and potentiometric titration method. Results of determinations were equal.

Active acidity is expressed by hydrogen ion concentration or pH value (pH). pH value is negative common logarithm of hydrogen ion concentration being in solution

$pH = - lg [H^+]$

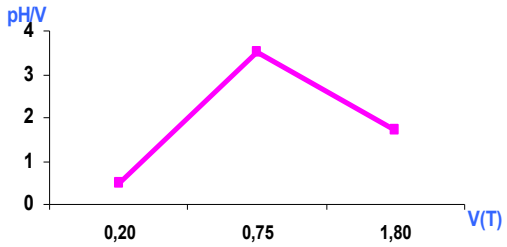
pH value of fresh milk defined by potentiometric method with usage of pH-metre range 6,55-6,75. pH-metre usage is fast enough, simple, exact and convenient method in using.

Results of potentiometric titration:

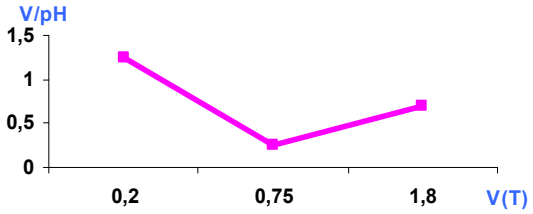
For comparison of properties of raw and sterilized milk we had been tested milk of following producers: Grodno «Milk World» (molochnij Mir), Brest «Savushkin product», Vertelishki.

Research results by defections of acidity of milk are bringing in Tabl.1.

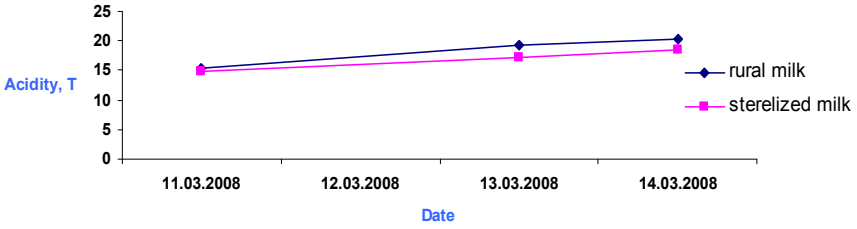
Potentiometric titration



Grant"s curve



Difference in changes of acidity at rural and sterilized milk during the time(Skidel)



Difference in changes of acidity at rural and sterilized milk during the time(Vertelishki)

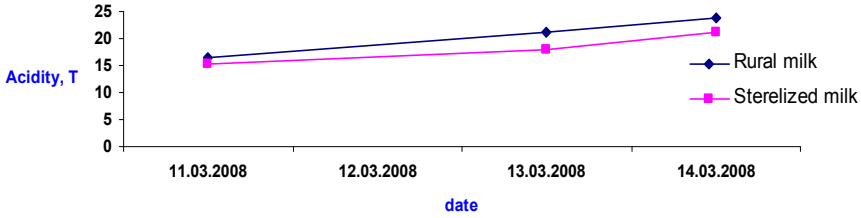


Table1
Identification of active acidity

Vertelishki	pH=6,45
«Milk World» (molochnij Mir)	pH= 6,49
Savushkin produkt	pH=6,43

During experiences were noted that acidity of fresh milk corresponds to legitimate value. It means that the content of vitamins and minerals in feed for cows matches to all norms. At storage of raw milk titratable acidity increases in process of developing in him of microorganisms which ferment milk sugar with formation of milk acid. Acidity of sterilized milk varies at storage. But it is insignificant changes and before finishing of keeping time matches to the norm. After change of keeping time acidity of sterilized milk starts to increase.
Active acidity does not coincide with the titrated. At storage of raw milk titratable acidity vary much faster than active. Therefore titratable acidity is criteria of quality rating of stored milk.

Table. 2

Titratable acidity	16	17	18	19	20	21
pH value	6,73	6,69	6,64	6,58	6,52	6,46

Titratable acidity	22	23	24	25	26
pH value	6,41	6,36	6,31	6,26	6,21

Mismatch of active and titrated acidities speaks about buffer value of milk which caused by contents in it amine and carboxylic groups and mixture of phosphates and citrates.

At storage pH of milk decreased and titratable acidity increased. This explains from the fact that at active acidity definition is allowed only hydrogen ions which are in form of solution. At titratable acidity definition in reaction with alkali enters not only free H^+ - ions but also bound ions.

Density as well as acidity is the relevant physico-chemical value which allows to judge about quality of milk as its value depends on milk structure. Density of milk varies from 1,027 to 1,032 g/cm^3. Size of density depends on the lactic period, diseases of animals, breeds and feed rations. Density of milk determined just after milking is lower then density measured in some hours on 0,8-1,5/cm3. It explains by escape of part of gass and rise of density of fat and proteins. Therefore density of stored milk is necessary to measure not earlier than in 2 hours after milking. At rise of fatness and contents of carbohydrates density of milk decreased. During experiment mentioned dependence was confirmed. Thus density of samples corresponds to the norm.

Table 3

	density, g/cm^3	**fatness, %**	**lactose, ND^{17,5}**
Vertelishki	1,028	3,2	4,49
«Milk World»	1,03	2,5	4,64
«Savushkin product»	1,027	3,3	3,98

Table 4

Identification of lactose and density

	density, g/cm^3	**lactose, ND^{17,5}**
Skidel	1,343	5
Vertelishki	1,3427	4,84
Ostrovo	1,3431	5,05

Specific carbohydrate of milk is lactose. It is disaccharide consisting of the remainders of B-glucose and of B-galactose. Its identification is very

important task. It is single sugar of cow milk (98 %) and its concentration compounds 4,5-5,0 g/100 ml.
We have used a refractometry method to identification of lactose. The procedure of carrying out of given analysis is enough simple and quick. Universality of method gives the chance to spare time and reagents. It is possible to recommend such procedure to usage in analytical labs of dairies.
Milk falsification.
Any deliberate change of structure and properties of natural milk is called Falsification. Following kinds of falsification of milk are possible. It is dilution with water, addition of skim milk or removal of cream, addition of skim milk (double falsification) and addition of neutralizers (soda, amonia) and preservative agents (formaldehyde, hydrogen peroxide) etc. It is impossible to use falsified milk (with addition of water) for production of cultured milk foods, cheese, and preserved milk products.
The most frequent cases of falsification of milk are watering, addition of soda and ammonia.

There are following methods of detection of this falsification:
1. Mix milk and alcohol in the ratio of 1:2. Blend mixture for some time and quickly pour out on a saucer. If milk is not diluted there will be flakes not late than in 5-7 seconds. If flakes occur in larger period milk is diluted by water. And the more is water in milk the more time it is required for occurrences of flakes.
2. Milk with water impurity on border of walls gives the wide blue ring and on a nail will not organize convex drop it spreads. If milk has solid impurities (flour, chalk, potash, etc.) on a nail will be deposit.
3. At suspicion on falsification of collected milk it is easily to establish its naturalness by an indirect way on density. It is considered to be that density of milk accepts approximately on 3 kg/m3 on each of 10 % of added water.

Except water in milk adds starch, chalk, soap, soda, lime, boric or salicylic acids and even gypsum.
To find out presence of these admixtures at milk it is necessary to filter part of milk through a paper filter and to add some drops of any acid, for example, acetic and lemon. False milk in difference from not falsified will bubbles from release of carbonic acid.

All it is done for falsification or for protection from quick sour fermentation. Actually using of these additives does not protect milk from sour fermentation. And that is the most important result of its using often brings to food poisoning.
Some falsifiers in sour milk add sugar that acid taste was not felt.
Starch and flour mix in milk, cream and sour cream for larger thickness. It reveals simply. Milk is more thickness to the bottom of ware. But it is impossible to hide flour and starchy taste of such milk.
If the deposit of this milk to boil ordinary paste will be received. Simultaneously mixed milk becomes blue from impurity of several drops of iodine tincture while pure milk from similar reaction becomes yellow.
By the way there is a concept "renew milk" when dried milk transform back in liquid with help of water and then such milk pour out-on packages or use for production of dairy products. To reveal such falsification is enough simply. Previously it is necessary to measure volume of milk by checked volume measures. Sometimes milk is bottle in smaller volume executed of thick glass.
We have checked milk samples on falsification. In result milk was not falsified.
During experiment we had been used procedure of semi-microanalysis which we wish to tender to analytical labs. This procedure is universal, quick, exact, low-cost and allows to economics reagents.
Differences in acidity changes at rural milk and sterilized milk:

Breeding:
Thus, ground data it has been established that identification of physicochemical parameters allows to estimate naturalness, quality and suitability of milk to processing any dairy products.

PHYSICAL CHARACTERISTICS OF SEA BUCKTHORN BERRIES GROWN IN 2007

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ABSTRACT

Sea buckthorn (*Hippophae rhamnoides* L.) is a native bush to Europe and Asia that produces orange to yellow berries. Its berries have a wide range of uses in medicine and also in food industry.
Harvested sea buckthorn berries proceed to several processing operations which all emphasize knowing all the necessary data about their physical properties like size, shape, weight, moisture content and firmness.
Size and shape showing the uniformity are important features to achieve the highest level of efficiency in processing. Moisture content refers to the juicy of the berries. Force and energy required to rupture the sea buckthorn berries depend on several factors as the strength of the skin, the firmness of the flesh, the viscosity of the juice, the turgid pressure, and the size of the fruit.

INTRODUCTION

Sea buckthorn (*Hippophae rhamnoides*) is native to Eurasia and the plant is noted for its impressive range of uses: for soil conservation, as an ornamental, in the tea industry, and especially as a fruit, which is rich in vitamin C and other nutritional and bioactive compounds (Tang, 2000).
The aim of this work was to determine physical characteristics like size, weight, firmness and moisture content of sea buckthorn berries grown in Estonia in 2007.
The properties of berry vary within the species and therefore the present study concentrates on measuring the physical characteristics of eleven different species of sea buckthorn berries.

MATERIALS AND METHODS

In 2007 several species of sea buckthorn berries grown in Estonia were gathered and analyzed: Avgustinka (AVR), Botanitseskaja (BOR), Botanitseskaja Aromatnaja (BOA), Botanitseskaja Ljubitelskaja (BOL), Gibrid Pertsika (HPR), Hergo (HER), Otradnaja (OTR), Podarok Sadu (PSR), Sirola (SIR), Trofimovskaja (TRR) and Vorobjevskaja (VOR). Most species are from Russia, HER and SIR from Germany.

1. Berry size and weight

Berry size was determined measuring berry basic parameters like width, length and thickness with a micrometer. Amount of berries taken under measurement was 30. Berry shape was calculated and presented as geometric mean diameter D_g (Mohsenin, 1970):

$$D_g = (LWT)^{\frac{1}{3}}$$

For calculating sphericity (Φ) the following equation was used (Mohsenin, 1986):

$$\Phi = \left(\frac{(LWT)^{\frac{1}{3}}}{L} \right) \cdot 100$$ where L is length, W width and T thickness.

