

INFLUENCE OF CHIA GEL ON STARCH THERMAL TRANSITIONS, MEASURED BY DSC DURING THE STORAGE OF CAKES

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ABSTRACT

The effect of polysaccharide chia gel, used as fat replacer by substitution of egg incorporation level in cakes on the starch retrogradation during the storage was analyzed by the method of Differential Scanning Calorimetry (DSC). The retrogradation transition appeared in the DSC curves as an endothermic peak with maximum and area correlating to the retrogradation temperature and enthalpy, respectively. The process was monitored during 12 days after cake's preparation and some other product's physical characteristics (weight loss, crumb firmness, crumb color) were also measured. The lowest degree of retrogradation was observed on the 12th day for the 20 % replacement samples, while the highest one was established for the control samples. Therefore, chia gel exhibited the expected retarding effect on starch retrogradation at 15 and 20 % level of egg replacement in the cake batter (low enthalpy values). In general, with increasing chia gel concentration, starch retrogradation slowed down in all storage stages.

Keywords: starch retrogradation, enthalpy, egg replacement, staling, cake storage, phase transition.

INTRODUCTION

Recently, the egg replacement approach has gained considerable interest among consumers for it's been linked to resolving health problems such as egg allergies, phenylketonuria, cholesterol level along with the vegan/vegetarian preferences, religious believes and issues related to economic factors important for producers and sustainable food supply [1]. Ingredients that are generally selected to replace egg carry functional properties, such as emulsification, foaming and viscosity/structure-building abilities in a formulation [2]. It was proven the chia gel can act as a fat substitute, as it can hydrate, improve viscosity and preserve the freshness of bakery products. This behaviour is explained by the water affinity of the emulsifiers, as well as their interactions with the starch, which affects the retrogradation of

the starch and the rate of its progressing [3]. It's been proven the gelatinized starches behaviour during cooling and storage affects considerably quality, consumers' acceptance and in general the shelf-life of the starchy foods [4]. Starch retrogradation is mostly affected by two factors: temperature and moisture content of the baked product [5]. It is negatively correlated with temperature and accelerates as temperature decreases. Studies showed that starch retrogradation slowed down when the moisture content of the starch gel was high [5]. Various chemical and physical methods have been applied to study the changes arising in the starch characteristics manifesting molecular transitions having impact on the properties of the whole material [6 - 9]. It turned to be there was no singular method depicting the retrogradation process at molecular and macroscopic viewpoint. Numerous studies demonstrate

the Differential Scanning Calorimetry (DSC) as reliable tool to characterize starch retrogradation [6, 10]. The DSC thermograms are informative towards the enthalpy change (ΔH) in the new orderly structures' formation after the melting of the crystallite together with the transition temperature parameters (onset, T_0 ; peak, T_p ; and end, T_e) as in the case with the retrograded starch quantitative measures of enthalpy change and transition temperatures of melting of the recrystallized amylopectin. [6].

The aim of the present research was to prove the Differential Scanning Calorimetry (DSC) application as a reliable method to investigate the influence of polysaccharide - chia gel, added in different concentrations, on the staling process in cake systems.

EXPERIMENTAL

The cake batter was prepared by mixing the following ingredients: white wheat flour type 500 (10 % moisture, 11.8 % protein content ($N \times 5.95$), 1.5 % fiber; Sofia Mel, Ltd.), 3.0 % fat cow's milk "My day" (UHT sterilized; Cremio, Ltd.), chicken eggs, sucrose "Sladeya" (Sugar factories, Ltd.), refined sunflower oil (9 % saturated fatty acids; Pearl Olive, Ltd.), dried chia seeds (Dragon superfoods, Mexico), baking powder and vanilla powder, all purchased from the market.

Preparation of the cake batter

The cake formulation variants are shown in Table 1.

The fresh eggs according to the basic recipe were replaced with the chia gel at levels of 10, 15 and 20 %, and the control sample was without the addition of gel.

