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Cocoa Shell as an Innovative Ingredient in Chocolate with a Strong Alcoholic Filling

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ARTICLE INFO	ABSTRACT
Article history:	The chocolate industry is constantly developing new products with different
Received: November 22, 2021 Accepted: February 7, 2022	fillings, toppings, flavours, etc. Chocolates with alcoholic filling are popular, especially with young adults. In chocolates with alcoholic filling different kinds
Keywords: chocolate, alcoholic filling bitter herbal liqueur sensory acceptability stability	of thickeners can be used to increase the viscosity of the filling and make production easier. In this study, we examined cocoa shells, a by-product of the chocolate industry, as a new ingredient and thickener of an alcoholic filling. Chocolates with bitter herbal liqueur were produced in a ball mill with four different fillers. A comparison of guar gum and cocoa shell as thickeners was conducted. The influence of the filling ingredients on the stability of the filled chocolate was determined by measuring colour and gloss over 30 days of accelerated fat bloom. The results showed that chocolates for which the cocoa shell was used were more stable than chocolates where guar gum was used. Evaluation of sensory properties of chocolates with bitter herbal liqueur and different thickeners was conducted by the trained sensory panel. The highest- rated, after sensory assessment, were dark chocolates that contained cocoa shells in the filling. Also, an online questionnaire was conducted to examine consumer preference for a new product. According to the results, it can be concluded that the production of this chocolate would be desirable.

Introduction

After the development of milk and white chocolate, producers also developed different products enrobed with chocolate. As different foods were coated in chocolate, chocolates with alcoholic fillings also became available on the market and were highly accepted by consumers (Talbot, 2009). The most common procedure for the production of chocolates with alcoholic filling is to prepare chocolate shells in which fillings are deposited. Fillings and shells are cooled to solidify low-viscosity fillings. After that, shells are sealed with a layer of chocolate mass, and additional cooling is conducted (Böhme et al., 2012). The viscosity of fillings plays an important role in the production of chocolate with alcoholic filling. It decreases as ethanol content increases and because of that, different kinds of thickeners are added to increase viscosity and make the production process easier (Böhme et al., 2012). Guar gum is often used in the

food industry because of its properties. It is used as a

alcoholic filling (bitter herbal liqueur) with two

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stabilizer and food additive but it can be a source of dietary fibres as well. Because it is resistant to breakdown and stable at low pH, it is used for improving the shelf life of different food products (Mudgil et al., 2011). The cocoa shell is a by-product of the chocolate industry that is still underutilized. This by-product has a valuable composition; it is mainly composed of dietary fibre but also contains proteins and fat similar to cocoa butter. During the processing of cocoa beans, some components that are present in cocoa beans like polyphenols and methylxanthines also migrate to the cocoa shell (Barišić et al., 2020). Analysis of the composition of dietary fibres from cocoa shells showed that pectic polysaccharides are highly represented. This could have a role in the utilization of cocoa shells as thickening agents (Redgwell et al., 2003). The aim of this study was to produce chocolate with

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different thickeners (guar gum and cocoa shell). In addition, the stability of these chocolates was determined during a storage period of 30 days. Sensory acceptability and consumer preference of these chocolates were also determined.

Materials and methods

Production of chocolates with strong alcoholic filling

The dark chocolate (1000 g) was produced according to the following recipe: 36% cocoa mass (DGF, France), 21.47% cocoa butter (DGF, France), 42% sugar (Sugar Factory Osijek, Croatia), 0.25% lecithin (A.C.E.F., Italy), 0.25% PGPR (Azelis, Croatia), and 0.03% vanillin (Acros organics, Belgium). It was produced in a custom-made laboratory ball mill (D&D metal Osijek, Croatia). The processing parameters were as follows: 5 kg of stainless steel balls (9.525 mm diameter), speed 60 rpm, temperature 55 °C and time 3 h. At the beginning of mixing, cocoa mass, cocoa butter and sugar were added. One hour before the end of mixing, lecithin and PGPR were added, and 30 minutes before the end, vanillin was added. Four different alcoholic fillings (50 g) were made by hand and their composition is presented in Table 1. After the production of chocolate mass in the ball mill, mass was hand tempered and temper index was measured with a Sollich Tempermeter E3 (Sollich, Bad Salzuflen, Germany) (temper index 4-6). The chocolate mass was dosed into moulds in a thin layer to form a capsule while shaking the mould on a vibrating table (Dedy, Essen, Germany). The moulds were then placed to cool at 8 °C for 30 minutes. After solidification of the chocolate, the alcoholic filling was dosed into the formed chocolate shells using the laboratory dispenser (Dedy, Essen, Germany), and again placed to cool (8 °C, 30 minutes). After that, the tempered chocolate mass was distributed evenly to close the shell. The chocolate was cooled again at 8 °C for 30 minutes.

Storage of chocolates – accelerated fat bloom

Chocolates were wrapped in aluminium foil and stored in the cooling incubator with controlled humidity (Climacell 111, MMM Medcenter GmbH, München, Germany). The stability was evaluated at two relative humidities: 65% and <50%. The temperature fluctuations were: 20 °C for 12 h and 29 °C for 12 h. Samples for analyses were taken immediately after the production of chocolates, and after 5, 10, 15, 20, 25 and 30 days of their storage.

Colour and gloss of chocolates

The colour of chocolates with the alcoholic filling was determined with Konica Minolta CR-400 chromameter (Konica Minolta, Tokyo, Japan). Colour measurement was performed in the measuring systems CIEL*a*b* and L*Ch. For each sample, a measurement was performed in five repetitions. Total colour change (ΔE) (1) and whiteness index (WI) (2) were calculated according to:

$$\Delta E = \sqrt{(L - L_0)^2 + (b - b_0)^2 + (a - a_0)^2}$$
(1)

$$WI = 100 - [(100 - L)^2 + a^2 + b^2]^{0.5}$$
(2)

where L_0 , a_0 and b_0 represent values for chocolates after cooling with which colour change is compared.

The gloss of chocolates with the alcoholic filling was determined with a Gloss Meter PCE-PGM 100 (PCE Instruments, Meschede, Germany). For each sample, the measurements were performed in five repetitions after which the mean value and standard deviation were calculated. The results are displayed in a gloss unit (GU).

Sensory analysis

Sensory analysis of chocolates with alcoholic filling after the production of samples was conducted by a sensory panel consisting of 5 trained assessors who have passed a basic course on sensory analysis and have perennial experience in sensory analysis of confectionery products. Sensory attributes that were evaluated were appearance; structure, break and firmness; chewiness; odour and flavour. Every parameter was rated by points from 1 to 5. Every parameter has its weighting factor with which points were multiplied and final weighted scores were calculated.

