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USE OF COCOA SHELL IN INSTANT COCOA POWDERS – RESEARCH OF DIFFERENT HYDROCOLLOID ADDITIONS AND DRYING TECHNIQUES

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original scientific paper

Summary

The aim of this research was to examine the influence of the addition of cocoa shell, xanthan gum, guar gum, gum arabic, and carboxymethylcellulose (CMC), as well as drying procedures on the properties of instant cocoa beverages. The cocoa shell was separated from the cocoa bean after roasting, milled, and after milling a fraction smaller than 71 μm was separated by sieving, and used in production. Instant powders were produced by agglomeration and drying (freeze-drying and oven-drying at 40 °C). The results showed that higher content of cocoa shell in instant cocoa beverages increased the wetting time, while the colour of the oven-dried powders was lighter compared to the freeze-dried samples. The bulk density of the powder was higher in samples with a higher content of shell. In samples with a higher amount of cocoa shell, the content of total polyphenols decreased compared to the samples without cocoa shell.

Keywords: cocoa beverage, cocoa shell, xanthan gum, guar gum, gum arabic, CMC, oven-drying, freeze-drying

Introduction

Instant cocoa powder is produced from cocoa powder, sugar and emulsifier as main raw materials, to which milk powder and hydrocolloids may be added as well, depending on the recipe. It is easily reconstituted in water or milk and has a long storage period. The production process includes agglomeration to increase dispersibility of the powder in milk or water. The agglomeration is a process in which cocoa powder is mixed with sugar and stabilizers with the addition of water to produce larger conglomerates, which are afterwards dried (Benković et al., 2015). Prolonged drying at elevated temperatures may cause loss of vitamins, minerals and other bioactive compounds, as well as influence colour and aroma of the final product. In addition, sugar crystallizes at relatively low temperatures and may cause problems during drying (Sedlar and Pervan, 2010; Hardy and Jideani, 2017). The alternative for conventional drying would be freeze-drying. The advantages of this process are the application of low temperatures and preservation of thermolabile compounds, better rehydration properties, and better efficiency of production. However, it requires higher energy input, both for freezing and freeze-drying, and demands moisture- and air-tight proof packaging (Hardy and Jideani, 2017).

As the demand for cocoa is increasing, while cocoa supply remains limited, it is justified to seek an alternative for cocoa powders in instant cocoa beverages. Cocoa shell is a by-product of cocoa industry with characteristic cocoa aroma, and is also rich in fibres, proteins and bioactive compounds. Use of cocoa shell in processing of cocoa products would reduce negative ecological and environmental effects of cocoa industry, and increase the production efficiency (Okiyama et al., 2017).

In our previous researches (Trgovac et al., 2022; Barišić et al., 2022; Barišić et al., 2021), we have successfully implemented cocoa shell in production of chocolate and chocolate fillings, and the aim of this research was to explore potential of using cocoa shell in production of instant cocoa beverages. Therefore, we investigated different proportions of cocoa shell along with the addition of different hydrocolloids (xanthan gum, guar gum, gum acacia and carboxymethyl cellulose (CMC)) as stabilizers, and compared the influence of oven-drying and freeze-drying.

Materials and methods

Samples

Cocoa shell was separated from the beans after the roasting, milled and sieved to obtain particles smaller than 71 μm.

Instant cocoa powders (Figure 1) were prepared according to recipes shown in Table 1, using cocoa

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powder (Kandit d.d. Osijek, Croatia), cocoa shell (substituting cocoa powder in proportions 5, 10 and 15 %), 69.5% sugar (Viro d.d. Virovitica, Croatia) and 0.4% lecithin (ACEF; Italy). Hydrocolloids: xsanthan gum, guar gum, gum arabic, powdered and liquid CMC were added in 0.1%. Instant powders were produced by agglomeration and drying in oven at 40 °C or by freeze-drying at the pressure of 0.25 mbar. Before freeze-drying, samples were frozen in ultra-freezer at –80 °C.