Weight was measured with analytical balance as the weight of 30 berries.

2. Berry firmness

Firmness was measured with texture analyzer TA- XT2i (Stable Micro Systems, UK) which evaluates the force required to rupture the sea buckthorn berry. For each test, a single berry was placed onto the plate and compressed with the probe. Experiment was repeated up to 40 berries.

3. Berry moisture content

To measure the berry moisture content the berry had to be comminuted to increase the efficiency and accuracy of results. The procedure was repeated three to four times using halogen moisture- analyzer Mettler Toledo HR83 (Mettler Toledo, Switzerland).

RESULTS AND DISCUSSION

1. Berry size and weight

It is clearly seen in Table 1 that berry size, weight and sphericity vary between different sea buckthorn species, but disparity is not so significant. BOR has the highest parameters: width, length and thickness 9,33mm, 11,92mm and 9,33mm, respectively. The smallest berry is HER with width 6,51mm, length 8,40mm and thickness 6,51mm. Geometric

mean diameter is according to size parameters greatest for BOR and smallest for HER.
About weight, as can be found according to size parameters, BOR is the heaviest and HER is the lightest.
Sphericity as the measure of dimensions for products, which do not have an exactly defined geometric form (Alfonso, 2007), reveal that sphericity does not depend on the weight nor geometric mean diameter. The heaviest and the biggest berry in the present study is BOR, but it does not have the highest value of sphericity, PSR does, 84,9 and 85,0%, respectively. Lightest and the smalles berry is HER, which sphericity is 84,4%, is one of the roundest berry under the observation. BOL and VOR both have the lowest value of sphericity, which is 76,5%.

Table 1. Size parameters, weight and sphericity for different species of sea buckthorn berries

Species	**Width = Thickness, mm**	**Length, mm**	**Dg, mm**	**Weight, g**	**Sphericity, %**
AVR	8,24±0,53	11,70±0,84	9,26±0,57	0,67	79,1
BOR	9,33±0,53	11,92±0,79	10,12±0,55	0,73	84,9
BOA	7,98±0,41	10,77±0,66	8,81±0,39	0,58	81,8
BOL	8,07±0,47	12,05±0,78	9,22±0,46	0,58	76,5
HPR	8,86±0,51	12,44±0,83	9,92±0,55	0,73	79,7
HER	6,51±0,37	8,40±0,45	7,09±0,35	0,30	84,4
OTR	7,97±0,45	10,60±0,75	8,76±0,48	0,49	82,6
PSR	7,81±0,38	9,97±0,58	8,47±0,38	0,53	85,0
SIR	7,92±0,51	10,65±0,68	8,73±0,49	0,48	82,0
TRR	8,53±0,55	12,01±0,67	9,56±0,56	0,67	79,6
VOR	7,85±0,56	11,72±0,84	8,97±0,59	0,65	76,5

2. Berry firmness and moisture content

Data given in Table 2 presents that the minimum values of firmness are not differing significantly staying around 117g, except HER which minimum firmness 162g. BOR and VOR maximum firmness is lower than other species around 256g, while the rest are similar to the average maximum value of 350g.

Berry moisture content also shown in Table 2 refers that moisture content in sea buckthorn berry is from 81,26% to 86,87%, averagely and differs by species. AVR has the highest moisture content reaching to 86,87%. On the other hand, HPR moisture content is only 81,26%.

Table 2
Berry firmness and moisture content accordingly to different species.

	Average value, g	Moisture content, %
AVR	200,16±28,95	86,87±0,81
BOR	148,43±21,32	85,25±0,44
BOA	190,83±27,05	83,58±0,66
BOL	233,52±31,56	83,33±1,58
HPR	208,63±31,12	81,26±0,48
HER	296,62±43,00	81,55±0,60
OTR	271,52±41,87	82,76±0,98
PSR	259,99±41,44	82,82±1,03
SIR	268,86±39,75	82,42±0,54
TRR	181,89±26,87	81,54±0,21
VOR	153,37±24,02	83,16±0,11

Discussion

The study revealed that Russian originated sea buckthorn berries are slightly bigger than German species HER and SIR which are one of the smallest berries under the research.
The widest berry is BOR- 9,33mm, the longest is HPR- 12,44mm and the thickest is also BOR- 9,33mm, which has also the greatest geometric mean diameter 10,12mm. The smallest berry among the others is HER, which width, length and thickness are 6,51mm, 8,40mm and 6,51mm, respectively. Generally, length of berries studied in the present research is between 8,40 and 12,44mm, width and thickness are in range of 6,51-9,33mm. Compared to results from scientific literature of sea buckthorn berries grown in Estonia in 2006, the berries have gone slightly shorter and thinner. In 2006 berry length was from 10,64 to 13,71mm, width and thickness from 7,79 to 9,03mm.

Weight of berry in the present study was between 0,30 and 0,73g, where the lightest berry was HER and the heaviest BOR, respectively. Compared to data from the year of 2006 when the weight of the berry was 0,49 to 0,85g, which is higher value than the present berries have.
Sphericity of sea buckthorn berries in the present study is between 76,5 and 85,0%,
BOL and VOR both have the lowest sphericity, PSR has the highest value of sphericity, respectively.
Firmness of berry depends on strength of the skin, the firmness of the flesh, the viscosity of the juice, the turgid pressure of the fruit and the size of the fruit (Khazaei, 2004) and that may be the reason why HER with the most discreet proportions, shows the highest firmness value which ranged from 162,06 to 374,51g. BOR which was the biggest berry, shows firmness from 106,81 to 256,13g. Generally, the firmness was between 154,67 and 282,21g, averagely.
Observing the results of the present study and Graphic 1, it can be said that there is a dependence between the geometric mean diameter and firmness of the berry. The smaller the berry geometric mean diameter is, the higher firmness value it has.
Moisture content in analyzed berries was in the range from 81,26 to 86,87%. The highest moisture content was in AVR and the lowest in HPR. Average moisture content taken all species and samples into consideration was 83,10%. Compared to results of year 2006, moisture content has decreased from the level 84,9% to 83,10%.
The present research shows that sea buckthorn berries have different physical characteristics depending on the variety of species and through years.

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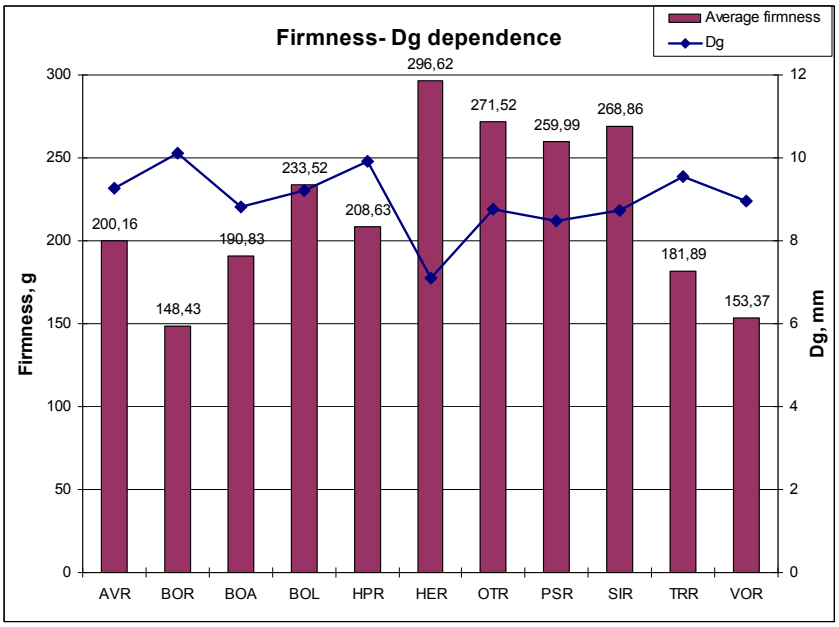

Figure 1
Berry firmness and geometric mean diameter dependence.

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APPLICATION OF TEXTURE ANALYZER IN THE QUALITY ANALYSIS OF BAKERY PRODUCTS

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INTRODUCTION

Bread is a basic foodstuff for the Center European people. There are several traditions on bread making in Hungary but the consumers and the industrial bread production requires a stable product quality. Because of the different quality parameters of the applied flours several additives used in production to stabilize bread quality, both in taste and texture.
Texture analysis is primarily concerned with the evaluation of mechanical characteristics where a material is subjected to a controlled force from which a deformation curve of its response is generated. These mechanical characteristics in food can be further sub-divided into primary and secondary sensory characteristics which have proven to be correlated to sensory perception. The primary characteristics parameters are the hardness, springness, adhesiveness and cohesiveness (Figure 1.) (Szczesniak et al (1963). Bourne (1978))
Texture analysis is an objective physical examination of baked products and gives direct information on the product quality, oppositely to dough rheology tests what are inform on the baking suitability of the flour, as raw material (Baik and Chinachoti, 2000; Charson and Sun, 2001; Szczesniak, 2002). This is why the texture analysis is one of the most helpful analytical methods of the product development, as it is suitable to quantify the effects of flour blends and additives on physical properties of crust and crumb of the breads. It is also suitable to examine the effects of storing and freezing on different sensory properties of these products and thence it is suitable to analyze the results of different recipes in product development.

In this study we have examined the possibilities of texture analysis on different (commercial and experimental) bread products to explore the possibilities of this equipment in product development.

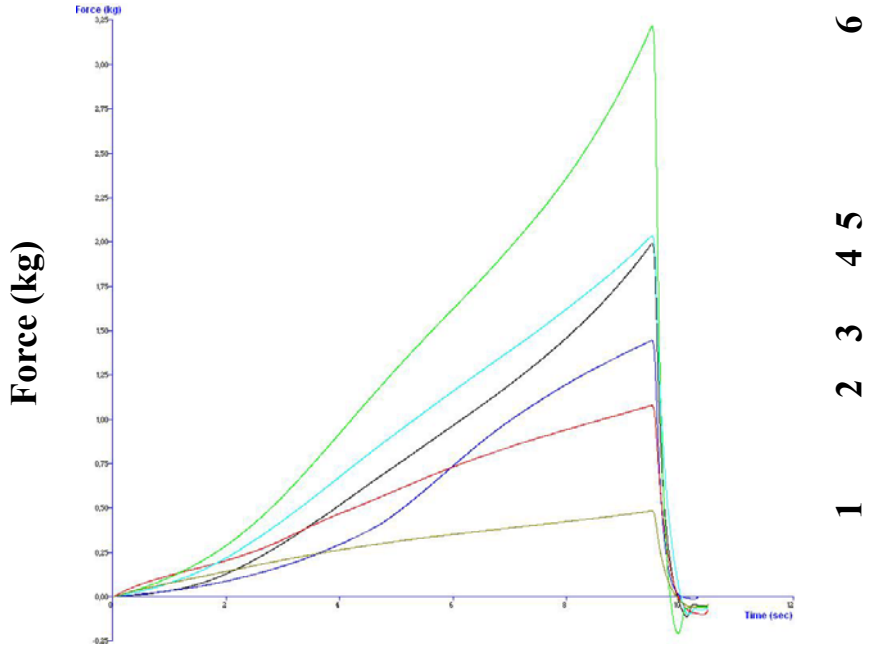

Figure 1
Results of texture analysis on commercial bread samples.

biscuit flour	**(6)**
strudel flour	**(5)**
bread from BL55	**(4)**
test loaf from BL80	**(3)**
white bread from shop	**(2)**
semi-brown bread from supermarket	**(1)**

MATERIAL AND METHODS

The analysed commercial bread and other bakery products were from a shop of an international hypermarket and from a shop of an entrepreneur.