Consumer perception of a new product

In order to determine consumers' perception and acceptability of chocolates with the alcoholic filling based on bitter herbal liqueur, an online questionnaire was conducted. The questionnaire was completed by 500 randomly selected adult respondents of various age groups. The questionnaire consisted of 11 questions, and its aim was to examine the frequency of consumption of chocolate and bitter herbal liqueur, as well as the preference of respondents to their combination in the form of filled chocolate. The survey was completely anonymous.

Statistical analysis

Statistical analysis was conducted using Statistica®, Version 13.4.0.14 (1984–2018 TIBCO Software Inc., California, USA). The least significant difference (LSD) test (<p-value 0.05) was used to determine whether statistically significant difference is present between samples.

Results and discussion

Colour and gloss of chocolates

The colour of chocolates with the alcoholic filling was determined in a period of 30 days of accelerated fat bloom at two different relative humidity conditions (<50 and 65%) and with temperature fluctuation between 20 °C and 29 °C every 12 hours to accelerate degradation.

Table 2 shows that all colour parameters of sample 1 increased over a storage period of 30 days. There is a difference between relative humidity conditions: the whiteness index changed more, by 2.73 points, for chocolates stored at a relative humidity of 65%. This is in accordance with the L* value, the parameter of

lightness, which shows the same trend. After 30 days of storage, the total colour change for chocolate sample 1 stored at a relative humidity of <50% was higher. Sample 2 had a similar composition as sample 1, except for the addition of a bitter herbal liqueur aroma. Table 3 shows the same trend of change as sample 1, and values were similar for all examined parameters. The whiteness index was lower for chocolates stored at a relative humidity of <50% compared to those stored at 65%. This shows that the relative humidity during storage plays a great role in the quality of chocolates because water can cause bloom formation on the surface of the chocolate. Water dissolves sugar present in chocolates which solidifies at the surface and causes whitening of chocolate (Lonchampt and Hartel, 2006).

Chocolate sample 3 had the same trend of increasing all colour parameters (Table 3) as other samples, but all parameters had lower values than sample 1, which had the same composition except for the cocoa shell. This could be because the cocoa shell has a significant content of soluble fibres (Okiyama et al., 2017), and could bind ethanol better than guar gum and thus prevent its migration from the filling to the chocolate shell.

Table 1. Compositions of alcoholic fillings used in the research (%)

Composition	Filling 1	Filling 2	Filling 3	Filling 4
Sugar	20	20	20	19.97
Milk powder	3.5	3.5	3.5	3.5
Cocoa powder	9	8.97	-	-
Guar gum	1	1	-	-
Cocoa shell	-	-	10	10
Coconut oil	28	28	28	28
Bitter herbal liqueur	38	38	38	38
Lecithin	0.25	0.25	0.25	0.25
PGPR	0.25	0.25	0.25	0.25
Aroma of bitter herbal liqueur	-	0.03	-	0.03



Figure 1. Dosing of alcoholic filling

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1-50 after production	$28.49\pm0.47^{\mathrm{a}}$	$7.97\pm0.19^{\mathrm{a}}$	7.08 ± 0.26^{a}	$10.66\pm0.28^{\rm a}$	$41.64\pm0.76^{\rm a}$		$27.70\pm0.39^{\rm a,b}$
1-50 after 5 days	$30.43\pm0.64^{\rm c,d}$	$8.74\pm0.44^{\rm b}$	$8.30\pm0.79^{\rm b}$	$12.06\pm0.87^{ m b}$	$43.45\pm1.25^{\rm b,c}$	$2.45\pm0.91^{\rm b,c}$	$29.39\pm0.46^{\rm d,e}$
1-50 after 10 days	$30.74\pm1.15^{\rm d}$	$9.67\pm0.41^{ m d}$	$9.87\pm0.69^{ m d}$	$13.82\pm0.75^{\rm d}$	$45.45\pm1.17^{ m d}$	$4.02\pm1.08^{\rm d,e}$	$29.37\pm0.91^{\rm d,e}$
l-50 after 15 days	$32.64\pm0.29^{\rm g}$	$10.40\pm0.25^{\rm f}$	$11.12\pm0.57^{\rm f,g}$	$15.23\pm0.58^{\rm f}$	$46.92\pm0.86^{\rm e,f}$	$6.30\pm0.50^{\rm g}$	$30.94\pm0.23^{ m h}$
1-50 after 20 days	$31.79\pm0.92^{\rm e,f}$	$9.91\pm0.32^{ m d,e}$	$10.35\pm0.62^{ m d.e}$	$14.33\pm0.65^{\rm d,e}$	$46.21\pm0.97^{ m d.e}$	$5.07\pm0.82^{\mathrm{f}}$	$30.30\pm0.75^{\rm f}$
1-50 after 25 days	$32.22\pm0.81^{\rm f,g}$	$9.93\pm0.32^{ m d,e}$	$11.20\pm0.56^{\rm f,g}$	$14.97\pm0.59^{\rm e,f}$	$48.43\pm0.98^{\mathrm{g,h}}$	$5.90\pm0.88^{\rm f,g}$	$30.59\pm0.60^{\rm g,h}$
l-50 after 30 days	$31.93\pm0.70^{\rm f,g}$	$9.95\pm0.32^{ m d,e}$	$11.00\pm0.63^{\rm f,g}$	$14.83\pm0.67^{\rm e,f}$	$47.84\pm1.01^{\rm f,g}$	$5.58\pm0.86^{\rm f,g}$	$30.33\pm0.49^{f,g,h}$
1-65 after production	$28.30\pm0.24^{\mathrm{a}}$	$8.15\pm0.04^{\rm a}$	$7.59\pm0.14^{\rm a}$	11.14 ± 0.08^{a}	$42.94\pm0.64^{\rm b,c}$		27.44 ± 0.20^{a}
1-65 after 5 days	$29.11\pm0.28^{\rm ab}$	$7.92\pm0.09^{\mathrm{a}}$	7.30 ± 0.22^{a}	$10.77\pm0.20^{\rm a}$	$42.55\pm0.63^{\rm a,b}$	$0.93\pm0.16^{\rm a}$	$28.29\pm0.22^{\rm b,c}$
1-65 after 10 days	$29.83\pm0.54^{\rm b,c}$	$8.54\pm0.17^{\rm b}$	$8.24\pm0.28^{\rm b}$	$11.87\pm0.31^{\rm b}$	$43.97\pm0.44^{ m c}$	$1.71\pm0.55^{\rm a,b}$	$28.84\pm0.43^{\rm c,d}$
1-65 after 15 days	$31.76 \pm 0.48^{ m e,f}$	$9.99\pm0.08^{ m d,e}$	$10.71\pm0.31^{ m e,f}$	$14.64\pm0.27^{\rm e,f}$	$46.99\pm0.73^{\rm e,f}$	$5.01\pm0.49^{ m e,f}$	$30.21\pm0.37^{\rm f,g}$
1-65 after 20 days	$31.02\pm0.92^{ m de}$	$9.13\pm0.16^{\rm c}$	$9.20\pm0.53^{ m c}$	$12.96\pm0.49^{ m c}$	$45.19\pm1.17^{ m d}$	$3.31\pm0.93^{ m c,d}$	$29.81\pm0.73\mathrm{e,f}$
1-65 after 25 days	$32.39\pm0.46^{\rm f,g}$	$9.91\pm0.16^{\rm d,e}$	$11.38\pm0.23^{\rm g}$	$15.09\pm0.2\textrm{T}^{\textrm{f}}$	$48.94\pm0.22^{\rm h}$	$5.85\pm0.46^{\rm f,g}$	$30.73\pm0.35^{\rm g,h}$
1-65 after 30 days	$31.83 \pm 0.54^{\rm e,f,g}$	$10.16\pm0.10^{\rm e,f}$	$11.10\pm0.30^{\rm f,g}$	$15.05\pm0.26^{\rm f}$	$47.49\pm0.62^{\rm f,g}$	$5.37\pm0.49^{\mathrm{f,g}}$	$30.19\pm0.43^{\rm f,g}$