Table 1. Recipes of instant cocoa powders

SAMPLE*	COCOA POWDER (%)	COCOA SHELL (%)	XANTHAN GUM (%)	GUAR GUM (%)	GUM ARABIC (%)	CMC POWDER (%)	CMC LIQUID (%)
KG	30	0	0.1				
GG	30	0		0.1			
GA	30	0			0.1		
CP	30	0				0.1	
CT	30	0					0.1
KG-LJ5	25	5	0.1				
GG-LJ5	25	5		0.1			
GA-LJ5	25	5			0.1		
CP-LJ5	25	5				0.1	
CT-LJ5	25	5					0.1
KG-LJ10	20	10	0.1				
GG-LJ10	20	10		0.1			
GA-LJ10	20	10			0.1		
CP-LJ10	20	10				0.1	
CT-LJ10	20	10					0.1
KG-LJ15	15	15	0.1				
GG-LJ15	15	15		0.1			
GA-LJ15	15	15			0.1		
CP-LJ15	15	15				0.1	
CT-LJ15	15	15					0.1

*All samples contained 69.5% sucrose and 0.4% lecithin.

Figure 1. Samples of instant cocoa powder with powdered CMC before drying (left to right: sample without cocoa shell, sample with 5% cocoa shell, sample with 10% cocoa shell and sample with 15% cocoa shell)

Colour

Colour was measured in CIEL*a*b* and LCh° systems, using chromameter (Konica Minolta CR-400, Tokio, Japan) using extension for powdered samples. Five measurements were done for each sample, and an average and standard deviation were calculated for the expression of the results.

Parameters are as follows:
L* - lightness (0 – black, 100 – white)
a* - negative values are green domain, positive ones are in red
b* - negative values are in blue domain, positive in yellow
C – chroma, the distance from the lightness axis, starts with 0 in the center
h° – hue, starts in red axis: 0 ° is +a*, 90 °C is +b*

Total colour difference was calculated in relation to the sample with xanthan gum, according to Equation 1:

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

where: L^*_0, b^*_0, a^*_0 represent values for sample with xanthan gum.

Wettability

Wettability of instant cocoa powders was determined according to Schubert (1980), and the obtained results are expressed as the time (in seconds, s) needed for all the powder (2 g) to wet and penetrate through the surface of the distilled water (20 mL) at ambient temperature. All analyses were done in triplicates.

Bulk density

Bulk density was determined according to Escalada Pla et al. (2012). Each sample was weighed at the analytical balance into the cup to fill the pre-determined volume. All analyses were done in triplicates.

Total phenolic compounds

The extraction of phenolic compounds was done according to Adamson et al. (1999). Instant cocoa powders were defatted with *n*-hexane, where 2 g of sample was extracted with 10 mL of *n*-hexane three times and air-dried for 24 h. Defatted samples were extracted three times with 5 mL of 70% methanol, using ultrasound for 30 min. Samples were centrifuged at 3000 rpm for 10 min and supernatants were collected in 10 mL-measuring flask and filled up with 70% methanol.
Modified Folin-Ciocalteu method, in acidic conditions (without Na_2CO_3) was used for measurement of total phenolic compounds (Vinson et al., 2001) to avoid interference of sugars. To 100 μL of the sample, 1 mL of 10% Folin-Ciocalteu reagent was added. After 2 min, the mixture was incubated for 20 min at ambient temperature in dark. Absorbance was measured at 750 nm, and quantification was done using gallic acid as the standard (concentration range 0.14 – 0.70 mg/mL). The results are expressed as mg of gallic acid equivalent per g of defatted sample (mg GAE/g).