Beforehand, chia seeds were soaked in drinking water (1:9 for 30 min) to form a gel as described by Borneo at al. [11]. The exact amount to add in each variant was considered (Table 1). The batter prepared in the amount of 450 g was transferred to a standard metal form (standardized method of trial laboratory baking, Department of Technology of cereals, feed, bakery and confectionery products, UFT - Plovdiv) and baked for 30 - 40 min in a conventional electric oven, preheated to 250°C, according to the following scheme: 10 minutes at 250°C, 10 min at 200°C and finally at 180°C until ready.

The control was baked in a preheated oven to 250°C for 35 min at 180°C. After baking the samples were allowed to cool down at room temperature for about 2 h, placed in plastic bags and stored at room temperature for number of days (1, 4, 7, 12 days).

Physical measurements of the cakes

The thermal transitions temperatures and the enthalpy were established by the Differential Scanning Calorimetry (DSC) method application. The state of the water (free or bound) in the cake system over the predefined periods of time was determined. Hardness and color of the crumb, as well as the loss of weight were also measured as the cakes' quality indicators.

Differential Scanning Colorimetry Analysis (DSC)

The differential scanning calorimeter DSC 204 F1 Phoenix NETZSCH-Gerätebau GmbH, Germany was used to study the retrogradation of starch in cakes upon heating. The instrument has been calibrated at certain temperature and enthalpy with an indium standard. The measurements were carried out in an argon atmosphere,

Table 1. Cake formulations recipe – control and samples with eggs partially substituted by chia gel.

Ingredients, 100 g	Control	Eggs replacement, %		
		10	15	20
White flour	25.5	25.5	25.5	25.5
Crystal sugar	33.0	33.0	33.0	33.0
Cow's milk 3.0 %	18.0	18.0	18.0	18.0
Chicken eggs	15.0	13.5	12.75	12.0
Sunflower oil	7.5	7.5	7.5	7.5
Chia gel	-	1.5	2.25	3.0
Baking powder	1.0	1.0	1.0	1.0
Aroma	0.1	0.1	0.1	0.1

at a flow rate of 20 ml/min. Samples of cake products, about 15 mg, are hermetically sealed in aluminium crucibles. They were then heated at 20°C to 150°C at a rate of 10 K/min to conditions like those of the center of the batter during the baking. The endothermic transitions of starch retrogradation were processed using the Proteus Analysis instrument (Netzsch, Germany) software and the enthalpy was expressed as J g⁻¹.

Hardness measurement

The hardness of the products crumb was measured using the StableMicroSystems TA-XT2Plus texture analyser, with a cylindrical probe, 25 mm in diameter, at a compression speed of 1 mm/s and strain fixed at 10 % with a waiting time between two compressions of 5 s [12]. The hardness of cakes crumb was examined by the texture profile method on the 1st, 4th, 7th, and 12th day of the storage. The samples were formed from three cake slices, 20 mm thick and placed one on top of the other. For each sample and for each day of the study, 3 slices were measured at 4 points.

Color analysis

The research was carried out with a colorimeter (PCE - CSM 5 portable colorimeter - Measuring geometry 8°/d, Ø 8 mm, light source D 65) in order to determine the qualitative and quantitative color indicators of the middle of the product following the meaning of the CIE Lab system parameters: L - illuminance (L = 0 - black, L = 100 - white), +a - red color, -a - green color, +b - yellow color, -b - blue color [13]. Each sample color coordinates represent the average values of several measured coordinates.

Color differences between individual products were determined by the ΔL, Δa and Δb values, and ΔE is a summary indicator of the final color difference. The difference in color change ΔE was determined by the formula [12]:

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (1)$$

where: $\Delta L = L_i - L_0$; $\Delta a = a_i - a_0$; $\Delta b = b_i - b_0$; "0" - reference ($L_0 = 94.3$; $a_0 = -0.92$; $b_0 = -0.67$ refers to white color); "i" - sample.

