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Physical Characteristic of Heat Resistant Chocolate Formulated with Konjac Glucomannan and Xanthan Gum-Based Hydrogel at Various Fat Content during Period of Crystal Growth (Maturation)

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ABSTRACT

Indirect addition of water into chocolate may form secondary sugar networks. This condition creates an increased melting temperature of chocolate. The purpose of this study was to increase the melting point of premium couverture chocolate without addition of fat/oil from other sources. Chocolate was formulated with coconut/palm sugar as sweetener at various fat levels (32%, 34%, and 36%). Aside from this, Konjac glucomannan and Xanthan gum-based hydrogel with a concentration of 3%, 5%, and 7% was added. Characterization of chocolate quality parameters with the addition of konjac glucomannan-based hydrogel (CKG) and xanthan gum-based hydrogel (CXG) was carried out. Moisture content, melting point, hardness, particle size and brown color analyses were carried out at intervals of 1, 5, 9 days of maturation (crystal growth period). The results showed that the addition of hydrogel influenced the melting point and hardness. As the period of crystal growth (maturation) increased, the melting point and hardness of the chocolate also increased.

1. INTRODUCTION

As a country with a geographical location around the equator, Indonesia has various plantation products. Cacao is one of the leading commodities with a high level of demand. According to BPS (2021) Indonesia's cacao production reached 713,400 tons in 2020. One of the main and most demanded cacao products is chocolate bar. The distinctive taste and aroma of chocolate are the main reason why chocolate products are favored by various groups of people.

However, although Indonesia produces a lot of cacao bean, the variation of cacao-based products are still very limited. According to Ogunwulu & Jayeola (2006), in tropical countries such as Indonesia, most chocolate products are imported. This is due to the low level of chocolate production. In addition, environmental factors also greatly affect the characteristics of chocolate. Chocolate that melts easily at a temperature range of

32-34^{o}C (Beckett, 2008) becomes an obstacle in the production and distribution in the areas with a fairly high temperature. Several alternatives for small-scale chocolate production have the potential to maximize the quality of chocolate in tropical countries (Saputro *et al*., 2017a; Saputro *et al*., 2019).

There have been many innovations in chocolate processing so that it does not melt easily. One of them by producing heat resistant chocolate. Several alternatives in the manufacture of heat-resistant chocolate include replacing cocoa butter with fat that has a higher melting point, increasing the viscosity of liquid fat and increasing the formation of sugar networks in chocolate (Killian & Coupland, 2012; Stortz & Marangoni, 2011). In principle, the presence of water in the chocolate formulation forms a secondary sugar network. Addition of water can be done directly or indirectly (Stortz & Marangoni, 2011). Lillah *et al*. (2017) and Jin *et al*. (2021) formulated chocolate using cocoa butter substitute to increase the heat stability of chocolate compounds. The use of fat emulsions in chocolate formulations has previously been done by Norton & Fryer (2012) and has the potential to produce chocolate with stable physical and microbial characteristics. In another study, Francis & Chidambaram (2019) have been dispersed hydrogels from a mixture of sodium alginate and pectin to produce low-fat and heat-resistant chocolate, while Skelhon *et al*. (2013) developed an agar-based hydrogel dispersion method on cocoa butter and chocolate to reduce fat content. Another innovation was carried out by adding chitosan hydrogel to canola oil as a fat substitute for chocolate (Soto *et al*., 2020).

The addition of chocolate fat substitutes in the form of hydrogels in chocolate formulations is interesting to be developed in the chocolate production process (Puscas *et al*., 2020). The hydrogel is thought to be able to maintain the mechanical stability of heat-resistant chocolate (Selvasekaran & Chidambaram, 2021). Meanwhile Saputro *et al*. (2017) formulated chocolate using palm sugar as an alternative natural sweetener. Nafingah *et al*. (2019) also formulated heat-resistant milk chocolate by using a combination of sucrose and palm sugar as sweeteners. Having more mineral, vitamin, and a higher water content (Saputro *et al*., 2019; 2020), palm sugar has the potential to form a strong secondary sugar network in chocolate (Beckett, 2017; Stortz & Marangoni, 2011).