Results and discussion

Samples of instant cocoa powder were prepared according to 20 recipes shown in Table 1. A part of each sample was oven-dried at 40 °C, and the other part was freeze-dried, so in total 40 samples were prepared. Colour parameters of the samples are shown in the Table 2. In the oven-dried samples, although in majority of cases cocoa shell added in 5% caused a slight decline of L* values, the increase of its proportion in the powder caused an increase of L* values. In freeze-dried samples this trend was not so pronounced, although it is visible that increase of cocoa shell contents generally increases L* values. Additionally, when compared to oven-dried counterpart, freeze-dried samples had lower values of L*, indicating that they were darker. The differences between a* and b* values between oven- and freeze-dried samples were less pronounced, also with lower values detected in freeze-dried samples. When comparing hydrocolloids, CMC resulted in higher values of measured colour parameters than other three. However, combined effect of all colour parameters are brown in all samples, with small differences in the shade. This is supported by calculated ΔE values shown in Figure 2. All ΔE values are below 4, what shows that colour differences are perceptible by human eye only by close observation. Chroma values and hue were also higher for oven-dried samples. All these imply that consumers would prefer oven-dried samples due to more pronounced and "clearer" brown, which is associated with higher content of cocoa (Folkenberg et al., 2007). Redgwell et al. (2003) reported higher proportions of fibers, mono- and polysaccharides in cocoa shell in relation to cocoa bean. Fibres are good water adsorbents and could contribute to darkening, along with Maillard reactions products and protein-tanin complexes which are usually generated during the thermal processing.