The indicator ΔE served to determine the color change of the cakes crumb, e.g. browning during the storage time, with L_0 , a_0 , b_0 being the coordinates of fresh

cakes, and L_i , a_i , b_i being the coordinates of stored cakes.

The index of whiteness of cakes was calculated (whiteness index) [14]:

$$Wi = 100 - \sqrt{(100 - L)^2 + a^2 + b^2} \quad (2)$$

The indicators were determined according to the CIE Lab system, where: L - illuminance (L = 0 - black, L = 100 - white), +a - red color, -a - green color, +b - yellow color, -b - blue color.

Loss of weight

The weight loss (%) during baking was calculated like loss of moisture as ratio between the weight of the cake samples and the weight of the cake batters as well as during the storage times.

RESULTS AND DISCUSSIONS

It is well known that the gelatinized starch undergoes a partial crystallization, usually named retrogradation, when cooled down to ambient or sub ambient temperatures [15 - 17]. This transition implies hardening of the starch gel and, therefore, is supposed to be responsible for the increased firmness of the stale bread crumb [18]. Bread starch, however, is not exhaustively gelatinized since the water content of doughs is not sufficient to sustain the process thoroughly [19 - 21]: a great deal of the dough water is indeed intimately associated with other hydrophilic components, like proteins, sugars, pentosans, etc. and becomes only partially available to enhance starch gelatinization while baking. The role of water is another important factor. Staling rate is supposed to depend on the moisture content, since a slower staling was observed in breads richer in water [22]; starch retrogradation would, nonetheless, be enhanced on increasing the water content up to 50 % w/w in starch gels, being reduced at larger moisture levels which, however, are largely above those of bread crumb [22 - 24].

Starch thermal transition in stale cake systems analyzed via DSC

The DSC - thermograms of the heated samples on day 1 are characterized by the absence of an endothermic peak, i.e. the thermograms obtained in all evaluated samples (10, 15 and 20 % egg reduction level) and the

control were identical (Fig. 1).

In general, the more the chia gel content increases, the more the retrogradation slows down - Fig. 2 to Fig. 7.

The storage period of the cake products easily reached the predefined time of 12 days, as no retrogradation transition was recorded by the Differential Scanning Calorimetry (DSC) method until the fourth day. From the obtained DSC curves, the temperature interval (ΔT) and the enthalpy (ΔH) of the endothermic phase transition determined the degree of staling of the cake samples during the storage. No endothermic phase transition was

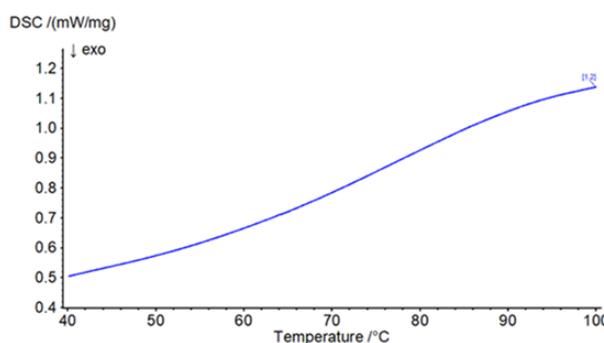


Fig. 1. Typical DSC- thermogram of the control and samples with reduced eggs, on day 1 of the cake's storage.

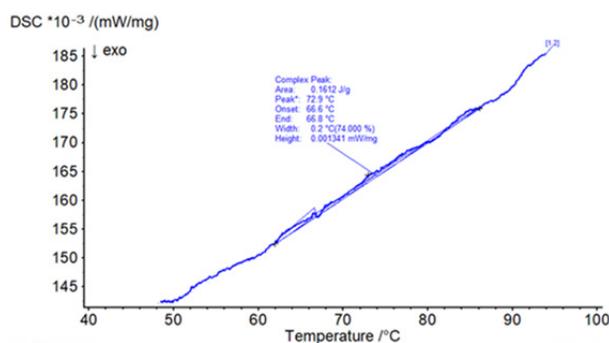


Fig. 2. DSC-thermogram of the sample with 10 % substituted eggs, on day 4 of the storage of the cake samples.