Six different samples were examined in this case: bread from BL55 (aestivum wheat flour with 0,55% ash content, recommended for pie) and BL80 (aestivum wheat flour with 0,8% ash content, recommended for white bread) flour, cake flour, white bread from a small shop, white and semi-brown bread from a hypermarket. In the second case a by product, dried apple pomace was milled to flour and added in 10, 20 and 30 % ratio to white bread flour to increase the fiber content of bread made from it. The crude protein content of these samples was determined by MSZ EN ISO 5983-1:2005., crude fiber, starch, dextryne, all and soluble carbohydrate and sugar content were determined by MSZ 6369-12:1979., crude fat content was determined by MSZ 6830-6:1984.Texture analysis was made by TA-XT Texture analyzer (Stable Micro Systems Ltd, Surrey UK).

RESULTS AND DISCUSSIONS

The results of texture analysis of different commercial bread products can be seen on Figure 1. In Hungary the bread from white flour (BL80) is the commonly consumed bread. It is visible that it has relatively low hardness. More than 25% hardness was shown by the test loaf made from BL80 what verifies that the industrially used flour additives decrease the hardness of crumb. In accordance to our expectations the application of smaller degree of meal results much harder dough and much harder bread crumb structure; so the breads made from BL55 wheat flour (recommended for pie) and from strudel flour have twice hardness as white bread has. The biscuit flour resulted bread with the highest hardness from the examined raw materials; its crumb has threefold hardness as white bread had. The 6th examined sample was a semi-brown bread made in the bakery of an international supermarket. This sample has the much weaker crumb; its hardness was half of the white bread made in a local bakery.

In the second experiment our aim was to increase the fiber content of bread by the addition of a low price by-product of juice processing. We have tried to find that apple pomace concentration what is high enough to decrease significantly the energy content of bread but low enough to keep the original structure of crumb. It can be seen on Figure 2 that 10% pomace addition did not changed the measurable hardness of bread, but 20% has caused about 15-20% increase of hardness.

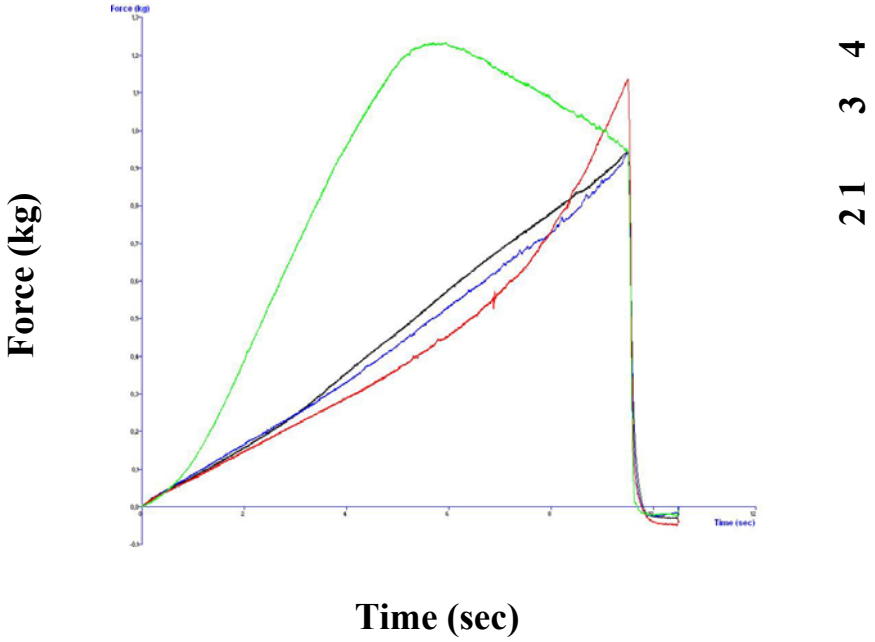

Figure 2
Results of texture analysis on breads from aestivum flour and apple pomace mixtures

30% apple pomace addition (4)
20% apple pomace addition (3)
Control (2)
10% apple pomace addition (1)

These results had agreed to the results of sensory analysis: the 10% pomace addition did not caused significant sensory effect on the test consumers, but 20 and 30% addition increased the density of crumb as much as the reaction of consumers was negative. On the other hand, chemical analysis proved that 10% pomace addition has almost the same effect on energy content as 20% addition while the increase of carbohydrate and protein content continuously decreased, fiber and fat

Table 1
Chemical and physical properties of test loafs made from apple pomace and white bread flour mixtures

	Protein content, %	Total carbohydrate %	Fat content, %	Ash content, %
Control	8,99	43,01	0,406	0,439
10% apple pomace addition	8,48	38,76	0,715	1,792
20% apple pomace addition	7,96	37,46	1,151	3,345
30% apple pomace addition	7,49	32,19	1,358	4,823

	Energy, kcal	Loaf mass, g	Loaf volume, ml	Hardness (kg)
Control	330	341	947	941
10% apple pomace addition	305	340	762	937
20% apple pomace addition	299	347	630	1147
30% apple pomace addition	267	347	497	1270

content continuously increased by the percentage of added apple pomace (Table 1.).

Conclusions

Texture analysis is suitable analytic method to compare bakery products and, after several reference data, may be suitable to determine the type of unknown samples. The results of instrumental measures are comparable to sensory analysis and it can result numerical results for the further statistical analysis.

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FOOD SAFETY AND FOOD PHYSICS – ASPECTS IN FOOD PROCESSING AND QUALITY CONTROL

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ABSTRACT

The paper deals with some special questions of aspects of food safety and application of methods of food physics in food processing and in food quality control and assurance, as well (Figure 1). The role of food safety has developed significantly in the last decades, so today the production and processing of safe and quality food is of primary importance. Modern food production is based on principles of GAP and GMP (including GHP) and different methods and tools for quality assurance (HACCP, ISO-9000, ISO-22000, TQM).

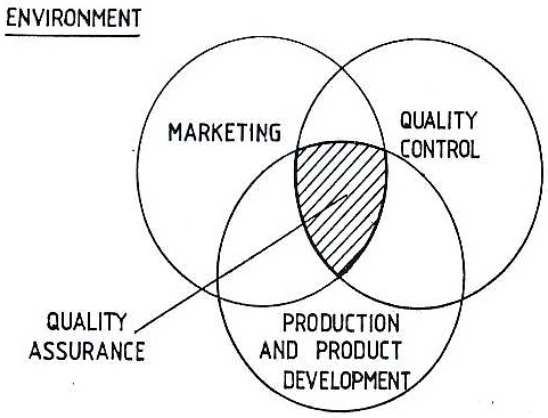

Figure 1
Fod processing and in food quality control and assurance.

There are many techniques and methods to produce safe food and to control the quality of foodstuffs. The modern, up-to-date technologies and measurement techniques involve the application of different physical methods – high pressure, pulsing electrical field, non-destructive techniques (e.g. INAA, NMR, NIR-NIT), radiation treatments - as well. Using radiation technologies (not only nuclear techniques) it is possible to fulfil some important expectations of modern food processing, e.g. decrease of microbial contamination, improvement of sensory properties, increase of storability of products.

Food physics as a bridge, between applied physics and food science

DISCUSSION

Factors determining the quality of food products

Basic requirement: safety (no harmful effects)
- Sensory properties, value of pleasure
- Quantity, volume
- Chemical composition
- Packaging, labelling
- Special (microbiological, toxicological, radiometrical) parameters

Rank of Hazards from Eating Food

The Experts	The public
1. Microbial safety	1. Pesticides
2. Over-nutrition	2. New food chemicals
3. Non-microbial safety	3. Chemical additives
a) contaminants	4. Familiar hazards
b) natural toxins	a) Fat & cholesterol
c) agrochemicals	b) Microbial spoilage
d) food additives	c) Junk foods

Food processing and food quality control

Food production is based on principles of GAP, GMP, GLP and GHP (Figure 2).

GAP – Good Agricultural Practice
GMP – Good Manifacturing Practice
GLP – Good Laboratiory Practice
GHP – Good Hygiene Practice

Quality control and assurance is based on principles of HACCP (Hazard Analysis Critical Control Points), ISO-9000 standards, TQM (Total Quality Management).

Safe food: from farm to fork in the whole chain
Safe food and quality food is not equal!
Quality food is safe, but safe food can be not quality food (e. g. not good sensory properties)

3 main topics:

- physical parameters of foodstuffs
- physical methods for investigation of foodstuffs
- physical methods for treatment and processing of foodstuffs

Modern food technologies and food measurements are based on the principles of physics and physical methods:

- dehydration
- freezing
- lyophylization
- high pressure
- ohmic processing
- pulsing electrical fields
- magnetic fields
- nondestructive techniques (e.g. NIR-NIT, NMR, PAS)

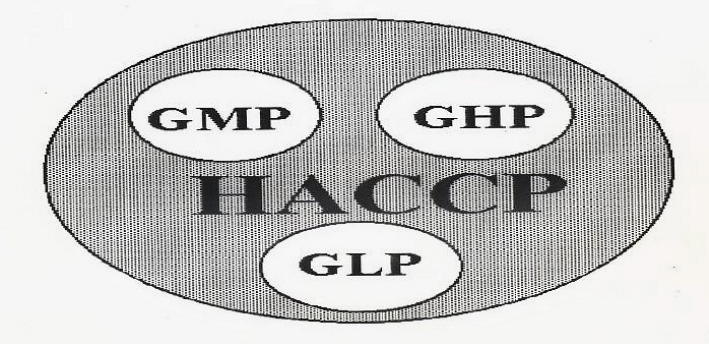

Figure 2
Components of Quality Assurance and connection
between HACCP and GMP

Radiation methods and techniques in the food sector

- Ionizing radiation techniques and technologies (gamma-sources, X-ray equipments, electron accelerators)
- Non-ionizing radiation techniques (light-technique, IR, UV, Laser, SYNERGOLUX: UV+ozone, polarized light in radiostimulation)
- Isotope techniques, tracer techniques
- Radio-analytical techniques (e. g. AA)
- Measurement techniques (quantity, level, thickness etc.)
- Radioecology

Expectations in modern food processing

- Decrease the microbial contamination, disinfection
- Increase the storability
- Improve the sensory properties
- Apply environment-friendly and economical technique

Important standards

ISO 9000 (International Standards for Quality Management)
ISO 14000 (International Standards for Environmental Management)
ISO 22000 (International Standards for Food Safety Management)
ISO 9000-2000 (combination of ISO 9000 and TQM)

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THE MANAGEMENT OF RISKS IN THE EDIBLE OILS INDUSTRY

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ABSTRACT

Risk Management System is an important part of the developing and executing of a business plan in the food industry. Effective implementation of Risk Management System promotes best practice concepts at the corporate/strategic level as well as a improving of technological operation. A proactive approach of the Risk Management System should form a core part of the decision-making process at all levels within an productive organization.
The European legislation in the field of industrial risks management identify two types of risk:

- the chronic risk (integrated control and prevention of pollution).
- the accidental risk: fires, explosions, noise, air pollution, water, waste pollution resulting from the radioactivity.