Table 2. Colour parameters of instant cocoa powders measured by chromameter

SAMPLE	L*	a*	b*	C	h°
OVEN DRYING					
KG	40.90 ± 0.04	10.51 ± 0.05	14.49 ± 0.04	17.90 ± 0.02	54.04 ± 0.19
GG	39.74 ± 0.02	10.88 ± 0.08	14.39 ± 0.06	18.04 ± 0.10	52.91 ± 0.12
GA	41.75 ± 0.02	9.88 ± 0.06	14.17 ± 0.07	17.28 ± 0.05	55.10 ± 0.26
CP	40.26 ± 0.02	10.69 ± 0.07	14.54 ± 0.04	18.05 ± 0.05	53.68 ± 0.19
CT	42.89 ± 0.02	10.83 ± 0.05	15.67 ± 0.05	19.05 ± 0.04	55.34 ± 0.17
KG-LJ5	39.42 ± 0.05	9.69 ± 0.04	13.34 ± 0.05	16.49 ± 0.03	54.01 ± 0.25
GG-LJ5	38.63 ± 0.02	9.80 ± 0.06	13.17 ± 0.02	16.42 ± 0.04	53.31 ± 0.21
GA-LJ5	38.04 ± 0.05	9.57 ± 0.06	12.96 ± 0.04	16.12 ± 0.05	53.75 ± 0.38
CP-LJ5	41.40 ± 0.46	9.60 ± 0.06	13.59 ± 0.03	16.64 ± 0.03	54.78 ± 0.20
CT-LJ5	41.13 ± 0.01	9.92 ± 0.01	13.85 ± 0.16	17.08 ± 0.04	54.51 ± 0.09
KG-LJ10	42.77 ± 0.01	9.50 ± 0.03	14.69 ± 0.13	17.48 ± 0.08	57.18 ± 0.15
GG-LJ10	40.96 ± 0.03	10.05 ± 0.06	14.68 ± 0.01	17.79 ± 0.03	55.61 ± 0.16
GA-LJ10	41.73 ± 0.02	10.26 ± 0.02	15.23 ± 0.03	18.37 ± 0.04	56.02 ± 0.04
CP-LJ10	43.13 ± 0.04	10.11 ± 0.06	15.46 ± 0.04	18.47 ± 0.03	56.82 ± 0.20
CT-LJ10	42.49 ± 0.02	9.62 ± 0.05	14.78 ± 0.03	17.63 ± 0.02	56.93 ± 0.16
KG-LJ15	44.61 ± 0.01	9.78 ± 0.03	16.06 ± 0.03	18.80 ± 0.01	58.65 ± 0.13
GG-LJ15	44.06 ± 0.05	9.71 ± 0.04	15.65 ± 0.03	18.42 ± 0.03	58.17 ± 0.15
GA-LJ15	42.39 ± 0.01	9.77 ± 0.05	15.06 ± 0.04	17.95 ± 0.02	57.03 ± 0.18
CP-LJ15	44.55 ± 0.04	9.55 ± 0.07	15.58 ± 0.06	18.27 ± 0.03	58.49 ± 0.26
CT-LJ15	44.46 ± 0.02	9.60 ± 0.05	15.63 ± 0.04	18.35 ± 0.02	58.43 ± 0.18
FREEZE-DRYING					
KG	37.42 ± 0.02	10.12 ± 0.04	12.55 ± 0.03	16.12 ± 0.01	51.13 ± 0.16
GG	38.81 ± 0.02	10.51 ± 0.04	13.64 ± 0.03	17.22 ± 0.01	52.41 ± 0.15
GA	36.46 ± 0.02	10.96 ± 0.03	12.70 ± 0.03	16.78 ± 0.03	49.18 ± 0.11
CP	38.03 ± 0.01	10.51 ± 0.03	13.23 ± 0.02	16.90 ± 0.01	51.55 ± 0.13
CT	39.54 ± 0.01	9.58 ± 0.05	11.89 ± 0.04	15.27 ± 0.02	51.14 ± 0.25
KG-LJ5	38.04 ± 0.05	9.57 ± 0.06	12.96 ± 0.04	16.12 ± 0.05	53.75 ± 0.38
GG-LJ5	37.59 ± 0.07	9.41 ± 0.05	12.50 ± 0.05	15.65 ± 0.02	53.03 ± 0.26
GA-LJ5	37.30 ± 0.03	9.68 ± 0.04	12.71 ± 0.04	15.98 ± 0.04	52.72 ± 0.13
CP-LJ5	37.39 ± 0.03	10.13 ± 0.06	13.20 ± 0.03	16.65 ± 0.03	52.50 ± 0.22
CT-LJ5	35.92 ± 0.02	10.01 ± 0.04	12.64 ± 0.06	16.12 ± 0.04	51.61 ± 0.20
KG-LJ10	39.71 ± 0.03	9.18 ± 0.07	12.98 ± 0.05	15.90 ± 0.03	54.73 ± 0.27
GG-LJ10	40.10 ± 0.03	9.22 ± 0.01	13.30 ± 0.05	16.18 ± 0.05	55.26 ± 0.08
GA-LJ10	34.97 ± 0.02	9.28 ± 0.04	11.36 ± 0.05	14.50 ± 0.30	50.46 ± 0.23
CP-LJ10	39.46 ± 0.38	9.39 ± 0.12	13.48 ± 0.09	16.43 ± 0.10	55.13 ± 0.38
CT-LJ10	38.36 ± 0.02	9.42 ± 0.04	13.31 ± 0.03	16.30 ± 0.03	54.71 ± 0.15
KG-LJ15	39.49 ± 0.03	9.25 ± 0.05	13.53 ± 0.03	16.39 ± 0.02	55.64 ± 0.20
GG-LJ15	37.16 ± 0.04	9.08 ± 0.03	12.03 ± 0.05	15.28 ± 0.03	52.95 ± 0.19
GA-LJ15	35.69 ± 0.02	9.09 ± 0.04	11.72 ± 0.03	14.84 ± 0.02	52.21 ± 0.19
CP-LJ15	38.57 ± 0.03	9.07 ± 0.05	12.95 ± 0.04	15.82 ± 0.05	55.00 ± 0.16
CT-LJ15	38.99 ± 0.06	8.89 ± 0.06	13.06 ± 0.03	15.80 ± 0.04	55.76 ± 0.21

KG – xanthan gum, GG - guar gum, GA – gum arabic, CP – powdered CMC, CT – liquid CMC, LJ5 – 5% cocoa shell, LJ10 – 10% cocoa shell, LJ15 – 15% cocoa shell; all samples contained 69.5% sugar and 0.4% lecithin