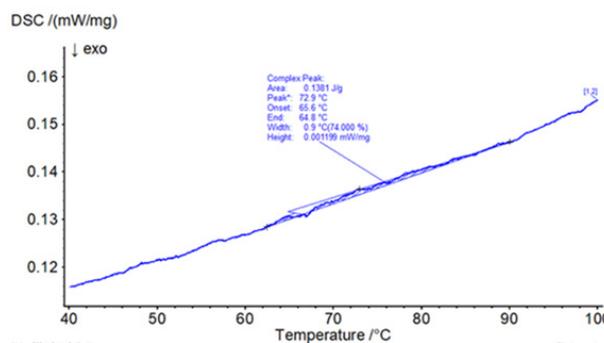


Fig. 3. DSC-thermogram of the sample with 15 % substituted eggs, on day 4 of the storage of the cake samples.

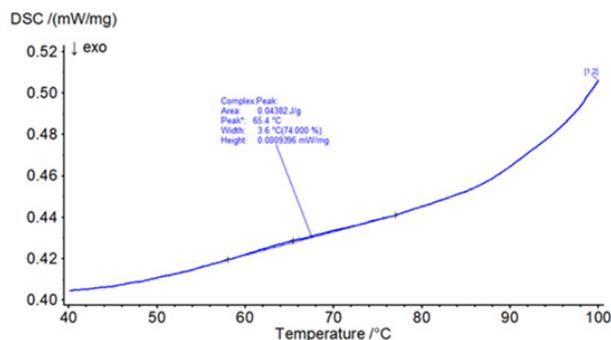


Fig. 4. DSC-thermogram of the sample with 20 % substituted eggs, on day 4 of the storage of the cake samples.

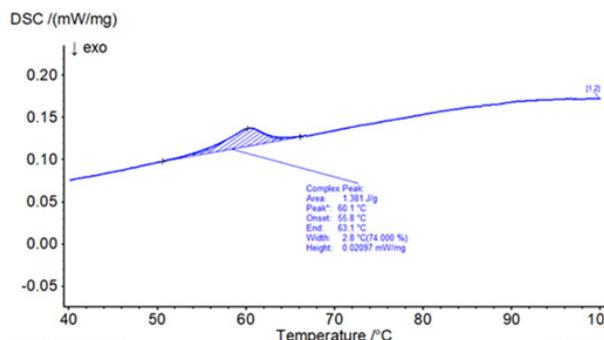


Fig. 5. DSC-thermogram of the sample with 10 % substituted eggs, on day 12 of the storage of the cake samples.

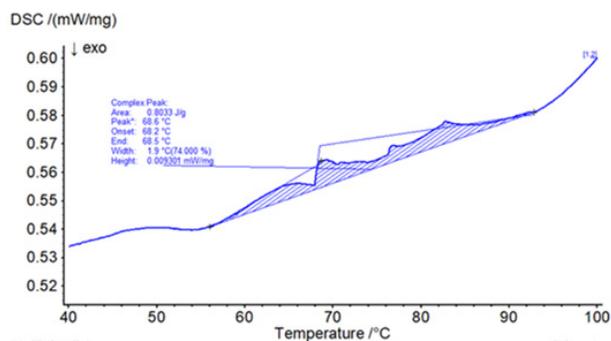


Fig. 6. DSC-thermogram of the sample with 15 % substituted eggs, on day 12 of the storage of the cake samples.