respectively); All values with the same letters in the same column are not significantly different at p>0.05

Table 3. Colour changes of the dark chocolate filled with bitter herbal liqueur, with guar gum as a filler and flavour compounds (chocolate sample 2)

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Sample	L^*	a*	p*	J	h°	$\Delta \mathrm{E}$	MI
2-50 after production	28.42 ± 0.22^{a}	$8.09\pm0.16^{\mathrm{a}}$	7.54 ± 0.30^{a}	11.06 ± 0.32^{a}	42.97 ± 0.71^{b}		27.57 ± 0.19^{a}
2-50 after 5 days	$30.33\pm0.33^{ m c}$	$8.84\pm0.14^{\rm b.c}$	$8.73\pm0.13^{\rm b,c}$	$12.43\pm0.18^{\rm b,c}$	$44.62\pm0.18^{\rm d,e}$	$2.37\pm0.33^{ m b}$	$29.23\pm0.27^{\rm c,d}$
2-50 after 10 days	$30.97\pm0.61^{ m d}$	9.70 ± 0.23^{d}	$10.31\pm0.59^{\rm d}$	$14.15\pm0.59^{\rm d}$	$46.71\pm1.02^{\rm f}$	$4.09\pm0.76^{\rm c}$	$29.53\pm0.44^{\rm d,e}$
2-50 after 15 days	$32.45\pm0.24^{ m g}$	$10.64\pm0.12^{\rm h}$	$11.94\pm0.25^{\rm f}$	$16.00\pm0.26^{\rm g}$	$48.30\pm0.40^{\rm hi}$	$6.49\pm0.28^{\rm f}$	$30.58\pm0.19^{\mathrm{g,h}}$
2-50 after 20 days	$31.05\pm0.57^{ m d}$	$10.02\pm0.18^{\rm e,f}$	$10.86\pm0.52^{\rm e}$	$14.77\pm0.51^{ m e}$	$47.28\pm0.87^{\rm f,g}$	$4.67\pm0.55^{\rm c,d}$	$29.48\pm0.48^{\rm d,e}$
2-50 after 25 days	$31.67\pm0.61^{ m e}$	$10.11\pm0.07^{\rm f,g}$	$11.70\pm0.37^{\rm f}$	$15.46\pm0.30^{\rm f}$	$49.17\pm0.86^{\rm j}$	$5.66\pm0.52^{\mathrm{e}}$	$29.94\pm0.49^{ m e,f}$
2-50 after 30 days	$31.70\pm0.59^{\mathrm{e}}$	$10.08\pm0.12^{\rm e,f,g}$	$11.64\pm0.23^{\rm f}$	$15.39\pm0.25^{\rm f}$	$49.10 i \pm 0.35^{ij}$	$5.62\pm0.44^{\mathrm{e}}$	$29.98\pm0.50^{\rm e,f}$
2-65 after production	$28.46\pm0.39^{\rm a}$	$8.09\pm0.10^{\mathrm{a}}$	$7.60\pm0.24^{\mathrm{a}}$	11.09 ± 0.23^{a}	$43.21\pm0.62^{\rm b,c}$		27.60 ± 0.32^{a}
2-65 after 5 days	$29.23\pm0.19^{ m b}$	$8.03\pm0.03^{\mathrm{a}}$	$7.21\pm0.16^{\mathrm{a}}$	$10.79\pm0.11^{\mathrm{a}}$	$41.92\pm0.61^{\mathrm{a}}$	$0.88\pm0.18^{\rm a}$	$28.42\pm0.17^{\rm b}$
2-65 after 10 days	$29.89\pm0.52^{ m c}$	$8.74\pm0.25^{ m b}$	$8.41\pm0.42^{ m b}$	$12.13\pm0.46^{\rm b}$	$43.87\pm0.65^{\rm c.d}$	$1.77\pm0.62^{ m b}$	$28.85\pm0.39^{\rm b,c}$
2-65 after 15 days	$31.77 \pm 0.41^{ m e,f}$	$10.13\pm0.17^{\rm f,g}$	$10.81\pm0.38^{\rm e}$	$14.82\pm0.39^{\rm e}$	$46.84\pm0.58^{\rm f}$	$5.06\pm0.46^{ m d,e}$	$30.18\pm0.33^{\rm f,g}$
2-65 after 20 days	$32.23\pm0.51^{\rm e.f.g}$	$8.94\pm0.08^{\rm c}$	$8.98\pm0.12^{ m c}$	$12.67\pm0.12^{ m c}$	$45.13\pm0.39^{\rm e}$	4.11 ± 0.43^{c}	$31.05\pm0.45^{\rm h}$
2-65 after 25 days	$32.01\pm0.34^{ m e,f,g}$	$9.91\pm0.19^{ m e}$	$11.02\pm0.43^{\mathrm{e}}$	$14.82\pm0.44^{\mathrm{e}}$	$48.02\pm0.61\mathrm{g,h}$	$5.26\pm0.50^{\rm d,e}$	$30.42\pm0.23^{\rm f,g}$
2-65 after 30 days	$32.29\pm0.38^{\rm f,g}$	$10.27\pm0.14^{ m g}$	$11.10\pm0.28^{\mathrm{e}}$	$15.13\pm0.25^{\rm e,f}$	$47.21\pm0.73^{\rm f,g}$	$5.64\pm0.37^{ m e}$	$30.62\pm0.31^{\rm g,h}$