The objective of this paper research is to define a Risk Management System in the edible oils industry and to identify the axes of reforms to conduct at the prevention of the industrial risks.

INTRODUCTION

Assessing and managing environmental risk in productive industrial fields [3] has been considered to some extent by Richards (1997) and Turner et al (1994). While Richards examined the commercial investment perspectives of risk, especially contamination Turner's work was from the perspective of using environmental management systems as a means of reducing exposure to the environmental risk.
The industrial risk are the followings[1]:

- industrial chronic risk (all forms of pollution which present a citizens health impact and on the environment) ;
- accidental industrial risk resulting from the presence of products or risks processes which might cause an accident involving serious immediate consequences for the staff, the citizen and the environment.

The European legislation deals with these two types of risk as a distinct:

- the chronic risk (integrated control and prevention of pollution).
- the accidental risk (the control of dangerous: fires, explosions, noise, air pollution, water, waste pollution resulting from the radioactivity[2].

The facilities of modest capacity are regarded as having low risks and they must meet the general technical requirements. The facilities of capacity more important are considered of the high risk and are therefore are the subject to the legal institutional authorization regime[4].

The Risk Management System[5,6] sets conditions for development and exploitation of the installation, analysis and measurement of the operation, the methods and means of intervention in case of accident, the emergency measures to adopt and obligations of the operator in terms of information and warning of the personnel.

RESULTS AND DISCUSSION

The general issue of the Industrial Risk Management are the followings:

- Industrial Hygiene;
- Air Sampling;
- Indoor Air Quality;
- Ergonomics;
- Chemical Safety;
- General Safety;
- Fire Safety ;
- Health Care Safety;
- Hazardous Waste Compliance ;
- Environmental Compliance;
- Environmental Audits.

European standards and legislation that must be applied in the oil industry are the followings:

- 97/23/EC: Pressure Equipment Directive (PED)
- 87/404/EEC: Simple Pressure Vessel Directive

- 99/36/EC: Transportable Pressure Equipment Directive (TPED)
- 98/37/EC: Machinery Directive
- 95/16/EC: Lifts Directive
- 73/23/EEC: Low Voltage Directive
- 94/9/EC: Directive for Equipment and Protective Systems intended for use in potentially Explosive Atmospheres (ATEX)
- 89/686/EEC: Personal Protective Equipment Directive
- 90/396/EEC: Directive for Appliances burning gaseous fuels
- 92/42/EEC: Hot Water Boiler Directive
- 93/42/EEC: Medical Devices Directive
- 2000/14/EC: Directive for Noise Emission in the environment by equipment for use outdoors
- ATEX Directive 99/92/EC regulating the minimum requirements for safety and health of workers in explosive atmospheres.
- ATEX Directive 94/9/EC concerning safety of equipment and safety systems to introduce equipment for explosive environments into the EU.

Pressure Equipment certification is realized for the equipment used in the edible oil industry with the followings items controlled:

- Verification of calculations/drawings before construction;
- Verification of welding files and material certificates;
- Inspections during the construction phase;
- Witnessing of hydrostatic testing;
- Stoking tests on steam equipment;
- Safety device and connection controls;
- Periodic visual controls both inside and outside;
- Determination of intrinsic boiler water quality.

The ATEX Directives aim to facilitate free movement and trade of equipment and safety systems intended for use in potentially explosive atmospheres by the following basic principles:

- Dividing hazardous areas into zones with regard to the frequency and duration of an explosive atmosphere;
- Preparing the documentation about explosion safety;
- Proposing adaptations and assessing working equipment and locations in order to ensure they comply with the regulations
- Preventing the generation of explosive atmospheres;
- Preventing the ignition of explosive atmospheres;

• Limiting the harmful consequences of an explosion in order to guarantee the well-being of employees.

Non-Destructive Testing (NDT) is an indispensable service used in Risk Management System evaluation. NDT includes several methods of examining materials, components and connections in order to identify and quantify material defects and degradations.

1. Conventional Testing Methods
- Radiographic Examination (RT),X-Ray or Gammagraphic Testing;
- Manual Ultrasonic Testing (UT)(Pulse-echo Method);
- Surface Examination using Magnetic or Penetrate Testing.

2. Special Examinations
- Eddy Current Testing;
- Guided Wave Examination;
- Positive Material Identification(PMI), Alloy Analysis;
- Time of Flight Diffraction Examination (ToFD; Mechanized Ultrasonic Testing);
- Corroscan (Mechanized Pulseecho Examination);
- Infrared Thermograph (tracing of heat loss);
- Magnetic Flux Leakage (MFL) Tank Floor Examinations;
- Endoscopy, Videoscopy;
- On-site Hardness Measurements;
- Hydrogen Induced Cracking Examination (HIC);
- Digital radiography as on stream technique to determine remaining wall thickness;
- Hot Hydrogen Attack Examination;
- Automatic Ultrasonic Testing (AUT) of Pipeline Girth Welds.

The policy of controlling industrial risk is related with the following fields:

1. Improving the management of urbanization in the area of industries at risk ;
2. the analyses and critical review of the industrial risks studies in the general industrial field;
3. consistent risks study report ;
4. implementation of the internal Management system of security ,including the Management system for the intervention in case of accident and Professional Employees management for the risks prevention ;
5. Implementation of the periodically internal &independent audit ;

6. The feed-back implementation and information dissemination, creation of a data banks easily accessible and efficient utilizable;
7. Civil and penal responsibility of the industrial sites .

The principal elements of Risk Management System:

1. a better identification and reduction of industrial risk to the source;
2. improving the role of the human factor and particularly of employees in the prevention of accidents,
1. safety design of facilities, an important part of their maintenance procedure;
2. create a scale of risk to the assessment of industrial accidents on the whole of classified installations ;
3. lead a reflection on the necessity of products "dangerous" and the manufacturing process "at risk" ;
4. reconsider the storage conditions ;
5. develop technologies to the lower risks ;
6. develop the establishment of management systems of security and the audits of internal security.
7. Public Information: establish a permanent dialog between industrial, governments and citizens, inform the citizen to assure it's own safety, regular days "Open Doors" activities, develop a genuine culture of risk, popularize the technical information of industrial risk, popularize the studies of risks, frequently updating information brochures.

Conclusions

The Risk Management System included three parts:

1. Improving the management of urbanization in the area of industries at risk ;
2. Industrial Risks Analyses System implementation in each industrial plant;
3. Management system of security modern and efficient.

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ELECTRICAL IMPEDANCE SPECTROSCOPY AS A POSSIBLE NONDESTRUCTIVE METHOD IN QUALITY ASSESSMENT

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ABSTRACT

The magnitude and phase angle of impedance were measured with a HP 4284A LCR meter on Gala apples purchased on the local market. Two

ECG electrodes (Fiab Spa) were applied on apple with skin and without skin at several places on the apple surface along the equatorial. The good electrical contact between the electrodes and apple was realized with a conducting gel. The impedance spectra were determined in frequency range from 10 Hz till 1 MHz at 1 V measuring voltage. Each measured spectrum after an open and short correction was approached by a circuit model consisting of a serial connection of impedance of apple skin and impedance of intracellular and extracellular apple flesh. The complex non-linear least squares method was applied with Mathlab program. This approaching method can allow getting the impedance of apple flesh under the skin without peeling.

INTRODUCTION

The electrical impedance spectrum - in low frequency range - of biological tissues depends on the state of cellular structure (Grimnes and Martinsen, 2000; Vozáry et al., 2007), therefore the parameters evaluated from measured spectrum can be used for quality assessment of fruits and vegetables (Vozáry et al., 2007, Harker and Maindonald, 1994). Recently there is a great demand on non-destructive investigating methods. In this work an attempt was made to determine the impedance parameters of apple tissue from the impedance spectrum measured on the whole apple with skin. The impedance spectrum measured on whole apple with skin was approached with impedance of model circuit consisting of serial resultant of apple skin impedance and apple flesh impedance. The model parameters, characterizing the apple skin and flesh were evaluated.

MATERIAL AND METHOD

Gala apples were purchased on the local market. Impedance along the equatorial (Fig.1.) was measured at two electrode arrangements on apples with and without skin. At first, the two electrodes were same distance – the diameter of the apple - far from each other: the two electrodes were at 1 and 7 point, at 2 and 8, 3 and 9, 4 and 10, 5 and 11, 6 and 12, respectively. At the second measurement one of the electrodes was at 12 point and the other electrode was placed to each point from 1 to 11.
The magnitude and the phase angle of impedance were measured with a HP 4284A LCR meter in frequency range from 10 Hz till 1 MHz at 1 V

measuring voltage. The apple surface, with skin and without skin, along the equatorial was touched with two ECG electrodes (Fiab Spa). The good electrical contact between the electrodes and apple was realized with a conducting gel. Each measured spectrum after an open and short correction was approached with impedance of an electrical model circuit consisting of a serial resultant of apple skin and apple flesh impedance.

$$Z = R_o + \frac{R - iR^2C\omega}{1 + R^2C^2\omega^2} + \frac{R_1}{1 + i(\tau_1\omega)^{\psi_1}} + \frac{R_2}{1 + i(\tau_2\omega)^{\psi_2}} \qquad (1)$$

The impedance of apple skin was modelled with a parallel RC circuit:

$$\frac{R - iR^2C\omega}{1 + R^2C^2\omega^2}, \qquad (2)$$

where R and C are the resistance and the capacitance of apple skin. The impedance of apple flesh was approached with serial resultant of two distributed elements corresponding to extra cellular and intracellular part of tissue:

$$\frac{R_1}{1 + i(\tau_1\omega)^{\psi_1}} + \frac{R_2}{1 + i(\tau_2\omega)^{\psi_2}}. \qquad (3)$$

The R_1 and R_2 are resistances, the distances between the two intersections of locus curves with real axis, τ_1 and τ_2 are relaxation times, and ψ_1 and ψ_2 exponents characterize the distribution of relaxation times.

R_o is the resistance of apple at high frequencies.

The complex non-linear least squares (CNLS) method was applied in curve fitting with MathLab program. The impedance parameters: the resistances, the capacitance, relaxation times, exponents were determined, too.