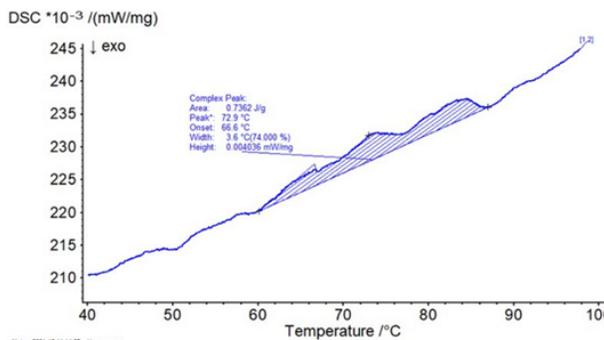


Fig. 7. DSC-thermogram of the sample with 20% substituted eggs, on day 12 of the storage of the cake samples.

recorded during the first three days of storage for all products; therefore, no data are available for this period. The endothermic peak temperature (T_{peak} , °C) on the 4th day of storage for the control was the highest (77.6°C) and the endothermic phase transition (Table 2) showed the widest temperature range compared to the chia gel samples. The lowest endothermic peak temperature on the 4th day of storage was recorded for the sample with 20 % replacement (65.4°C), where there was almost no phase transition (Table 2).

Some researchers found out the energy used during the baking of the final product can be conducted in two directions: either towards the degradation of the complex between the emulsifiers and starch or the breakdown of water-starch bonds [25 - 27]. Depending on the rate of starch amylose deposition, an endothermic phase transition of the crystal structure of the retrograded amylose fraction takes place during the storage. The higher energy levels of these thermal transitions explain the presence of accumulation zones of amylose crystals, the melting of which requires a higher temperature [28]. The preliminary measurements in this work showed the

storage time could be extended to 21 days. The lowest enthalpy values were observed on the day 21st for the 15 and 20 % replacement samples (0.803 and 0.736 J g⁻¹, respectively), and the highest energy level was observed for the control and the 10% substitution (2.313 and 1.381 J g⁻¹, respectively) (Table 2). Therefore, chia gel exhibited the expected retarding effect on starch retrogradation at 15 and 20 % egg replacement level in cake batter (low enthalpy values). In general, with increasing the gel concentration, starch retrogradation slowed down for all stages of the storage (Table 2).

Firmness of the stale cake crumb

The effect of chia gel, added at different concentrations, to replace eggs in the cakes system, on the firmness of the stale cakes crumb, is presented in Fig. 8.

During the storage, the hardness of all cakes apparently increased because of the staling process. This initial trend was notable from day 4th to 6th (statistically insignificant differences, $p \leq 0.05$, data not shown); samples with 10 % and 15 % egg reduction differed from the rest with a less rigid crumb, in contrast to the

Table 2. Values of the thermal transitions.

Samples	Storage, days	T_{min} , °C	T_{peak} , °C	T_{max} , °C	ΔH , J g ⁻¹
Control	4	63.0	76.6	92.5	0.163
	7	66.8	77.6	82.6	0.239
	12	63.0	69.6	76.3	0.390
	21	59.8	78.5	88.0	2.313
10 % eggs replacement	4	62.0	72.9	86.0	0.161
	7	67.2	70.7	86.0	0.227
	12	60.0	67.3	72.5	0.332
	21	50.6	60.1	66.2	1.381
15 % eggs replacement	4	62.5	72.9	90	0.138
	7	67.0	69.8	83.0	0.222
	12	67.0	69.9	86.0	0.327
	21	56.0	68.6	92.8	0.803
20 % eggs replacement	4	58.0	65.4	77.0	0.044
	7	60.0	69.7	84.1	0.206
	12	57.7	72.9	88.9	0.304
	21	60.0	72.9	87.0	0.736

20 % reduction sample, that showed a rapid increase in hardness ($p \leq 0.05$).

The increase in hardness was particularly dramatic after the day 7th, continuing to increase significantly until the end of the storage period. On the day 7th, the highest values were for the sample with 20 % (35.5 N) and 15 % egg reduction (34 N) ($p \leq 0.05$). Samples with egg replacement level 10 % and 15 %, also demonstrated a high final crumb firmness value (42 and 43 N, respectively, Fig. 8).

Some authors investigated [3] the changes occurring in the cake crumb firmness related to perpetually increasing storage time in the context of staling process, possibly due to starch retrogradation [29], in egg-

replacement cakes system.