The development of healthy heat-resistant chocolate in Indonesia has urgency to be developed considering the large potential of cacao to be processed into finished products. This research focused on the development of heat-resistant chocolate which was formulated using palm sugar and the addition of Xanthan Gum and Konjac glucomannan- based hydrogels. Xanthan Gum and Konjac glucomannan are known to have high fiber content and acts as prebiotics. The combination of chocolate formulation with the addition of prebiotic-rich hydrogel and palm sugar which is high in vitamins and minerals has the potential to produce healthy and heat-resistant chocolate. Physical analysis of chocolate in term of moisture content, melting point, particle size, hardness, color, and storage time were carried out.

2. MATERIALS AND METHODS

2.1. Raw Material

The raw materials in this study were obtained from several different places. Cocoa paste and cocoa butter were obtained from the Coffee and Cocoa Research Institute (ICCRI), Jember. Palm sugar was produced by Muria Food Lion Superindo Jakarta. Sugar (sucrose) was produced by PT. Sugar Group. Xanthan Gum and Konjac Glucomannan were obtained from Subur Kimia Jaya, Bandung.

2.2. Sample Preparation

2.2.1. Hydrogel Preparation

Konjac Glucomannan and Xanthan Gum-based hydrogels with proportions of 3%, 5% and 7% were used based on their gelling ability and the acceptable daily intake (ADI) of the two ingredients (Mortensen *et al*., 2017a; 2017b). Distilled water was prepared and put into a beaker glass to make a hydrogel. Xanthan Gum and Konjac glucomannan were then slowly added to the beaker. The mixing process was carried out using a hand mixer at a speed of 6500 rpm for 5 minutes until the mixture formed a gel. This mixing treatment was chosen with the aim of forming a gel in a short time using a high-speed mixer. Afterwards, the hydrogel was stored at 10°C in a thermostatic cabinet to be used in chocolate formulations.

2.2.2. Preparation of Chocolate

Chocolate was made using a two-stage mixing process. Stage 1 was done by making dark chocolate paste with a fat content of 32%. Dark chocolate paste was made from 37% of cocoa powder, 13% of cocoa mass, 15% of cocoa butter and 35% of sugar. The sweetener used was palm sugar. All ingredients were mixed and ground using Wonder Premier Melanger as an alternative tool for small scale chocolate processing with a capacity of 1.5 kg. The process of mixing, reducing particle size and conching was done for 10 hours. The conching process requires a relatively high temperature, this condition was achieved by heating the melanger using a heat gun for 5 minutes every 30 minutes. The second stage of mixing process was done to add the hydrogel into dark chocolate. Three proportions of konjac glucomannan and Xanthan gum-based hydrogel, namely 3%, 5% and 7% and three proportions of fat content, namely 32%, 34% and 36% were used as the treatments. The proportion of fat content was selected based on the composition of the minimum amount of cocoa fat (18%) and cocoa solids without fat (14%) in dark chocolate (Devos *et al*., 2021). The refernce chocolate in this study was chocolate with a fat content of 32% without addition of hydrogel, formulated with white sugar/sucrose (CGP) and palm sugar (CGS).

The tempering process was carried out manually according to Kurniasari *et al*. (2019) in a square cake pan. The chocolate was heated at 50 °C for 9 minutes to melt the fat crystals. Then, the chocolate was gradually cooled in a container filled with water and ice gel. The melted chocolate was maintained at 27 °C for 9 minutes accompanied by a stirring process. The final stage of tempering was to increase the temperature of the chocolate until 32 °C (in the oven). The tempered chocolate was molded and vibrated on a vibrating table for 5 minutes to remove air bubbles. Afterwards, the chocolate was cooled in the thermostatic cabinet until the chocolate completely solidified and was able to be removed from the mold. Chocolate was then stored for further analyses. The chocolate research design is shown in Table 1.

2.3. Analysis Method

2.3.1. Water Content

Water content analysis was carried out using the thermogravimetric method. The sample was placed in a cup and weighed as the initial mass. The samples were then placed in an oven at 105 °C for 24 hours. After being baked, the sample was placed in a desiccator for ± 15 minutes and then weighed as the final mass.

2.3.2. Color

The brown color value was measured using a Konica Minolta CR-400 CIELab chromameter. The color parameters included were lightness (L*), redness (a*) and yellowness (b*).