Table 4. Colour changes of the dark chocolate filled with bitter herbal liqueur, with cocoa shell as a filler (chocolate sample 3) during accel bloom for 30 days (12 h at 20 °C/12 h at 29 °C)	c (chocolate sample 3) during accelerated	
	e 4. Colour changes of the dark chocolate filled with bitter herb	bloom for 30 days (12 h at 20 °C/12 h at 29 °C)

fat

Sample	г.	a*	D*	0	n ⁻	ΔE	W1
3-50 after production	$29.21\pm0.04^{\rm a}$	$8.26\pm0.17^{\rm a,b}$	$7.46\pm0.24^{\rm a,b,c}$	$11.27 \pm 0.41 {\rm ^{a,b,c}}$	$42.79 ~\pm~ 1.24^{a}$		28.34 ± 0.02^{a}
3-50 after 5 days	$30.32\pm0.24^{\mathrm{b}}$	$8.71\pm0.26^{ m c}$	$8.03\pm0.54^{\circ}$	$11.86\pm0.54^{\rm c,d}$	42.62 ± 1.22^{a}	$1.40\pm0.38^{\rm a,b}$	$29.32\pm0.20^{\rm c,d}$
3-50 after 10 days	$30.65\pm0.67^{\rm b,c}$	$9.22\pm0.32^{ m d}$	$9.10\pm0.42^{ m d,e}$	12.95 ± 0.51 e.f	$44.62 \pm 0.55^{\rm b}$	$2.40\pm0.71^{ m c}$	$29.45\pm0.51\mathrm{c.d.e}$
3-50 after 15 days	$32.18\pm0.65^{\rm e}$	$10.45\pm0.35^{ m g}$	$10.90\pm0.56^{\rm h}$	$15.10\pm0.65^{\rm i}$	$46.19\pm0.61^{\rm c,d}$	$5.05\pm0.81^{\rm f}$	$30.51\pm0.45^{\rm f}$
3-50 after 20 days	$31.35\pm0.72^{ m d}$	$9.60\pm0.29^{\mathrm{e}}$	$9.52\pm0.46^{\rm e,f}$	$13.52\pm0.51^{\rm f,g}$	$44.74\pm0.71^{\rm b}$	$3.26\pm0.76^{\rm d,e}$	$30.03\pm0.55\mathrm{e,f}$
3-50 after 25 days	$31.51\pm0.51^{\rm d}$	$9.96\pm0.28^{\rm f}$	$11.02\pm0.50^{\rm h}$	$14.86\pm0.55^{\rm i}$	$47.88\pm0.58^{\rm e}$	$4.58\pm0.65^{\rm e,f}$	$29.91\pm0.36^{\mathrm{e}}$
3-50 after 30 days	$31.63\pm0.46^{\rm d,e}$	$10.04\pm0.17^{\rm f}$	$10.88\pm0.52^{\rm h}$	$14.81\pm0.48^{\rm i}$	$47.25\pm0.98^{\rm d,e}$	$4.56\pm0.61^{\rm e}$	$30.05\pm0.32^{\rm e,f}$
3-65 after production	$29.03\pm0.53^{\mathrm{a}}$	$8.08\pm0.16^{\rm a,b}$	7.41 ± 0.41 a.b	$10.95\pm0.39^{\rm a,b}$	42.35 ± 1.11^{a}		28.19 ± 0.43^{a}
3-65 after 5 days	29.13 ± 0.39^{a}	$7.97\pm0.15^{ m a}$	$7.11\pm0.35^{\mathrm{a}}$	$10.69\pm0.32^{\mathrm{a}}$	41.72 ± 1.11^{a}	$0.56\pm0.18^{\mathrm{a}}$	$28.33\pm0.30^{\rm a}$
3-65 after 10 days	29.66 ± 0.53^{a}	$8.38\pm0.10^{\rm b}$	$7.76\pm0.31^{ m b,c}$	$11.42\pm0.27^{ m b.c}$	$42.82\pm0.86^{\rm a}$	$0.82\pm0.49^{\mathrm{a}}$	$28.73\pm0.43^{\rm a,b}$
3-65 after 15 days	$30.62 \pm 0.52^{b,c}$	$9.87\pm0.22^{ m e,f}$	$10.05\pm0.61^{\rm f,g}$	$14.09\pm0.59^{\rm g,h}$	$45.46 \pm 1.16^{\mathrm{b,c}}$	$3.58\pm0.68^{\rm d}$	$29.21\pm0.37^{b,d}$
3-65 after 20 days	30.57 ± 0.27 b.c	$8.92 \pm 0.32^{\mathrm{c,d}}$	$8.76\pm0.60^{\rm d}$	$12.50\pm0.62^{\rm d,e}$	$44.46\pm1.22^{\rm b}$	$2.24\pm0.55^{\rm b,c}$	$29.45\pm0.17^{c.d.e}$
3-65 after 25 days	$31.21\pm0.67^{\rm c,d}$	$9.89\pm0.20^{\rm e,f}$	$10.95\pm0.30^{ m h}$	$14.75\pm0.35^{\rm i}$	$47.89\pm0.34^{\rm e}$	$4.56\pm0.44^{\rm e,f}$	$29.64\pm0.58^{\rm d,e}$
3-65 after 30 days	$30.45\pm0.32^{\mathrm{b}}$	$9.92\pm0.24^{ m f}$	$10.62\pm0.71\mathrm{g,h}$	$14.53\pm0.66^{\rm h,i}$	$46.92\pm1.35^{\rm d,e}$	$3.98\pm0.61^{\rm d,e}$	$28.95\pm0.30^{\rm b,c}$

Table 5. Colour changes of the dark chocolate filled with bitter herbal liqueur, with cocoa shell as a filler and added aroma (chocolate sample 4) during accelerated fat bloom for 30 days (12 h at 20 $^{\circ}C/12$ h at 29 $^{\circ}C)$