The use of whey protein isolate alone as an egg substitute was found to delay the hardening of the cakes, this effect being greater after four days of storage at room temperature in case the protein isolate was used in combination with an emulsifier [3]. This behavior is explained by the water affinity of the emulsifiers, as well as their interactions with the starch, which affects the retrogradation and its rate of progressing [3].

Colorimetry

The color of bakery products is a significant factor affecting the consumers' acceptance. The crumb color parameters L, a, b, whiteness index (WI) and ΔE are

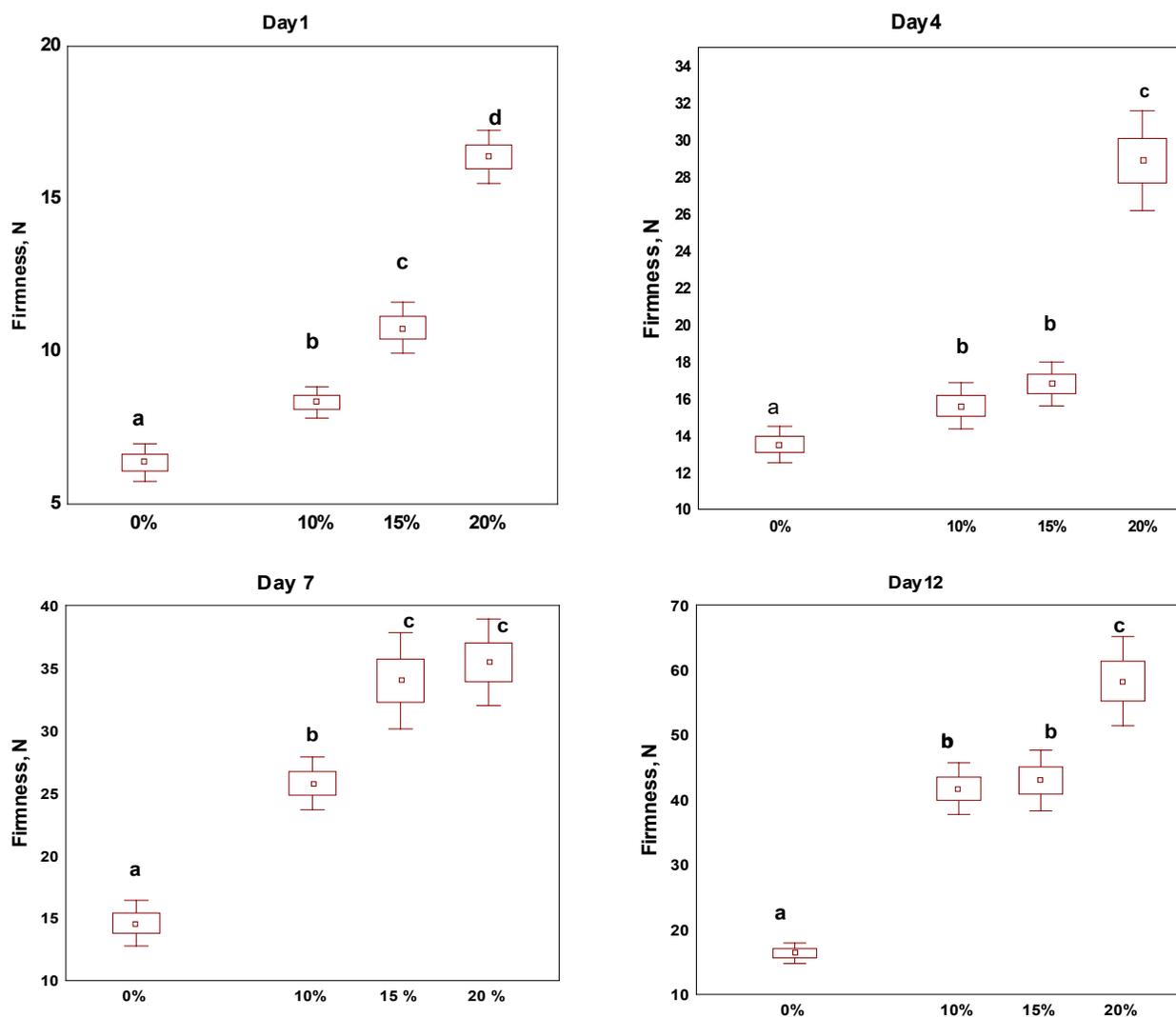


Fig. 8. Effect of chia gel added in different concentrations on the hardness of cakes crumb during storage (*Values marked with different letters are statistically different, $p \leq 0.05$; Mean; Whisker: Mean \pm 0.95 Conf. Interval).