Table 1. Research design of hydrogel chocolate

No	Treatments		Hydrocolloid Type	
	Hydrocolloid Level	Fat level	Xanthan Gum	Konjac glucomannan
1	3%	32%	XG3F32	KG3F32
2		34%	XG3F34	KG3F34
3		36%	XG3F36	KG3F36
4	5%	32%	XG5F32	KG5F32
5		34%	XG5F34	KG5F34
6		36%	XG5F36	KG5F36
7	7%	32%	XG7F32	KG7F32
8		34%	XG7F34	KG7F34
9		36%	XG7F36	KG7F36

2.3.3. Hardness

The value of chocolate hardness was measured using a Brookfield Texture Analyzer with a TA39 probe and a test speed of 0.5 mm/s with a target depth of 3 mm. The type of test used was a compression test. The Texture Analyzer was connected to a computer equipped with CT3TM software as a data display for hardness values. The data set displayed was the load in grams. The resulting data is then converted into compression strength values in N/mm^2. The formulation of the hardness value is shown in Equation 1.

$$\sigma = \frac{Load\ x\ a}{\pi r^2} \quad (1)$$

where σ is compression strength (N/mm^2), a is gravity acceleration (mm/s^2), and r is probe radius (mm).

2.3.4. Melting Point

The melting point value of chocolate was measured manually using a water bath filled with water at a temperature of 27 °C. A 250 ml beaker glass was placed in a water bath and filled with water until the water level in the beaker and water bath were the same. A sample of chocolate with dimensions of 1 cm x 1 cm x 1 cm was placed on a concave plastic spoon and then immersed in the water in the beaker. Water temperature was measured using a thermocouple.

2.3.5. Particle Size

As much as 0.5 g of chocolate that has been melted in the oven for 30 min was dissolved in 10 ml of palm oil. The mixture was shaken horizontally for 5 min. After the mixture was evenly distributed, the sample was heated in an oven at 55 °C for one hour. The samples were then tested using an Olympus CX23 optical microscope and analyzed using Optilab software equipped with Image raster 3.0.

2.3.6. Data Analysis

Data analysis in the form of ANOVA test was performed using IBM SPSS 25.0 software. Before the one-way ANOVA test, homogeneity test was carried out using Levene's test. Post-hoc test was performed using the Tukey test. Three-way ANOVA test was conducted to determine the significant effect of fat content, hydrocolloid content and period of crystal formation (maturation) on chocolate quality. PCA analysis was performed using IBM SPSS 25.0 software to determine the relationship among parameters. The PCA values displayed are the PCA score plot and the PCA loading plot.

3. RESULTS AND DISCUSSION

3.1. The Relationship Among Maturation Period, Fat Content and Hydrogel on Chocolate Quality Parameters

The relationship among treatments on chocolate quality (Table 2) was tested using multivariate analysis. In CKG chocolate, the maturation period, fat content and hydrocolloid content significantly affected the melting point, particle size and hardness parameters of chocolate. The interaction between maturation period and fat content, maturation period and hydrogel content and interaction between fat content and hydrogel content significantly affected particle size and yellowness. The lightness value was only influenced by the interaction between the maturation time and the hydrocolloid content.

Table 2. The relationship between the duration of the crystal growth period (maturation), fat content and hydrogel with chocolate quality parameters

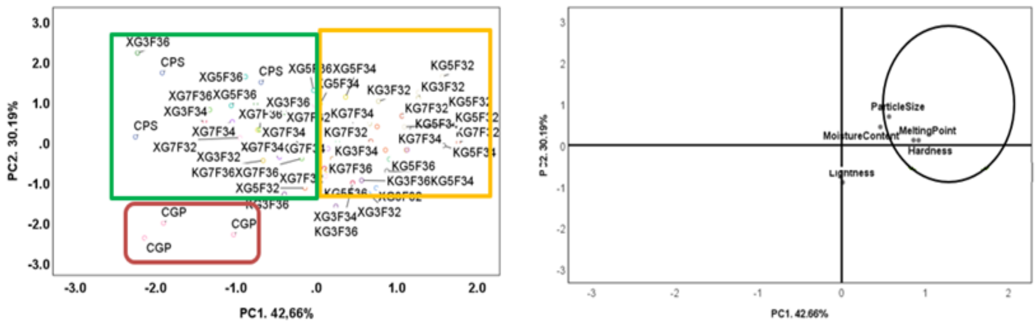
Konjac Glucomannan Chocolate								
Treatment Variables	**Melting Point**	**Particle Size**	**Hardness**	**L***	**a***	**b***	**hue**	**Chroma**
Day	*	*	*	-	-	-	-	-
Fat level	*	*	*	-	-	*	-	-
Hydrogel	*	*	*	-	-	*	-	-
Day*Fat	-	*	-	-	-	*	-	-
Day*Hydrogel	-	*	-	*	-	*	-	-
Fat*Hydrogel	-	*	-	-	-	*	-	-
Day*fat*hydrogel	-	-	-	-	-	-	-	-