Sample	L^*	a^*	\mathbf{b}^*	С	$^{\rm h^{\circ}}$	ΔE	MI
4-50 after production	28.66 ± 0.42^{a}	$8.22\pm0.06^{\rm a}$	7.56 ± 0.12^{a}	11.09 ± 0.11^{a}	$42.84\pm0.87^{\rm ab}$		$27.79\pm0.37^{\mathrm{a}}$
4-50 after 5 days	$30.18\pm0.39^{\rm b,c}$	$8.93\pm0.15^{\rm c}$	$8.82\pm0.25^{ m c}$	$12.55\pm0.28^{ m c}$	$44.65\pm0.35^{\rm d}$	$2.12\pm0.29^{\rm b,c}$	$29.06\pm0.35^{b,c}$
4-50 after 10 days	$30.27 \pm 0.43^{\circ}$	$9.35\pm0.25^{ m d}$	$9.61\pm0.42^{ m d}$	$13.41\pm0.46^{\rm d}$	$45.77\pm0.70^{ m e}$	$2.85\pm0.51^{\rm c,d}$	$28.99\pm0.33^{\rm b,c}$
4-50 after 15 days	$31.53\pm0.72^{ m d}$	$10.27\pm0.15^{\rm g}$	$11.26\pm0.32^{\rm f,g}$	$15.24\pm0.33^{\rm g}$	$47.61\pm0.47^{\rm f,g}$	$5.13\pm0.53^{ m g}$	$29.86\pm0.60^{\rm d}$
4-50 after 20 days	$30.97 \pm 0.30^{ m c,d}$	$9.75\pm0.27^{\mathrm{e}}$	$10.25\pm0.66^{\rm e}$	$14.15\pm0.66^{\rm e}$	$46.41\pm1.08^{\rm e}$	$3.89\pm0.53^{ m e.f}$	$29.53 \pm 0.30^{c,d}$
4-50 after 25 days	$31.57\pm0.05^{ m d}$	$9.94\pm0.09^{\rm e,f}$	$11.39\pm0.23^{ m g}$	$15.12\pm0.23^{\rm f,g}$	$48.88\pm0.38^{\rm h}$	$5.11\pm0.18^{ m g}$	29.92 ± 0.07^{d}
4-50 after 30 days	$31.45\pm0.96^{ m d}$	$10.08\pm0.30^{\rm f,g}$	$11.03\pm0.46^{\rm f}$	$14.94\pm0.53^{\rm f,g}$	$47.58\pm0.54^{\rm f,g}$	$4.85\pm0.84^{\rm g}$	29.84 ± 0.75^{d}
4-65 after production	$28.76\pm0.33^{\mathrm{a}}$	$8.09\pm0.11^{\rm a}$	$7.68\pm0.15^{a,b}$	11.15 ± 0.16^{a}	$43.49\pm0.49^{\rm bc}$		$27.89\pm0.29^{\mathrm{a}}$
4-65 after 5 days	$29.20\pm0.56^{\rm a}$	8.01 ± 0.32^{a}	$7.22\pm0.43^{ m a}$	10.78 ± 0.53^{a}	$42.02\pm0.69^{\rm a}$	$0.93\pm0.13^{\mathrm{a}}$	$28.38\pm0.43^{\rm a,b}$
4-65 after 10 days	$29.39\pm1.22^{\rm a,b}$	$8.57\pm0.43^{ m b}$	$8.18\pm0.83^{\rm b}$	$11.85\pm0.88^{\rm b}$	$43.60\pm1.57^{\rm b,c,d}$	$1.34\pm0.99^{\rm a,b}$	$28.40\pm0.95^{\rm a,b}$
4-65 after 15 days	$31.64\pm0.25^{ m d}$	$10.12\pm0.19^{\rm f,g}$	$10.79\pm0.36^{\rm e,f}$	$14.80\pm0.37^{\rm f,g}$	$46.82 \pm 0.71^{ m e,f}$	$4.71\pm0.40^{\rm f,g}$	$30.05\pm0.16^{\rm d}$
4-65 after 20 days	$31.46\pm1.24^{ m d}$	$9.23\pm0.19^{ m d}$	$9.08\pm0.45^{ m c,d}$	12.95 ± 0.43 ^{c,d}	$44.54\pm1.01\mathrm{cd}$	$3.29\pm1.08^{\rm d,e}$	$30.25\pm1.06^{\rm d}$
4-65 after 25 days	$31.45\pm0.33^{ m d}$	$9.77\pm0.12^{\mathrm{e}}$	$10.85\pm0.26^{\rm f,g}$	$14.60\pm0.27^{\rm e,f}$	$48.00\pm0.37^{\rm g,h}$	$4.50\pm0.30^{\rm f,g}$	29.91 ± 0.28^{d}
4-65 after 30 days	$31.67\pm0.61^{ m d}$	$9.95\pm0.22^{ m e,f}$	$10.94\pm0.87^{\rm f,g}$	$14.79\pm0.79^{\rm f,g}$	$47.63\pm1.59^{\rm f,g}$	$4.76\pm0.94^{\rm f,g}$	30.08 ± 0.39^{d}

When comparing samples 3 and 4 (Table 4 and 5), both of which had a cocoa shell in their fillings, it can be concluded that sample 4 had higher values of total colour change and whiteness index after 30 days than sample 3, especially at the relative humidity of 65%. Sample 2 also had higher values of these parameters compared to sample 1 at that same humidity. Both samples with higher values had the aroma of bitter herbal liqueur in their composition, which leads to the conclusion that this aroma caused changes on the surface of chocolates. It is possible that aroma evaporation from chocolate filling also caused changes on the surface of the chocolate.

Colour changes show that ethanol from the filling causes changes in chocolate surface, especially at fluctuations of temperature and high relative humidity. Bohme et al. (2012) concluded that alcoholic filling in chocolate causes changes in the chocolate shell that envelopes it. Ethanol tends to evaporate at lower temperatures than water, and in such conditions (as in the present research) can cause micro-cracks in a chocolate shell. In their study, it was shown that ethanol content increases in chocolate shells during the storage period where fluctuation of temperature is also present. After the migration of ethanol to the chocolate surface, this compound dissolves sugar at the surface of the chocolate shell. After moisture and ethanol evaporate from the surface of the chocolate, sugar crystals remain on the surface in the form of bloom (Geschwindner and Drouven, 2009). Since chocolates with added cocoa shells showed better stability (lower total colour change and whiteness index) it can be concluded that fibres from cocoa shells could hinder the migration of ethanol.