Table 3. Stale cake crumbs color scores in egg replaced, with chia gel, samples.

Chia gel	Storage time	L*	a*	b*	WI	ΔE
0 %	1	67.52 ± 2.49 ^c	8.19 ± 0.51 ^{cd}	27.48±1.41 ^c	56.62±2.70 ^b	11.45±1.68 ^a
	4	68.90 ± 1.97 ^c	7.34 ± 0.33 ^c	27.94±0.99 ^d	57.52±1.97 ^{bc}	40.56±3.92 ^a
	7	70.28 ± 3.47 ^c	6.06 ± 0.51 ^c	25.19±1.85 ^{bc}	60.51±5.23 ^{bc}	37.16±4.28 ^a
	12	70.26 ± 3.38 ^c	5.99 ± 0.42 ^b	25.77±2.18 ^c	60.09±4.14 ^c	37.74±4.13 ^a
10 %	1	49.83 ± 2.47 ^a	7.73 ± 0.56 ^c	23.17±1.56 ^b	44.15±1.73 ^a	19.87±1.65 ^b
	4	49.83 ± 4.68 ^a	7.72±1.02 ^c	24.21±1.94 ^c	43.65±3.74 ^a	53.68±3.62 ^c
	7	50.56 ± 3.83 ^a	7.98±1.02 ^{cd}	24.78±1.74 ^{bc}	44.04±2.95 ^a	52.98±2.85 ^c
	12	51.77 ± 4.49 ^a	8.07±0.95 ^{bc}	24.94±2.33 ^{bc}	44.97±3.26 ^a	52.25±3.14 ^d
15 %	1	59.67 ± 4.48 ^b	6.02±0.68 ^b	22.91±3.19 ^b	53.06±4.61 ^b	16.57±3.80 ^b
	4	62.30 ± 4.03 ^b	3.90±0.43 ^b	21.10±3.35 ^b	56.44±3.78 ^{bc}	41.04±3.75 ^a
	7	63.69 ± 3.17 ^{bc}	4.61±0.53 ^b	22.33±3.69 ^b	57.00±4.08 ^b	40.23±4.14 ^a
	12	62.81 ± 4.52 ^{bc}	4.45±0.51 ^{ab}	21.71±3.17 ^b	56.49±4.67 ^b	40.85±4.67 ^b
20 %	1	46.58 ± 6.37 ^a	4.33±0.35 ^a	16.69±2.17 ^a	43.81±4.84 ^a	28.02±3.13 ^c
	4	54.79 ± 5.58 ^{ab}	2.75±0.15 ^a	16.32±1.78 ^a	51.76±5.00 ^b	45.35±4.89 ^b
	7	58.57 ± 6.65 ^b	2.50±0.10 ^a	16.58±1.75 ^a	55.16±5.84 ^b	41.69±5.68 ^{ab}
	12	57.65 ± 5.60 ^b	3.12±0.15 ^a	16.58±2.23 ^a	54.28±5.06 ^b	42.72±4.96 ^{bc}

*Values marked with different letters in columns are statistically significant $p \leq 0.05$

presented in Table 3.