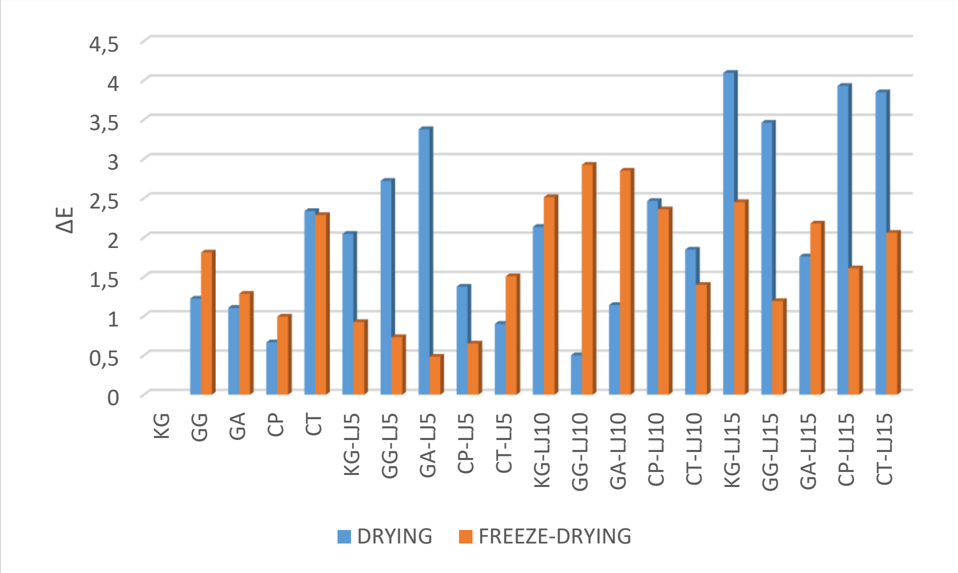

Figure 2. Total colour difference (ΔE) between instant cocoa powder samples dried in certain regime. Sample with xanthan gum was taken as a reference

(KG – xanthan gum, GG - guar gum, GA – gum arabic, CP – powdered CMC, CT – liquid CMC, LJ5 – 5% cocoa shell, LJ10 – 10% cocoa shell, LJ15 – 15% cocoa shell; all samples contained 69.5% sugar and 0.4% lecithin)

Wettability was better in the samples without added cocoa shell (Figure 3). The time needed for all the powder to penetrate through the surface increased with the higher content of cocoa shell. Oven-dried samples with 15% of cocoa shell needed significantly longer time to wet than freeze-dried counterparts, unlike the samples with 5 and 10% of cocoa shell, where better wettability was achieved by oven-drying. Wettability of these samples (app. 10 – 40 s) was in the range reported by Buljat et al. (2019) for samples of foam mat dried cocoa powders. Gum arabic significantly prolonged wetting time when instant cocoa powders were prepared without and with 5% cocoa shell, while samples with higher amounts of shell showed this effect in cases when guar and xanthan were used.. This points out that selection of hydrocolloids should largely depend on the raw materials and their proportions in the formulations. Shittu and Lawal (2005) reported that particle size, fat contents and bulk density influence wettability, while Cvitanović et al. (2010) reported that wettability may be improved by the addition of sugar (it has the highest solubility), agglomeration, addition of lecithin or by their combination.