As can be seen from Table 6, all chocolates had the highest gloss immediately after the production. After the first 5 days, all chocolates had significantly lower gloss than after the production. This is probably due to temperature fluctuation and the humidity because relative humidity of 65% showed a greater effect on gloss loss than <50%. In addition, after 30 days chocolates at higher humidity had lower gloss than at lower relative humidity. The filling of chocolates also showed an effect on this parameter, especially in chocolates stored at a relative humidity of <50%. Chocolates that did not have added bitter herbal aroma had higher values of gloss compared to those with the added aroma. Samples without added aroma showed that ones with cocoa shells had lower gloss values than those with guar gum. With the addition of aroma in the filling, gloss values were opposite.

Fat bloom formation of chocolates was increased because of storage conditions (fluctuation of temperature). During the period of 30 days, in addition to sugar bloom, whitening of the surface in the form of white line occurs because of cocoa butter melting and solidifying in an unstable cocoa butter crystal form (Pastor et al., 2007).

Table 6. Gloss of dark chocolates with bitter herbal liqueur filling during accelerated fat bloom for 30 days (12 h at 20 °C/12 hat 29 °C)

	1-50 (GU)	2-50 (GU)	3-50 (GU)	4-50 (GU)
After production	$25.54\pm8.93^{\text{b}}$	9.30 ± 1.13^{d}	$16.54\pm5.13^{\rm c}$	7.18 ± 1.34^{e}
After 5 days	3.04 ± 2.71^{a}	$0.70\pm0.11^{a,b}$	12.30 ± 4.07^{b}	$1.90\pm0.30^{b,c}$
After 10 days	2.66 ± 3.59^{a}	$0.38\pm0.08^{\rm a}$	$1.44\pm0.86^{\rm a}$	$0.40\pm0.06^{\rm a}$
After 15 days	3.92 ± 1.09^{a}	$1.12\pm0.12^{a,b}$	$2.80\pm0.68^{\rm a}$	$1.40\pm0.54^{a,b}$
After 20 days	$1.74\pm0.62^{\rm a}$	1.26 ± 0.34^{b}	$2.36\pm0.54^{\rm a}$	$0.98\pm0.35^{a,b}$
After 25 days	$5.62\pm2.99^{\mathrm{a}}$	1.24 ± 0.34^{b}	$2.64\pm0.30^{\rm a}$	$2.62\pm0.90^{\text{c,d}}$
After 30 days	6.98 ± 2.33^{a}	$2.10\pm0.70^{\circ}$	$4.46 \pm 1.94^{\rm a}$	$3.20\pm1.20^{\rm d}$
	1-65 (GU)	2-65 (GU)	3-65 (GU)	4-65 (GU)
After production	$17.76 \pm 7.73^{\mathrm{B}}$	$6.84\pm0.91^{\rm D}$	$12.72\pm4.05^{\mathrm{B}}$	$2.36\pm0.86^{\rm C}$
After 5 days	$0.36\pm0.33^{\rm A}$	$0.46\pm0.30^{\rm A}$	$0.22\pm0.13^{\rm A}$	$0.56\pm0.31^{\rm A}$
After 10 days	$0.68\pm0.29^{\rm A}$	$1.00\pm0.53^{A,B}$	$0.62\pm0.17^{\rm A}$	$0.12\pm0.08^{\rm A}$
After 15 days	$0.84\pm0.10^{\rm A}$	$1.16\pm0.35^{\text{A},\text{B},\text{C}}$	$0.86\pm0.24^{\rm A}$	$0.96\pm0.37^{\mathrm{A},\mathrm{B}}$
After 20 days	$1.02\pm0.25^{\rm A}$	$1.30\pm0.55^{\mathrm{B},\mathrm{C}}$	$0.88\pm0.20^{\rm A}$	$0.84\pm0.41^{\mathrm{A},\mathrm{B}}$
After 25 days	$1.10\pm0.53^{\rm A}$	$1.78\pm0.61^{\mathrm{B,C}}$	$0.98\pm0.35^{\rm A}$	$1.66\pm0.62^{\text{B},\text{C}}$
After 30 days	$1.10\pm0.44^{\rm A}$	$1.86\pm0.31^{\rm C}$	$1.38\pm0.88^{\rm A}$	$1.96 \pm 1.08^{\circ}$

*1- dark chocolate filled with guar gum, and bitter herbal liqueur; 2- dark chocolate filled with guar gum, bitter herbal liqueur, and aroma f bitter herbal liqueur; 3- dark chocolate filled with cocoa shell, and bitter herbal liqueur; 4-dark chocolate filled with cocoa shell, bitter herbal liqueur, and aroma of bitter herbal liqueur; 50 and 65 represent relative humidities at which chocolates were stored (<50 and 65% respectively); All values with the same letters in the same column are not significantly different at $p \ge 0.05$

Sensory analysis

Results of the sensory analysis of chocolates are presented in Figure 2. It shows that the appearance of samples 1 and 2, which had guar gum in the composition of filling, had slightly lower points than samples 3 and 4, which had a cocoa shell in their composition.

Sample 3 had the best-rated structure, break and firmness and sample 1 had the best-rated odour. According to the ratings of the sensory panel, sample 1 had the best taste compared to the other samples, followed by sample 4 whose taste score was also high. Sensory properties are one of the most important parameters that need to be examined when developing a new product. They show consumers' liking and preference which are affected by the product quality and attributes (Herawati et al., 2019).

Figure 3 shows that sample 3, produced with cocoa shell as a filler, received the highest total weighted score (12.92), followed by sample 4, with cocoa shell and aroma, with a total weighted score of 12.80. Sample 1, with guar gum as a filler, received a 12.65 total weighted score, while sample 2, with guar gum

and aroma, received a total weighted score of 12.05. According to the total number of weighted points, all samples of filled chocolate belonged to the same quality category. An acceptable quality category was determined for all samples of filled chocolate ranging from a total weighted score of 11.2 to 13.1. The above shows that the best-rated dark chocolates contained cocoa shells in the filling. The cocoa shell contains different bioactive and aroma components typical for cocoa (Okiyama et al., 2017), which could affect the acceptability of these chocolates.

Consumer perception of a new product

Figure 4 shows that men prefer chocolate filled with bitter herbal liqueur more than women (78.74% and 59.68%, respectively). According to the age of consumers, chocolate with bitter herbal liqueur would be the most preferred among the age of 18-30 years (75.51%). All other age groups also preferred this type of chocolate, except for groups 41-50 and the group of those who are more than 61 years old. More than 50% of these groups answered no.