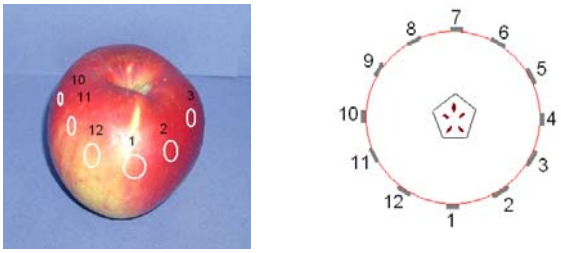


Figure 1
The places of electrodes during impedance measurement on equatorial of apple

RESULTS AND DISCUSSION

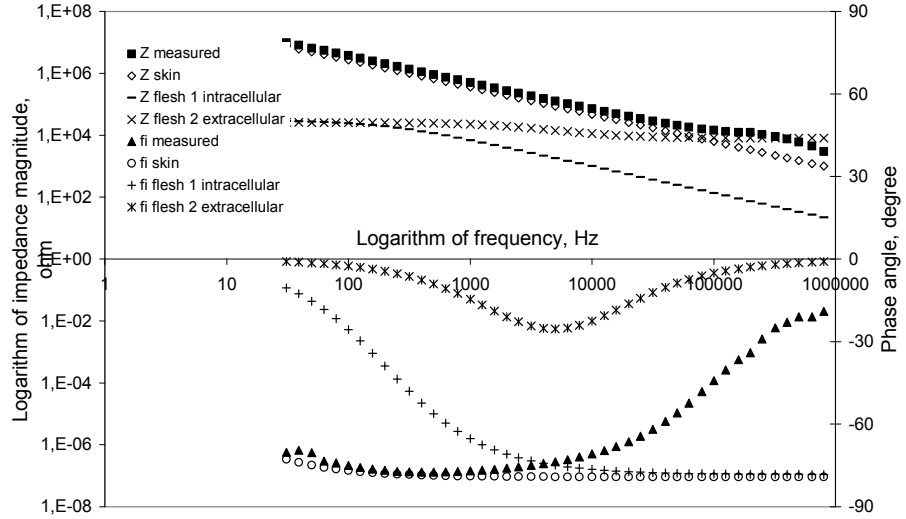


Figure 2
A typical spectrum of magnitude and phase angle of
Impedance measured on apple with skin and the result of approach.
The measured spectrum can be expressed with sum of impedance
of skin, extracellular and intracellular part of apple flesh.

The CNLS method can allow getting the impedance of apple flesh separately from the impedance of apple skin using the measured spectrum of whole apple with skin (Fig.2.). The impedance of apple flesh can be described well with resultant of extracellular and intracellular impedance (equation 3). Really, it is known for other living tissues, too, that the impedance of intracellular part differs from the impedance of extracellular part (Grimnes and Martinsen, 2000). For example, the impedance of nectarine fruit can be represented by such model circuit, which contains the extracellular resistance, capacitance and the intracellular resistance, capacitance, too (Harker and Maindonald, 1994).
The impedance locus curve measured on whole apple with skin gives good agreement with the approaching curve (Fig. 3.)
The value of impedance measured on apple with skin is highest, when electrodes are near to each other. On the other hand the highest impedance on apple without skin can be measured, if the electrodes are

far from each other (Fig. 4). These results can be explained by the different ratio of current length to current cross-section on the surface of apple, where the skin impedance is determinative, and inside of whole apple, in apple flesh. In the case of peeled apple the current goes through interior of apple.

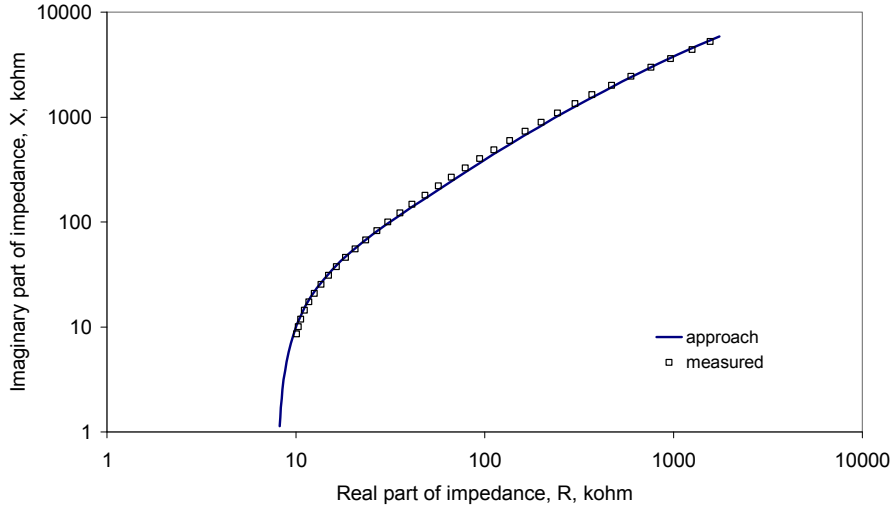


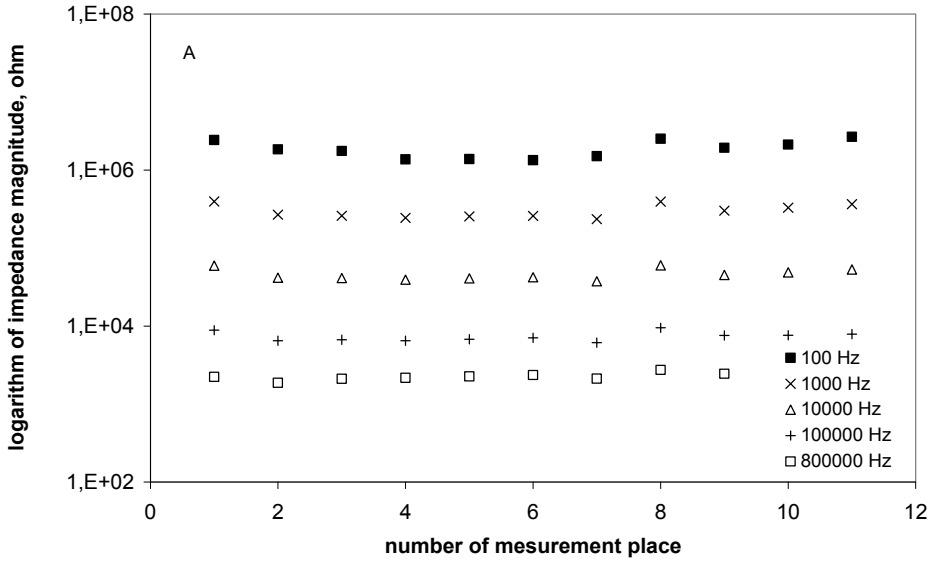
Figure 3
The impedance spectrum measured on whole apple with skin and the approached curve got with equation (1)

CONCLUSION

The CNLS approaching method can allow getting the impedance of apple flesh under the skin without peeling.

ACKNOWLEDGEMENT

This work was supported by Hungarian Research Fund (OTKA, grants T 042911)



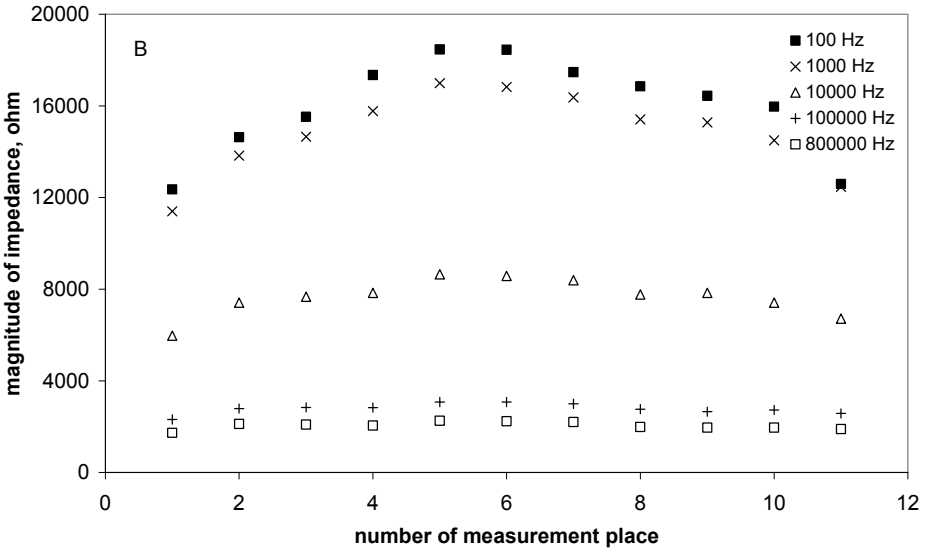


Figure 4
Impedance of apple with skin (A) and without skin (B) measured at various places on equatorial of apple and at various frequencies.

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EFFECT OF SOY FLOUR, CORN FLOUR AND GLUTEN ADDITION ON QUALITY CHARACTERISTICS OF BREADING

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Bread crumbs are typically derived from bread which have been either dried or toasted and are often used in the food industry to enhance the fried-like texture that consumers typically enjoy. In this study, soybean flour (5,10, 15%), corn flour (5,10,15%) and gluten (3,6,9%) were added to bread formulation. Effect of soybean flour, corn flour and gluten on quality characteristics of dried bread crumb were evaluated. Moisture, protein, color, water binding capacity, oil absorption, and compressive force of dried bread crumbs were determined. Addition of gluten increased water binding capacity especially for 6% and 9% levels. On the other hand, both corn and soy flour addition decreased water binding capacity in each particle size in comparison with control sample. Gluten added breading absorbed less oil than corn and soy flour added breadings. 15% soy flour addition considerably increased compressive force while gluten addition reasonably decreased the compressive force as compared with control sample ($p<0.0001$).

INTRODUCTION

The term ‘breading’ can be described as the flour produced from ground bread that is applied on foods to generate a coating with desirable texture (Dyson 1992). Breadings are used to provide texture, flavor, color and appearance. They form a protective layer on the surface of the food and inhibit the transfer of moisture and fat between the sample and the frying medium (Mallikarjunan et al. 1997). Some specific functions are desired during elaboration of breading formulation, such as reduction of oil absorption during deep-frying, cohesiveness and adhesiveness to several sorts of food surfaces, development of desirable sensorial characteristics such as crispness, color and appearance and nutritional value (Dyson 1992; Triveli 1993).

Crispness is the most critical coating characteristic of breaded fried foods. Consumer demands for crisp, breaded fried foods have driven researchers to quantify this attribute and develop techniques that aid in extending product crispness. Incorporation of different ingredients into breading formulations is one of the most promising methods for enhancing product crispness (Tameshia et al. 2006)**.** Wheat flour gives flavor and texture to the product, and its main components- starch and protein- play a structural role in technological properties (Loewe 1992). Corn meal develops good texture, flavor and aroma for the final product, minimizes color variation and density, diminishes water absorption of frozen product during storage, and decreases oil absorption during frying (Burger 1992). Vital wheat gluten increases viscosity of breading, improves texture and crispness of the final product because of the formation of a matrix during the process (Kalin 1979; Loewe 1992), and decreases solid losses during deep- frying (Parinyasiri et al. 1991). Moreover, the addition of gluten is associated with greater adhesion and better structure and texture (Breuil, 2001).