Xanthan Gum Chocolate								
Treatment Variables	**Melting Point**	**Particle Size**	**Hardness**	**L***	**a***	**b***	**hue**	**Chroma**
Day	*	-	*	-		-	-	-
Fat level	*	*	*	-		-	-	*
Hydrogel	*	*	*	-		*	*	*
Day*Fat	*	-	-	-		-	-	-
Day*Hydrogel	*	-	*	-		*	-	*
Fat*Hydrogel	-	*	*	-		*	-	*
Day*fat*hydrogel	-	-	*	-		-	-	*

[*]Significantly different at $p<0.05$

In CXG chocolate, the melting point value was influenced by the maturation period, fat content, hydrocolloid content, the interaction between the maturation period and fat content and the interaction between the maturation period and hydrocolloid content. Particle size was only affected by fat content, hydrogel content and the interaction between the two. The maturation period had no effect on the particle size of CXG chocolate. Chocolate hardness was affected by all treatment variables except the interaction between maturation period and fat content. Color parameters were yellowness, hue and chroma which were influenced by hydrocolloid content. In more detail, the yellowness value was also influenced by the interaction between the maturation period and the hydrogel content and the interaction between the fat content and the hydrocolloid content. While the chroma value was not affected by the maturation period and the interaction between the maturation period and fat content.

Based on the above-mentioned explanation, fat content, hydrocolloid content and period of crystal growth (maturation) were the variables that mostly influenced the quality of chocolate in term of melting point, particle size and hardness. While the effect of the treatment variable on several color parameters was not significant. This could be probably addressed to the high-water content which caused the surface of the chocolate to be not smooth, making the color become uneven (Briones *et al*., 2006).



(a) (b)

Figure 1. PCA Analysis of Chocolate Hydrogel (a) PCA score plot and (b) PCA loading plot

The PCA analysis in Figure 1 provides an overview of the relationship between chocolate formulated using palm sugar and the addition of a hydrogel. Based on the PCA score plot in general, it can be seen that the chocolate samples formulated with the addition of hydrogels tended to be close to each other regardless of the hydrocolloid content and fat content. Chocolate with the addition of Xanthan Gum-based hydrogel tended to cluster in the negative PC1, while the chocolate with the addition of Konjac Glucomannan-based hydrogel was dominant in the positive PC1. CPS reference chocolate formed a group with CXG. The CGP reference chocolate grouped separately on PC1 and PC2 were negative. The tendency of CPS reference chocolate to group with CXG showed that the addition of Xanthan Gum-based hydrogel to chocolate formulated using palm sugar at the proportion of fat content and certain hydrocolloid content did not show a clear difference.

The PCA data (Figure 1) shows that the first two factors explain the variance of more than 72% with PC1: 42.63% and PC2: 30.19%. In general, the effect of using different types of sugar can be observed based on PC2. CPS reference chocolate showed a positive PC2 value while CGP had a negative PC2 value (Figure 1a). This shows that the use of two different types of sugar affected the chocolate parameters. CPS reference chocolate had a higher moisture content, hardness and melting point than CGP (Figure 1b). Particle size and water content with a larger loading value were more dominantly influenced by the use of the type of sugar. This was supported by the research of Saputro *et al*. (2019) which explains that the water content of palm sugar is higher than sugar (sucrose). Meanwhile, the negative lightness value on PC2 indicated that CGP had a higher brightness value than CPS chocolate (Figure 1b).

The chocolate hydrogel can be observed through PC1 and PC2. Two different types of hydrocolloids (Konjac glucomannan and Xanthan Gum) had a tendency based on the trend in PC 1. CKG chocolate had a tendency in positive PC1 values while CXG had a tendency in negative PC1. From these, it can be concluded that the water content, particle size, hardness and melting point of CKG was greater. Particle size and moisture content were also more dominant than the melting point value and hardness was indicated by a larger loading value (Figure 1b). Based on Figure 1a, it can be seen that CKG and CXG chocolates showed a predominance of positive PC2 values. This shows that chocolate formulated with palm sugar and added with hydrogel has a higher moisture content and particle size and lower lightness value (Figure 1b).