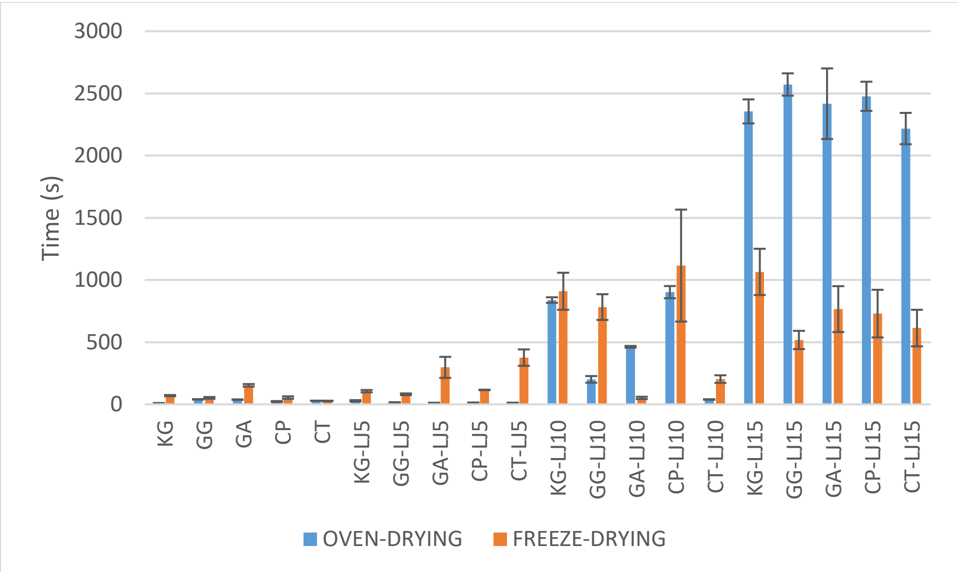

Figure 3. Wettability of instant cocoa powder samples

(KG – xanthan gum, GG - guar gum, GA – gum arabic, CP – powdered CMC, CT – liquid CMC, LJ5 – 5% cocoa shell, LJ10 – 10% cocoa shell, LJ15 – 15% cocoa shell; all samples contained 69.5% sugar and 0.4% lecithin)

Bulk density of the samples ranged 0.95 – 1.09 g/cm^3 for oven-dried, and 0.93 – 1.06 g/cm^3 for freeze-dried samples (Figure 4). Freeze-dried samples had smaller bulk density than their oven-dried counterparts, with the exception of the samples with 10% of cocoa shell. During freeze-drying, water sublimates, while during oven-drying water evaporates. Ice particles take more space than liquid water, therefore larger porosity is a common result of freeze-drying (Buljat et al., 2019). Chaloeichitratham et al. (2018) reported lower bulk density of freeze-dried curry compared to oven-dried samples.

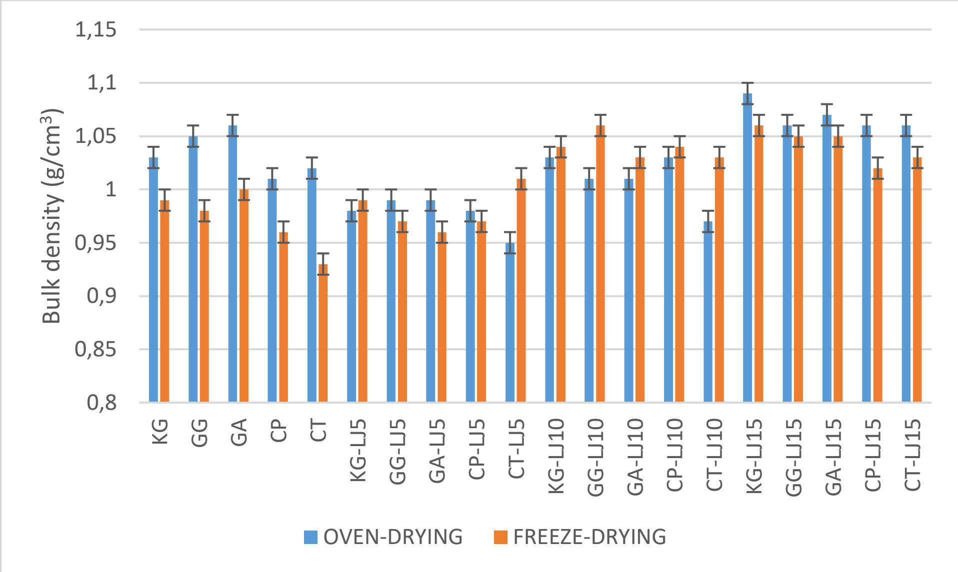

Figure 4. Bulk density of instant cocoa powder samples
(KG – xanthan gum, GG - guar gum, GA – gum arabic, CP – powdered CMC, CT – liquid CMC, LJ5 – 5% cocoa shell, LJ10 – 10% cocoa shell, LJ15 – 15% cocoa shell; all samples contained 69.5% sugar and 0.4% lecithin)