Color parameters indicated the increase of the concentration of chia gel at egg replacement level to 20 % resulted in darkening of the crumb (L) ($p < 0.05$) and fading of the yellow (b) and green (a) shade of the samples (Table 3). No significant difference ($p > 0.05$) was found in terms of WI between the control and the 15 % chia sample (Table 3). The crumb color parameters L, a, b was similar for the products with 10 % and 20 % substitution for L, the control and 10 % replacement for a, and 10 % and 15 % samples for +b, respectively. Increasing the concentration of chia resulted in a significant difference ($p < 0.05$) in the ΔE values (Table 3). Therefore, the crumb of the chia gel products was characterized by the darker color than the control, with no chia added.

Weight loss

Weight loss is due to the removal of moisture and other volatiles during baking and storage of bakery products [30]. It is noted that very high and very low values generally result in extremely dry and extremely moist products [30]. Therefore, both extreme situations would adversely affect the quality of the product [29]. The importance of the “weight loss” indicator for the

freshness of confectionery products makes it a key technological parameter in the production of egg-replaced cakes [31].

The effect of chia gel added at different levels to replace eggs on cakes samples weight loss during storage is shown in Fig. 9. As it can be seen from the figure, weight loss increases with the increase of storage time.

Weight losses (%) in all investigated products stored for one day are comparable and statistically insignificant ($p \leq 0.05$, Fig. 9). The weight loss (%) after seven days until the end of the storage time for the sample with 20 % replaced eggs was the lowest, and for the rest of the gel-containing samples it was comparable to the control ($p \leq 0.05$).

In general, as replacement levels increase, weight loss (%) rises, with this trend being more pronounced during the storage (20 %). This effect can be explained by the reduced protein content of the products as it is cited in the literature. Enrichment of muffins with different types of animal and vegetable proteins as egg substitutes did not result in a significant difference in weight loss values [32]. Another study [33] reported that wheat flours with higher protein content (i.e., bread flours) gave the lowest weight loss values in an eggless cake, whereas, in contrast, the highest losses were found when using cake flour (lowest protein content) [33].

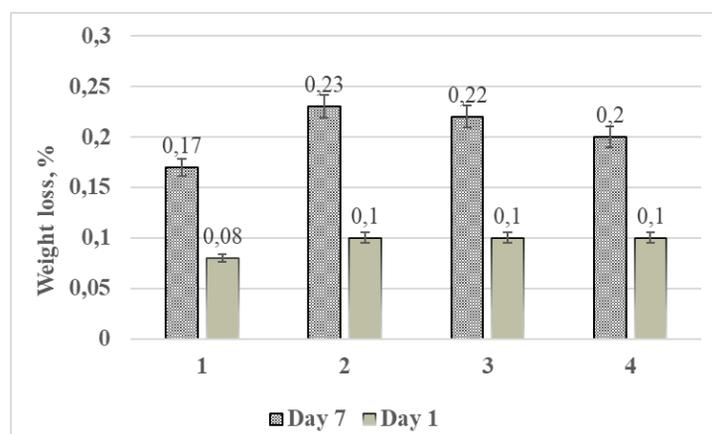


Fig. 9. Effect of chia gel added at different egg replacement levels on weight loss of cake samples during storage: 20 % replacement level (1); control (2); 10 % replacement level (3); 15 % replacement level (4).

CONCLUSIONS

It is clear the amount of retrograded starch in stale baking goods is just one more factor in the multidimension cake system associated with overall mechanical properties of the stale crumb texture and product aging along with other hydrophilic components (proteins, sugars, pentosans). Differential Scanning Calorimetry (DSC) application proved to be a reliable approach to investigate the influence of polysaccharide - chia gel, added in different concentrations, on the staling process in cake systems. The DSC-thermograms of the samples with substituted eggs demonstrated the starch retrogradation was significantly delayed due to the increased moisture content of the starch gel, which also meant delayed staling beyond the preset period of 12 days. The increase in the hardness of the products during storage depended on the concentration of the incorporated chia gel, water loss and starch retrogradation run together with crumb aging. The highest values were observed for products with 20 % egg reduction. During the storage, lower weight loss values were observed again for the 20 % replacement level compared to the rest of the samples and the control. Using chia gel in higher concentrations resulted in less weight loss and as mentioned above it could rely the starch retrogradation delay.

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