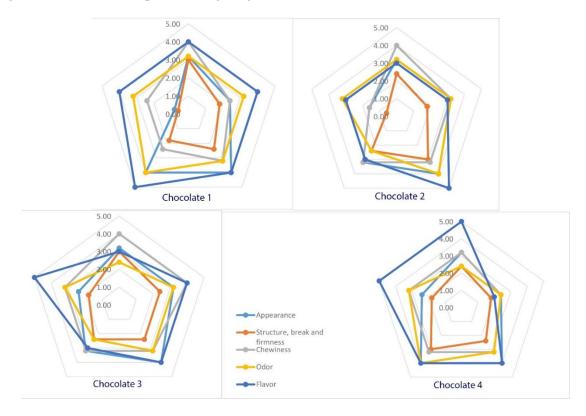


Figure 2. Sensory properties of chocolates filled with bitter herbal liqueur (1- dark chocolate filled with guar gum, and bitter herbal liqueur; 2- dark chocolate filled with guar gum, bitter herbal liqueur, and aroma of bitter herbal liqueur; 3- dark chocolate filled with cocoa shell, and bitter herbal liqueur; 4-dark chocolate filled with cocoa shell, bitter herbal liqueur, and aroma of bitter herbal liqueur)

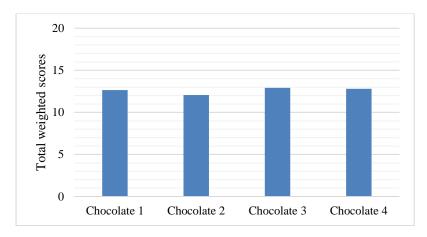


Figure 3. Total weighted scores of chocolates with bitter herbal liqueur filling (1- dark chocolate filled with guar gum, and bitter herbal liqueur; 2- dark chocolate filled with guar gum, bitter herbal liqueur, and aroma of bitter herbal liqueur; 3- dark chocolate filled with cocoa shell, and bitter herbal liqueur; 4-dark chocolate filled with cocoa shell, bitter herbal liqueur, and aroma of bitter herbal liqueur)

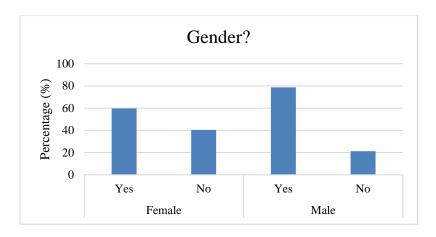


Figure 4. Consumer's preference of chocolate filled with bitter herbal liqueur according to gender

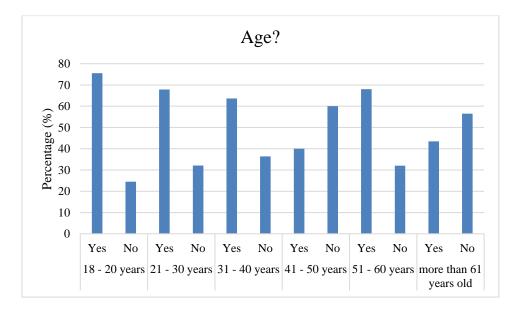


Figure 5. Consumer's preference of chocolate filled with bitter herbal liqueur according to age

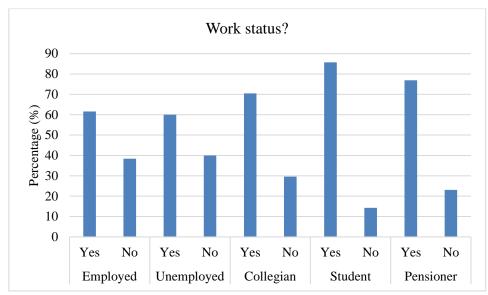


Figure 6. Consumer's preference of chocolate filled with bitter herbal liqueur according to work status

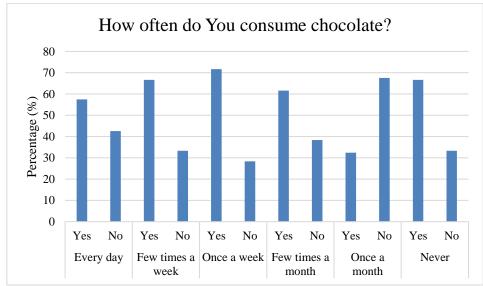


Figure 7. Consumer's preference of chocolate filled with bitter herbal liqueur according to frequency of chocolate consumption

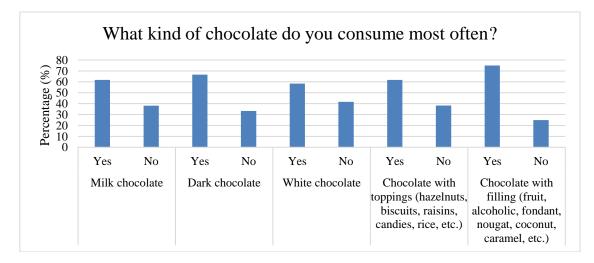


Figure 8. Consumer's preference of chocolate filled with bitter herbal liqueur according to chocolate they prefer

According to work status (Figure 6), a group that would be the most interested in the consumption of chocolate filled with bitter herbal liqueur would be college students. All other groups also showed a great interest (more than 50%) in consumption. The lowest interest for consumption was among the unemployed population. Most respondents stated that they consume chocolate several times a week, and 66.67% of them would be interested in the consumption of chocolate filled with bitter herbal liqueur. Of the respondents who consume chocolate once a week, 71.67% stated that they would be interested in this chocolate, while most respondents that consume chocolate once a month would not be interested in this chocolate. Interestingly, 66.67% of respondents who never consume chocolate would try this combination (Figure 7).

In this questionnaire, the majority of respondents answered that they consume chocolate with toppings and 61.78% of these respondents would also be interested in chocolate filled with bitter herbal liqueur. The group of respondents that would most likely be interested in the consumption of this chocolate was the one that prefers chocolate with different fillings (Figure 8). The second most interested group was the one that prefers dark chocolate, which can be because they favour the bitter taste that is characteristic of dark chocolate.

Out of the respondents who stated that they consumed liqueurs, 76.08% would be interested in chocolate filled with bitter herbal liqueur, while respondents who did not consume liqueurs mostly would not be interested in the consumption of this chocolate (61.84%) (Figure 9).