According to literature data, content of phenolics in cocoa powder is 48 ± 2.1 mg/g (Crozier et al., 2011). Contents of total phenolic compounds (TPC) in this research ranged from 18.41 mg GAE/g of defatted sample for the freeze-dried sample with xanthan gum and 15% of cocoa shell to 32.88 mg GAE/g of defatted sample for oven-dried sample with powdered CMC and 5% of cocoa shell (Figure 5). All oven-dried samples except the sample with guar gum and without cocoa shell had higher TPC content than their freeze-dried counterparts, and increase of cocoa shell contents decreased TPC content what was already observed in our previous research on chocolate (Barišić et al., 2020), and in Karim et al. (2014) who reported lower TPC in cocoa shell in relation to cocoa bean. Although freeze-drying preserves thermolabile compounds and higher content of TPC has been reported for freeze-dried cocoa beans (Majid and Rining, 2018) and freeze-dried jujube (Wojdylo et al., 2019), the results of this research may be explained by formation of coloured and reducing compounds during oven-drying (Platzer et al., 2021; Everette et al., 2010). The influence of hydrocolloids on preservation of phenolic compounds did not follow any trend and it depended both on drying process and the proportion of cocoa shell in the sample.

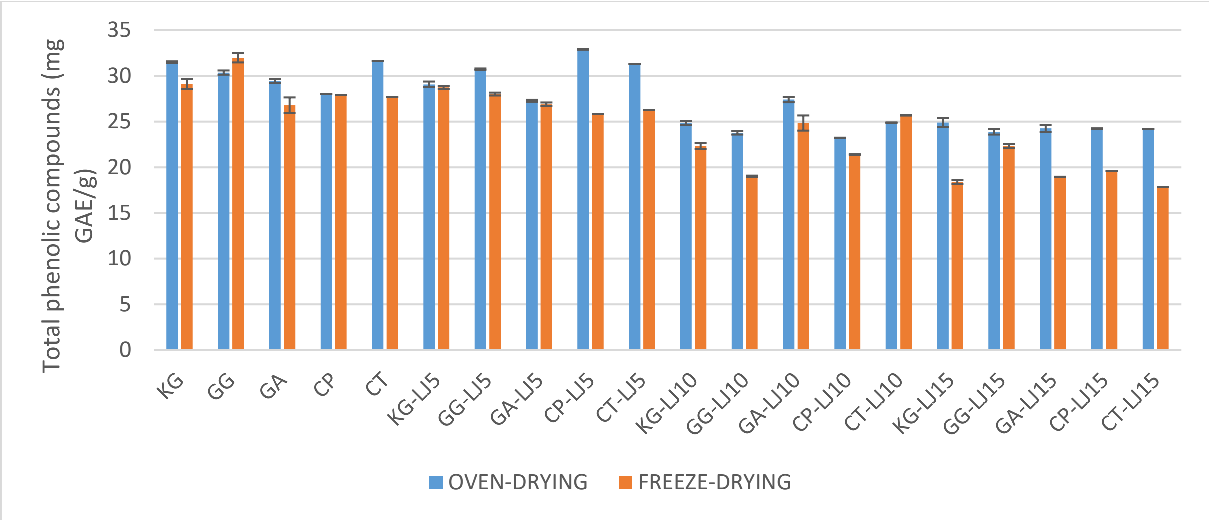

Figure 5. Content of total phenolic compounds of instant cocoa powder samples
(GAE- gallic acid equivalents, KG – xanthan gum, GG - guar gum, GA – gum arabic, CP – powdered CMC, CT – liquid CMC, LJ5 – 5% cocoa shell, LJ10 – 10% cocoa shell, LJ15 – 15% cocoa shell; all samples contained 69.5% sugar and 0.4% lecithin)

Conclusion

The results showed that increasing the content of cocoa shell in instant cocoa beverages increased the wetting time, and the colour of convection-dried powders was lighter compared to the freeze-dried samples. The bulk density of the powders was higher in samples with a higher content of shell. In samples with a higher proportion of cocoa shell, the content of total phenolics decreased compared to the samples without cocoa shell.

Although none of the hydrocolloids used in the research stand out as the best one, additional research on other properties (e.g. microbial quality, particle size, polyphenolic profile, fiber content etc.) and sensory analysis could reveal this issue.

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