Water binding is the ability of an ingredient to contribute to the gel formation of firmness when water has been added. In foods, the term ‘water binding’ is often used to convey a general tendency for water to associate with hydrophilic substances including cellular materials. Increased water binding capacity results in better taste, softer crumb, delayed staling and longer keepability (23).

Dogan et al. (2005) studied the effects of soy and rice flour addition to the batter formulation on quality of deep-fat fried chicken nuggets. Salvador

et al. (2005) evaluated addition of different ingredients on the characteristics of a batter coating for fried seafood. Maskat and Kerr (2002) studied coating characteristics of fried chicken breasts prepared with different particle size breading.

The studies carried out on breading quality usually include addition of ingredients to the batter formulation. However, limited work has demonstrated the use of edible ingredients in breading formula. In this study, we replaced wheat flour with different levels of soy flour, gluten and corn flour to evaluate the effects of this ingredients on breading in breaded, fried chicken breasts.

MATERIALS AND METHODS

Materials

Wheat flour (0,55% ash, 10,7% protein) and soy flour (54% protein, 1,5% fat) samples were obtained from a local market, in İzmir. Corn flour was obtained from Teknik Tarım Ürünleri Ticaret ve Ltd Şti. Gluten was supplied from Orion Nişasta ve Kimya A.Ş.San.ve Tic.Ltd. Şti.

Bread Crumb Production

Breading was produced by bread making process (Uluoz 1965). Bread samples were cut into slices and conventional oven drying was conducted at 75 ^{o}C by using a commercial electrical oven. Bread crumbs were dried under 8% moisture content and ground using KT-3303 Laboratory Mill. After grinding, the breading was classified into two particle sizes; small (<670 μm) and large (>670 μm) Samples of two different particle sizes were stored in air tight containers.

Chicken Breast Preparation

Chicken breasts were purchased from a local market, in İzmir. Chicken breasts were battered, breaded and deep-fat fried for 240 s at 160 ^{o}C. Batter mix was prepared by mixing corn meal, wheat starch and modified potato starch (Emflo 991, E 1414) at a ratio of 1:1:1 by weight. 50% (w/v) water was added to the batter mix to prepare the batter solution. Chicken breasts were immersed in batter solution for 15 s, drained for 15 s, breaded and fried in 2,5 L of corn oil at 160 ^{o}C for 240 s using a fryer.

Methods

Moisture content of breading samples were determined according to ICC Method 110/1 (ICC, 1982). Water binding capacity was determined according to AACC Method 56-20 (AACC, 1995). The color of the

samples was measured using Minolta Chroma-Meter CR-310 Reflectance Colorimeter (Osaka, Japan) based on the L^*, a^*, b^* system (Hunter 1973). Oil absorption of coatings were determined by weight difference before and after frying.

Resistance to compressive force was determined using TA-XT Plus Texture Analyzer (Stable Micro Systems, England) equipped with a 50 kg load cell. Compression test was determined according to Maskat and Kerr (2002) with some modifications. A cylindrical sample holder with inside diameter of 5 cm was used to hold the samples. Coatings were peeled from the fried pieces and placed into the sample holder to a height of 15 mm. A cylinder probe with a diameter of 35 mm was used to compress the samples at a rate of 1 mm/sec. The force required to compress the samples over a distance of 7,5 mm (50% of the initial height of the samples in the sample holder) was recorded. The breading samples were analyzed 10 minutes after frying.

RESULTS AND DISCUSSION

Breading samples were dried to under 8,0% moisture content as indicated in Tireki et al. (2005). Moisture content of the breading samples changed between 6,69 and 8,22%.

Water binding capacity of the breading samples formed from small particle size changed between 2,64 and 3,45 g/g, while breading samples formed from large particle size ranged from 2,64 to 3,18 g/g (Table 1). Large particle size breadings bound less water than small particle size. As long as small particle size breadings were concerned, addition of gluten increased water binding capacity especially for 6% and 9% levels. On the other hand, both corn and soy flour addition decreased water binding capacity in each particle size in comparison with control sample. Increase in water binding due to gluten addition may be attributed to increase in protein content. In the area of batters and breadings, addition of gluten protein fraction and damaged starch are more pronounced when water binding capacity is concerned.

As long as breadings formed from small particle size were concerned, gluten added breading absorbed less oil than corn and soy flour added breadings. Oil uptake of the breading changed between 16,15 and 33,15%. On the other hand, when breading formed from large particle size were considered, it was observed that corn addition decreased oil

absorption more than other ingredients as compared with control sample. When two results were considered, gluten remarkably decreased oil absorption as compared with control sample. Similarly, Salvador et al. (2005) found that gluten addition decreased fat content after frying in comparison with control sample.

	Moisture (%)		Water Binding Cap (g/g)		Oil Uptake (%)		Compressive Force (g)	
p.s.	**small**	**large**	**small**	**large**	**small**	**large**	**small**	**large**
c	7,43	7,85	3,15	3,18	20,00	34,58	3049,3	4769,5
c5	7,83	7,31	2,87	2,71	31,01	19,67	3085,5	5211
c10	8,22	8,21	2,81	2,85	33,15	18,58	3004	4756
c15	8,04	7,83	2,64	2,64	28,79	18,51	3296,5	4543,5
s5	7,23	7,18	2,83	2,87	28,83	32,64	3243,8	3573
s10	7,24	7,7	2,82	2,69	26,66	32,6	3312	3850
s15	7,60	7,85	2,74	2,56	31,66	33,76	5140	6264,5
g3	7,14	7,16	3,17	3,08	16,23	26,09	1872,4	2979
g6	6,69	6,89	3,25	3,10	16,20	28,71	1655,1	2027,7
g9	7,29	7,42	3,45	3,13	16,15	27,51	1984,5	2198,4
LSD	**0,76**	**0,76**	**0,17**	**0,2**	**9,29**	**9,29**	**373,2**	**373,2**

	Color After Frying					
	L		a		b	
p.s.	**small**	**large**	**small**	**large**	**small**	**large**
c	68,54	67,51	2,89	3,21	34,53	34,56
c5	69,11	63,11	4,61	7,41	36,62	38,22
c10	66,73	70,63	4,99	2,45	38,8	37,12
c15	71,08	66,18	2,28	4,36	35,7	37,12
s5	61,92	60,25	6,65	10,75	39,87	39,95
s10	63.00	60,01	5,43	9,20	37,91	38,16
s15	60,39	62,66	9,48	7,99	39,86	37,99
g3	61,10	60,6	6,04	5,83	35,98	35,69
g6	63,56	61,33	5,15	6,87	35,5	34,96
g9	70,94	66,51	4,38	4,53	36,95	37,67
LSD	**1,69**	**1,69**	**1,26**	**1,26**	**3,08**	**3,08**

p.s: particle size, c: control, c5: 5%corn flour, c10: 10% corn flour, c15: 15% corn flour, s5: 5% soy flour, s10: 10% soy flour, s15: 15% soy flour, g3: 3% gluten, g6: 6% gluten, g9: 9% gluten, LSD: Least Significant Difference

Crispness is a very important eating characteristic of breading coated chicken breasts. The crispness of the chicken breast is a result of its coating's textural structure. Maximum compressive force of breading with small particle size changed between 1655,1 and 5140 g., while compressive force of breading with large particle size changed between 2027,7 and 6264,5 g. LSD value for compressive force is 373,2 (Table 1).
Table 1. The properties of breadings formed from different particle size
The compressive force increased as the particle size of breading used to form the coating increased. Similarly, Maskat and Kerr (2002) reported that hardness values increased with increasing particle size of the breading. 15% soy flour addition considerably increased compressive force while gluten addition reasonably decreased the compressive force as compared with control sample ($p<0.0001$). Similarly, Salvador et al. (2005) reported that gluten addition decreased peak force values in texture measurements.
Color of breadings after frying were determined. When L* value of small particle size breading was considered, 15% soy flour added breading was significantly darker than control or corn flour and gluten added breadings ($p<0.0001$). Doğan et al. (2005) also reported that soy flour addition provided the darkest and reddest colored nuggets. This could be related to the high amount of protein in soy flour undergoing Maillard reactions. Baixauli et al. (2002) reported that addition of corn flour to the coating gave a more yellow product. Similarly, in our study, corn flour addition increased b* value of breadings.

Statıstıcal analysıs
The statistical evaluation of the results was performed using the Statistical Analysis System (SAS, 1999). Means were seperated when significant using PROC GLM procedure of SAS. LSD Test was used for indicating the differences between the samples. Correlations were determined using PROC CORR procedure of SAS.

Correlatıon coeffıcıents among characterıstıcs of breadıngs

A high negative correlation was observed between water binding capacity and hardness of breadings (r = -0.70, p= 0.0005). The less water bound the breading, the harder was its texture. Gluten protein was found to bind water more than control, suggesting that the differences in water binding capacity may also be attributed to differences in protein content. Altunakar et al. (2006) studied the effects of hydrocolloids on apperant viscosity of batters and quality of chicken nuggets. Gum added batters were found to result in softer products (decreased force) due to their high water holding capacities. In our study, gluten- having the highest water holding capacity- had the lowest force results similar to gums.

Conclusıon

We can say from our experiments that gluten, in every percentage, is the most suitable ingredient among the ingredients used in this study. Its high water binding capacity provides decrease in particle hardness by forming crisp breading which consumers enjoy. Soy flour addition increased hardness of the breadings and decreased water binding capacity. Especially 15% soy flour addition had unfavorible effect on breading texture. Corn flour addition did not resulted in improved breading quality. As a result, replacement of a small portion of wheat flour with gluten can be recommended to be used in breadings.

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PRELIMINARY RESULTS FOR CONTENT OF NATURAL AND ARTIFICIAL RADIONUCLIDES IN SOME MARINE OBJECTS FROM BLACK SEA COAST

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ABSTRACT

The studies related to radioactive pollution and behavior of natural and man-made radionuclides in Black-sea water basin are rather limited and give no complete picture of the radioecological status of the region. There is lack of systematic data concerning their physical and chemical peculiarities as well as their concentration levels in bottom sediments, algae, mussel, fish, sea snails and other Black sea marine organisms.
That is why results from preliminary research of natural and artificial radionuclides in mussels, rapa whelk, algae, fish and so from the Bulgarian Black sea coastline are presented in this paper. The technogenic ceasium-137 together with the natural uranium-238, bismuth-214 and

potassium-40 are determined. The isotopes of uranium are determined by both gamma-spectrometry and alpha-spectrometry after radiochemical procedure for purification and concentration.