Most respondents stated that they consume bitter liqueur and 83.42% would be interested in chocolate filled with bitter herbal liqueur. Most respondents who consume special liqueurs or do not like liqueurs would not be interested in this chocolate (Figure 10).

Most respondents who consume bitter herbal liqueur would also be interested in its combination with chocolate. Most respondents stated that they consume bitter herbal liqueur several times a year, and 80.69% of them would be interested in this type of chocolate. Respondents who consume this liqueur several times a month would be most interested in this chocolate, namely 93.83% of them. Interestingly, 72.09% of respondents who never consume bitter herbal liqueur would be interested in the consumption of this chocolate (Figure 11).

Respondents who tried chocolate with alcoholic filling would be interested in chocolate filled with bitter herbal liqueur (66.09%), while respondents who did not try chocolate with alcoholic filling, in general, would not be interested in such chocolate (Figure 12). From this questionnaire, we can conclude that chocolate with bitter herbal liqueur would be acceptable to most of the surveyed consumers. This combination of chocolate and alcohol would be mostly consumed by people aged 18 - 20.

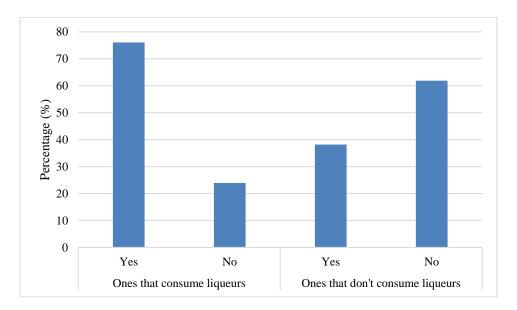


Figure 9. Consumer's preference of chocolate filled with bitter herbal liqueur according to liqueur consumption

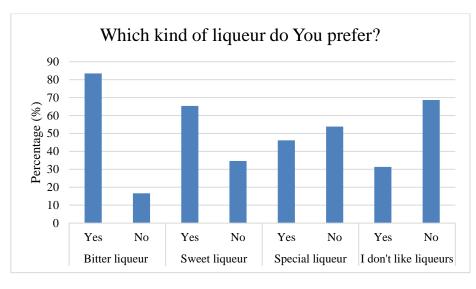


Figure 10. Consumer's preference of chocolate filled with bitter herbal liqueur according to liqueur they prefer

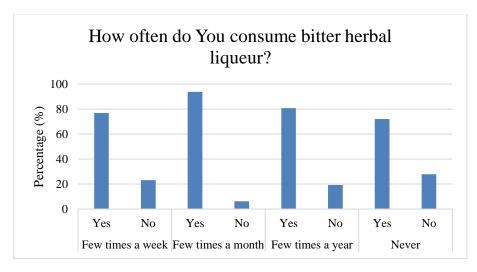


Figure 11. Consumer's preference of chocolate filled with bitter herbal liqueur according to frequency of bitter herbal liqueur consumption

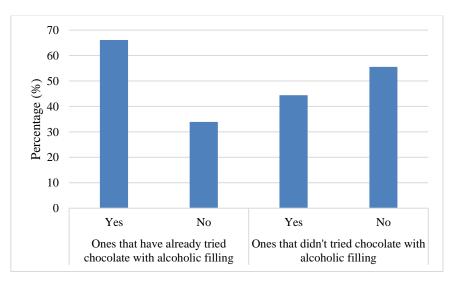


Figure 12. Consumer's preference of chocolate filled with bitter herbal liqueur according to prior consumption of chocolate with alcoholic filling

Conclusions

This paper gives a new perspective for the utilization of cocoa shells as thickeners in chocolate fillings. It was shown that total colour change and whiteness index were lower in chocolates that had a cocoa shell in filling compared to those that contained guar gum. Also, the sensory analysis showed that chocolates with cocoa shells had better overall acceptability and were better rated for flavour compared to chocolates with guar gum. This could be affected by aroma compounds that are present in the cocoa shell. The questionnaire that examined preference for this type of chocolate showed that most of the consumers would be interested in the consumption of chocolates with bitter herbal liqueur, especially the younger population.

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References

- Barišić, V., Jozinović, A., Flanjak, I., Šubarić, D., Babić, J., Miličević, B., Doko, K., Ačkar, Đ. (2020): Difficulties with Use of Cocoa Bean Shell in Food Production and High Voltage Electrical Discharge as a Possible Solution. Sustainability 12 (10), 3981.
- Böhme, B., Kretzschmar, R., Schneider, Y., Fiala, P., Rohm, H. (2012): Effect of Alcohol in Starch-Thickened Fillings on the Storage Stability of Dark Chocolate Pralines. J. Am. Oil Chem. Soc. 89 (3), 447– 454.
- Geschwindner, G., Drouven, H. (2009): Manufacturing processes: chocolate panning and inclusions. In Science and Technology of Enrobed and Filled Chocolate, Confectionery and Bakery Products; Talbot, G., Ed.; Woodhead Publishing Limited, Abington Hall, Granta Park, Great Abington, Cambridge, UK,; pp. 397–413.
- Herawati, E.R.N., Nurhayati, R., Angwar, M. (2019): Sensory, chemical, and nutritional characteristic of coffee-chocolate instant drink (chocomix-fee) from Nglanggeran, Gunungkidul, Yogyakarta. *IOP Conf. Ser.: Mater. Sci. Eng.* 633, 012041.

- Lonchampt, P., Hartel, R. W. (2006): Surface bloom on improperly tempered chocolate. *Eur. J. Lipid Sci. Technol.* 108 (2), 159–168.
- Mudgil, D., Barak, S., Khatkar, B. S. (2011): Guar gum: processing, properties and food applications—A Review. *J. Food Sci. Technol.* 51 (3), 409–418.
- Okiyama, D.C.G., Navarro, S.L.B., Rodrigues, C.E.C. (2017): Cocoa shell and its compounds: Applications in the food industry. *Trends Food Sci. Technol.* 63, 103–112.
- Pastor, C., Santamaría, J., Chiralt, A., Aguilera, J.M. (2007): Gloss and Colour of Dark Chocolate During Storage. *Food Sci. Technol. Int.* 13 (1), 27–34.
- Redgwell, R., Trovato, V., Merinat, S., Curti, D., Hediger, S., Manez, A. (2003): Dietary fibre in cocoa shell: characterisation of component polysaccharides. *Food Chem.* 81 (1), 103–112.

Talbot, G. (2009): Technology of coated and filled chocolate, confectionery and bakery products. Woodhead Publishing Limited, Abington Hall, Granta Park, Great Abington, Cambridge, UK.