INTRODUCTION

Fish and marine delicacies occupy larger part in the menu of bulgarians with the increase of food culture. They have high nutritious value and are precious dietetic food for children as well as sick and old people. Their exceptional gustatory qualities and unique food components increases the market for them.

The changes in Black sea ecosystem in the last years caused reduction in the list of industrial fish varieties and draught amounts of them. This turned the attention of a lot of fishing organizations and firms towards draught of some non fish resources – the predatory snail veined rapa whelk *(Rapana venosa)*, the blue mussel *(Mytilus galloprovincialis,),* the mussels of genus Donax and others.

This makes them interesting objective of research regarding their radioecological cleanliness and increase of human health safety. The different representatives of marine flora and fauna are known as some kind of filter for different pollutants including radionuklides. Their level of contamination can be indicative of the ambient distribution of radionuclides, contamination level of a given area and this data may be helpful in evaluating the potential risk for the biosphere and population. Some initial results from study of samples from Black sea coast are commented in this paper.

MATERIALS AND METHODS

Sampling of bottom sediments, algae, mussels and veined rapa whelk was done in the period between 2004 and 2006. The greatest variety of collected samples was from the region of Varna bay, Emine cape and Galata. In 2005 the sampling was extended with samples from the Southern part of Black sea coast – Arkutino, Sinemorets, Arapja and Burgas bay.

The sampling in Varna bay was performed by Scientific Research Boat "Prof. Valkanov" owned by the Institute of Fishing Resources", Varna getting up to six miles out to sea. Samples of different spieces of mussels

(*Mytilus galloprovincialis, Anadara inaequivalvis)*, algae (*Algae, Chlorophyta*) and bottom sediments were collected.
All samples were analyzed by gamma-spectrometry using procedure validated in the Laboratory of radioecology in the Institute of Soil Science "N. Poushkarov" (Naydenov M., 2001). The following radionuclides were determined – the technogenious ceasium-137 and the naturally existing uranium-138, bismuth-214, actinium-228 and potassium-40. A high-purity germanium detector with 20% efficiency was used. The experimental errors were less then 10%.
The samples of algae, blue mussels and veined rapa whelk for which the gamma-spectrometry analyses showed relatively higher results where analysed additionally to determine the isotopes of uranium through radiochemical procedure and alpha-spectrometry. The radiochemical procedure used for separation and concentration of uranium isotopes is based on liquid-liquid extraction with trioctylphosphinoxide, coprecipitation with LaF and electrodeposition (Naydenov M., 2001). The chemical yield varied between 50 and 80%. The spectrometer used for the measurements included a PIPS detector and Multichamal analyzer CANBERRA. The Lower Limit of Detection (LLD) for the uranium isotopes, calculated for 1 000 min measuring time was between 0.6 and 1.2 mBq per sample. The uncertainties of the reported results are evaluated considering counting statistics and calibration errors. Although the selected method is more expensive and labour intensive its higher sensitivity allows determination of lower activities.

RESULTS AND DISCUSSION

Some initial results from study of samples from Black sea coast are commented in this paper. The data obtained by gamma-spectrometry of the samples collected in the period 2004 to 2005 is shown in Table 1. In most of the samples the radionuclides studied are detectable within the admissible errors.

Table 1

Results of gamma-specrtometry analyses of samples from Black sea (2004- 2005) [Bq/kg]

Sampling place	Sample type	Cs-137	U-238	Bi-214	Ac-228	K-40
1. Varna – fishing beach (2004)	*Mytilus galloprovincialis*	3 ± 2	7 ± 4	17 ± 4	15 ± 5	98 ± 10
2. Varna – fishing beach, (2004)	*Mytilus gallop.*- shells	< 1	4 ± 3	2 ± 1	< 2	< 50
3. Galata – 6 miles to the east (2004)	*Mytilus galloprovincialis*	2±0,5	5 ± 2	3 ± 1	4 ± 2	30 ± 5
4. Kaliakra (2004)	*Mytilus galloprovincialis*	2±0,5	6 ± 4	7 ± 2	3 ± 2	< 50
5. Galata – 6 miles to the east (2004)	*Anadara inaequivalvis*	< 1	6 ± 3	< 1	4 ± 2	< 50
6. Galata – 6 miles to the east (2004)	*Rapana venosa*	10±2	5 ± 3	< 1	< 2	70 ± 5
7. Varna – fishing beach (2005)	*Chlorophyta*	2±0,5	6 ± 2	3 ± 1	3 ± 1	210 ± 10
8. Varna – beach coast (2005)	*Algae*	2±0,5	7 ± 2	8 ± 2	7 ± 2	270 ± 10
9. Arkutino (2005)	*Brown Algae*	2±0,5	< 5	3 ± 2	7 ± 3	30 ± 3
10. Arkutino (2005)	*Chlorophyta*	<1	< 5	3 ± 2	< 2	20 ± 2
11. Sinemoretz (2005)	*Chlorophyta*	< 1	< 5	< 1	< 2	40 ± 4
12. Arapja (2005)	*Chlorophyta*	< 1	< 5	< 1	4 ± 1	100 ± 10

Table 1 continued

Sampling place	Sample type	Cs-137	U-238	Bi-214	Ac-228	K-40
13. Tsar's beach, Gradina camp. (2005)	*Brown Algae*	3 ± 1	29 ± 12	20 ± 3	37 ± 6	1500±15
14. Burgas bay, depth. 20 m (2004)	Sediment	2±0,5	21±5	10 ± 1	18 ± 2	350± 10
15. Burgas bay, depth. 20 m (2005)	Sediment	13± 1	11 ± 4	17 ± 3	17±5	825 ± 30

The following radionuclides were determined – the technogenious ceasium-137 and the natural uranium-238, bismuth-214, actinium-228 and potassium-40. In all of the analyzed samples of blue mussels (*Mytilus galloprovincialis*) and sediments the measured radionuclides were in detectable amounts. The same is observed for the samples of algae collected from Varna bay and brown algae from Sothern Black sea coast. In the samples of green algae (*Chlorophyta*) from Sothern Black sea coast potassium-40 only is in detectable amounts while the other radionuclides are below the lowest limit of detection for the method used. The specific activity of ceasium-137 varies between 2 and 10 $Bq.kg^{-1}$ and the highest values for this radionuclide are determined in veined rapa whelk (*Rapana venosa*).

The highest radioactivity caused by natural radionuclides was determined in a sample of brown algae from the coast of "Gradina" camping (about 30 Bq uranium-238 per kg dry weights and actinium-228 - about 40 $Bq.kg^{-1}$ dry weights). The values reported are within the range of background amounts (Strezov, A, 1995; Strezov, A, 1996).

Our results obtained correspond with data from investigations of other sea basins. At studying the ecosystem of Red sea (El-Arabi, 2002; Sam, A.K.,1998) the authors compare the data for natural radioactivity in marine sediments with such for samples from the 30-km terrestrial coast zone. A conclusion is made that the antropogenic influence as well as the transfer from terrestrial area to marine ecosystem is insignificant. The

highest values for ceasium-137 are registered in brown algae from the harbour areas.

Table 2
Results of gamma-specrtometry analyses of samples from Black sea (2006) [Bq/kg]

Sampling place	**Sample type**	**Cs-137**	**U-238**	**Bi-214**
Galata	*Rapana venosa*	< 1	< 3	1 ± 0,5
Galata	*Rapana venosa* (shells*)*	4 ± 2	4 ± 2	3 ± 1
.Galata	*Mytilus galloprovincialis*	2 ± 1	7 ± 3	5 ± 2
.Galata	*Mytilus galloprovincialis* (shells)	0,3 ± 0,2	4 ± 1	1 ± 0,5
Galata	*Anadara inaequivalvis*	1,2 ± 0,5	7 ± 3	5 ± 1
Galata	*Anadara inaequivalvis* (shells)	0,4 ± 0,2	5 ± 1	2,5 ± 0.5

Sampling place	**Sample type**	**Ac-228**	**K-40**
Galata	*Rapana venosa*	< 2	50 ± 5
Galata	*Rapana venosa* (shells*)*	3 ± 2	73 ± 7
.Galata	*Mytilus galloprovincialis*	4 ± 2	160 ± 30
.Galata	*Mytilus galloprovincialis* shells)	1,2 ± 0,5	30 ± 6
Galata	*Anadara inaequivalvis*	3 ± 2	40 ± 5
Galata	*Anadara inaequivalvis* (shells)	2,3 ± 0,5	20 ± 2

The monitoring of mussels and veined rapa whelks contamination, which are often included in the food diet is of a particular importance for evaluating the danger of additional impact to the radiation load of people by food. The data presented in table 1 show that all the radionuclides determined are in detectable amounts in mussels and veined rapa whelks. Because of the explicably greater interest towards those spieces we analysed separately the different parts (fleshy part and shells) in order to determine where the radionuclides accumulate more intensively. The results are shown in Table 2.
The data obtained as a result of the analyses of the samples collected in 2006 confirm the higher concentrations of ceasium-137 in veined rapa

welks. The analysis of the separated parts showed stronger accumulation in the fleshy part of mussels. In case of veined rapa welks ceasium is more concentrated in the shell. The same is observed for the other radionuclides analysed – for mussles the accumulation is stronger in the fleshy part, while for veined rapa welks in the shell. The differences in phisiology and ways of feeding may be a reason for that.

The mussels in Black sea are active filtrators – for a twenty-four hours period one mussel 3-4 cm in size filtrates 21 l of water (Kiseleva M. I., 1981), detaining in its body nutritious substances together with pollutants. The phytoplankton is basic element in mussels food and together with it they take radioactive elements which accumulate in the fleshy part. The veined rapa welks have rapacious way of feeding. They use different types of mollusca fish carcasses as food because of which the accumulation in the fleshy part is less.

The recent investigations of Black sea show that it is one one of the most polluted basins in the world (Golemanski**,** V., 1998) and this makes it an important medium for study of the processes controlling the geochemistry of uranium in the liquid and hard phase of the sediments. Uranium compared to plutonium and americium is more conservative in its behaviour so that it retains longer in water ([Hunt G.J, 1990; Polikarpov G., 1966). Because of that higher specific activities for this radionuclide are logically expected in marine organisms.

The use of alpha-spectrometry analysis is one possibility for extending the research and accomplishing more precise measurements. For part of the samples radiochemical analysis combined with alpha-spectrometry measurements were done for determination of uranium where the specific activity of the radionuclides studied by gamma-spectrometry was bellow or a little bit above the minimum detectable activity (MDA). The results are presented in Table 3.

The data obtained indicates once more lower accumulation of uranium in veined rapa welks compared to that in the mussels. Some shifting of the secular equilibrium between the two isotopes of uranium (238 and 234) towards 234 may be remarked. Such shifting is typical of uranium in water and because of that such ratio should be expected in marine organisms as well {Strezov, A, 1996}.

The accumulation in algae is comparable to that in mussels with secular equilibrium between the two isotopes of uranium shifted in the same direction.

Table 3
Results of alpha-specrtometry analyses of samples from Black sea (2004) [Bq/kg]

Sampling place	Sample type	U-234	U-235
Galata – 6 miles to the east	*Mytilus galloprovincialis*	6 ± 1	0,4 ± 0,1
Kaliakra	*Mytilus galloprovincialis*	7 ± 1	0,5 ± 0,1
Varna – fishing beach	*Chlorophyta*	7 ± 1	0,4 ± 0,1
Galata	*Rapana venosa*	1 ± 0,2	< 0,1

Sampling place	Sample type	U-238
Galata – 6 miles to the east	*Mytilus galloprovincialis*	4 ± 1
Kaliakra	*Mytilus galloprovincialis*	5 ± 1
Varna – fishing beach	*Chlorophyta*	5 ± 1
Galata	*Rapana venosa*	1,4 ± 0,2

Conclusions:

1. The radionuclides studied are in detectable amount in all the analyzed samples of mussels and sediments and do not exceed the fallout values cited in the literature.
2. In case of mussels the radionuclides are accumulated in the fleshy part while for veined rapa welks – in shells.
3. Part of the determined radionuclides are under the detectable levels in algae. Relatively higher specific activities are reported in the brown algae.
4. There are no statistically significant differences in the levels of contamination between the samples from the different sampling locations along the Black sea coast.

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DISTRIBUTION OF ^{3}H IN OBJECTS FROM BR "SREBARNA" AND SURFACE WATERS FROM NORTH AND SOUTH BULGARIA

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ABSTRACT

By reason of the big radiation-hygienic significance of tritium for the individual its specific activity is determined in soils, plants (agricultural and aquatic) and water from BR"Srebarna" and surface waters from North and South Bulgaria. The determined levels of specific activity are low, due to global cycling tritium, and show the lack of local tritium contamination.

INTRODUCTION

The tritium in the environment is produced by the interaction of cosmic rays with nucleus of chemical elements in the atmosphere, the nuclear bomb test, the operation of nuclear reactors and nuclear fuel reprocessing plants. Tritium is one of the more important radionuclides for doze assessment, characterizing and determining the radiation situation in the regions around nuclear fuel cycle industries. As an isotope of hydrogen, the tritium is includes in many organic compounds, as well as in genetic-information macromolecules. The beta-decay of tritium leads to disrupt of the molecular structure and the intermolecular connection by effect of ionizing radiation of beta-particles as well as the transformation of tritium to isotope of helium.

By these reasons, the investigation of specifics of the behaviour of tritium in the environment and the pathways for incoming in human organisms is an important purpose, as well as his high migration mobility motivates the necessity of long-term dynamic monitoring.

The purpose of this paper is investigation of the distribution of "free" tritium in elements of ecosystem of the Biospheric reserve "Srebarna" and surface water from North and South Bulgaria.

MATERIAL AND METHODS

The specific activity of tritium in elements of ecosystem of the Biospheric reserve "Srebarna" and his restricted area was determined in period 1998-2000 years. Twice annual (in spring and autumn) was performed sampling for analysis water, water growing plants, /wodna tchuma (*Egeria densa)*, wodna leshta (Lemna minor), drebna mehurka (*Ultricularia vulgaris)* and rogolistnik (*Ceratophyllum demersum)/*, soil, agriculture plants and products (wheat, maize, sunflower, bean, potatoes and lucerne), virgin-soil grass and reed *(*Phragmites astralis). In the next years, single samples of surface water from Northern and Southern Bulgaria have been taken.

The specific activity of tritium in biological material and soil was determinate as free water, obtained from low-temperature vacuum distillation; water samples were distilled after filtration by routine procedure. The next step of sample preparation for tritium analysis

includes electrolysis isotope enrichment, with mean isotope enrichment factor (6.25 ± 0.35) for the system applied.
The activity of tritium in enriched samples was determinate on LCC Beckman LS 9800 with liquid scintillation cocktail Ultima Gold LLT (8 ml enriched water+10 ml cocktail) and time of measurement for each sample 500 min (5 cycles, each 5x20 min). The results obtained for specific activity of tritium was presents in Bq/l for all objects in this investigation.

RESULTS AND DISCUSSION

The annual mean specific activity of "free" tritium in soil and plants from region of the reserve "Srebarna" is between 3÷6 Bg/l for the 3 years of investigation (Table 1).
The lack of significant difference in concentration of the isotope between soil and plant may be connected with specifics of the behaviour and incoming. The tritium absorbed in soil as HTO following the same transport mechanism as normal water, distributes in soil profile and is accumulate from the plants. Besides the roots, the plants assimilates tritium from atmospheric moisture by surface of the leafs.
Simultaneously one significant part of soil HTO returns back to the atmosphere by evaporation from soil and leafs of the plants [Brudenell et al., 1997; Choi et al., 2005, 2007]. In the case of normal emission of tritium from generating sources between these processes is establish dynamic equilibrium, following to equal tritium concentration between in soil water, surface air and plants [Peterson, S., et al., 2000].
We did not specify differences in specific activity of tritium in water content of the different kinds of agriculture plants and between vegetative parts of plants (leafs and stems) and reproductive parts as seed-grains. Such a difference is establish after correction of specific activities with water content in these organs. In this case the tritium content in the seed-grains is in such order lower than in stems and leafs.
The results obtained for specific activity of ^{3}H in water from the lake and water plants (Table 1) confirms established relation [Murphy, 1984, 1993], that free water tritium in water plants is the same in the water.

Table 1
The annual mean specific activity of ^{3}H (Bq/l water) in soil, agriculture plants and products, grass, water and water plants.

Sample	Site of sampling	1998 г	1999 г	2000 г
Soil, 0 – 10 cm	Southern lock	3 ± 1	5 ± 1	4 ± 1
Soil, 0 – 10 cm	Fazanarija	4 ± 1	4 ± 1	5 ± 2
Soil, 0 – 10 cm	Northern lock	4 ± 2	5 ± 2	4 ± 2
Maize, plants	Northern lock	4 ± 2	4 ± 2	4 ± 2
Maize, seed-grains	Northern lock	6 ± 2	4 ± 2	5 ± 2
Sunflower, plants	Northern lock	3 ± 1	3 ± 2	5 ± 2
Sunflower, seed-grains	Northern lock	4 ± 1	5 ± 2	4 ± 1
Wheat, plants	Northern lock	4 ± 2	3 ± 1	3 ± 1
Wheat, seed-grains	Northern lock	4 ± 1	4 ± 1	5 ± 2
Bean, seed-grains	West from museum	3 ± 1	3 ± 1	4 ± 2
Potatos, klubeni	West from museum	4 ± 1	3 ± 1	4 ± 2
Lucerne, plants	Northern lock	3 ± 1	5 ± 2	4 ± 1
Lucerne, plants	Southern lock	3 ± 1	4 ± 2	3 ± 2
Lucerne, plants	Fazanarija	4 ± 2	3 ± 2	4 ± 1
Grass, mixed	Northern lock	5 ± 2	4 ± 1	4 ± 2
Grass, mixed	Southern lock	3 ± 1	4 ± 2	3 ± 1
Reed	Southern lock	4 ± 2	3 ± 1	3 ± 2
Water	Northern lock	3 ± 1	4 ± 2	5 ± 1
Water	Southern lock	3 ± 1	4 ± 2	4 ± 2
Wodna tchuma	Southern lock	3 ± 2	3 ± 2	4 ± 1
Wodna leshta	Southern lock	3 ± 1	3 ± 1	4 ± 1
Mehurka	Southern lock	3 ± 1	4 ± 1	4 ± 2
Rogolistnik	Southern lock	3 ± 2	5 ± 2	5 ± 1

In investigated water samples from Danube river (Kozloduj, Oriahovo, Belene, Svishtov and Silistra) the specific activity of ^{3}H vary from below the minimum detectable activity of the analyses (MDC=2.2 Bq/l) to 4.4 Bq/l (Table 2).A comparison between the annual mean specific activity of HTO in Danube river for period 1998-2000 years (Table 1, Northern lock) with these for 2004-2007 year shows no difference between these data. An exception is only for warm channel of NPP “Kozloduj” in 2006 year - twice higher concentration of 9.0 Bq/l HTO.

Table 2.
Specific activity of ^{3}H (Bq/l water) in surface water from Northern and Southern Bulgaria

Site of sampling	Specific activity of ^{3}H, Bq/l			
	2004 y.	2005 y.	2006 y.	2007 y.
Kozloduj, the port	3,0 ± 1,0	< 2,2	< 2,2	3,0 ± 1.0
NPP "Kozloduj" cold channel	3,0 ± 1,0	-	3,0 ± 1,0	3,0 ± 1,0
NPP "Kozloduj, warm channel	-	-	9,0 ± 2,0	4,0 ± 1,0
Harletz	-	< 2,2	< 2,2	3,0 ± 1,0
Dam lake "Septemvrijci"	3,0 ± 1,0	< 2,2	< 2,2	< 2,2
Miziia	-	-	< 2,2	< 2,2
Oriahovo, the port	-	< 2,2	4,0 ± 1,0	3,0 ± 1,0
Belene, the port	4,0 ± 1,0	< 2,2	4,0 ± 1,0	4,0 ± 1,0
Swishtov, the monuments	-	3.0 ± 1.0	4,0 ± 1,0	4,0 ± 1,0
Silistra, the border	-	< 2,2	< 2,2	-
Dam lake "Dospat"	< 2,2	< 2,2	< 2,2	< 2,2
Dam lake "Shiroka Poliana"	< 2,2	< 2,2	< 2,2	< 2,2
Dam lake "Goliam Beglik"	< 2,2	< 2,2	< 2,2	< 2,2
River Tcherna Mesta	-	-	< 2,2	< 2,2
Dam lake Biala Mesta	-	-	< 2.2	< 2.2
Smolia lakes	3.0 ± 1,0	< 2,2	< 2,2	< 2,2
River Jakoruda	-	-	< 2,2	< 2,2
River Dobrionishte	< 2,2	< 2,2	< 2,2	-
River Dolna Mesta	< 2,2	< 2,2	< 2,2	< 2,2

Our results coincides with the data from [Villa and Manjon, 2004], who in water samples from Danube river, taken before outflow of the warm channel of NPP "Kozloduj (2003 year) determine specific activity of tritium 1.7–3.0 Bq/l, and from the warm channel–10.7 Bq/l.
The measurements of specific activity of tritium in surface water from other sites in Northern Bulgaria almost were below MDC and no significant dynamics was assessed. The same concentrations and dynamics were assessed in surface water from Southern Bulgaria, all results were below MDC with the only exception-water from Smolian lakes for 2004 year.

Conclusions

The results obtained shows that the level of tritium in elements of ekosystem of BR"Srebarna" and surface waters from Northern and Southern Bulgaria are in the limits of the typical tritium concentration for Central Europe and at this stage of investigation any tritium contamination in Bulgarian was not assessed.
The estimated annual mean specific activity of tritium is near to the average values in the environment, typical in the recent years for this globally dispersed radionuclide.

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CONTENT