Based on the value of the loading plot (Figure 1b), the parameters of melting point, hardness, moisture content and particle size of chocolate were close to each other, the value of water content and particle size was more dominant with a larger loading value. This showed that there was a correlation between parameters that influence each other. An increase in the particle size, it indicated that the value of water content, melting point and hardness also increased. According to Afoakwa *et al.* (2008a) the value of chocolate hardness increases if the particle size gets smaller, whereas in this study the opposite value was produced. This phenomenon may occur because the addition of hydrogel in the chocolate formulation forms sticky patches so the particle size becomes bigger. Afoakwa *et al.* (2008b) added that the sugar content in the formulation also had an effect in increasing the particle size.

3.2. Color

Chocolate color is influenced by interactions among chocolate particles (Afoakwa *et al.*, 2008b; Saputro *et al.*, 2017), water content and the presence of amorphous sugars (Afoakwa *et al.*, 2008b). Chocolate with a small particle size has the ability to reflect light because the surface of the particles is larger, resulting in a brighter color (Afoakwa *et al.*, 2008a). The fluctuating value of color parameters is probably due to the starting material and high-water content that affects the brown color. Chocolate sweetened with palm sugar has a lower brightness value than chocolate sweetened with white sugar (sucrose). Palm sugar with a higher moisture content has the potential to cause blooming during the chocolate maturation process which results in an uneven color appearance (Fadilah, 2021; Fadilah *et al.*, 2022). Unstable brown crystals form more stable crystals and produce a white surface color (Buscato *et al.*, 2018).

3.3. Water Content

The water content in chocolate has an influence on other parameters such as flow properties and hardness (Afoakwa, 2016). In addition, chocolate with a high-water content has the potential to form particles that clump together (Afoakwa, 2016). According to Beckett (2017) the appropriate moisture content to produce chocolate with good physical characteristics is less than 2%. Based on the results of the study, in each treatment the water content value produced had a water content above 2% (Figure 2). The use of palm sugar and the addition of hydrogel in the chocolate formulation had a great effect on increasing the water content of chocolate.

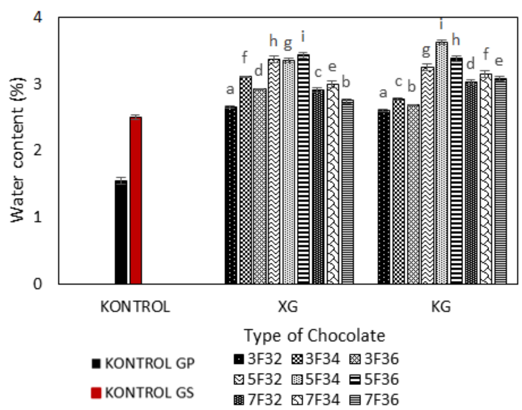


Figure 2. Chocolate moisture content

3.4. Melting Point

Cocoa fat in a stable condition melt at a temperature of around 32-34°C (Afoakwa *et al*., 2007; Beckett, 2008). The melting point of chocolate is influenced by several factors such as the initial moisture content of the material, fat content, particle size and the success of the tempering process (Saputro *et al*., 2017; Nafingah *et al*., 2019). A good tempering process forms stable V crystals and produce a glossy appearance, have a snap, and maintain shelf life (Afoakwa *et al*., 2007; Afoakwa *et al*., 2008b; Beckett, 2008).

The use of palm sugar and the addition of hydrogels in the chocolate formulation affected the formation of secondary sugar networks due to the high-water content value. This network plays a role in increasing the melting point of chocolate (Stortz & Marangoni, 2011). In addition, hydrogels also have the ability to prevent fat migration in chocolate which results in an increase in the melting point of chocolate (Suri & Basu, 2021). In this study, the melting point of chocolate has a high enough value above 34°C (Figure 3). The melting point value of CKG tended to be higher than CXG, this might due to the absorption ability and water holding capacity of Konjac glucomannan of 100 grams of water per gram of hydrocolloid (Bangun, 2021; Koroskenyi & McCarthy, 2001). This value is greater than Xanthan Gum with a capacity of 19.2 grams of water per 100 grams of hydrocolloids (Karaman *et al*., 2014). In addition, the effect of storage (maturation) of chocolate had a tendency to increase the melting point of chocolate. According to Saputro *et al*. (2017) chocolate storage has an effect on the formation of more stable beta V crystals.

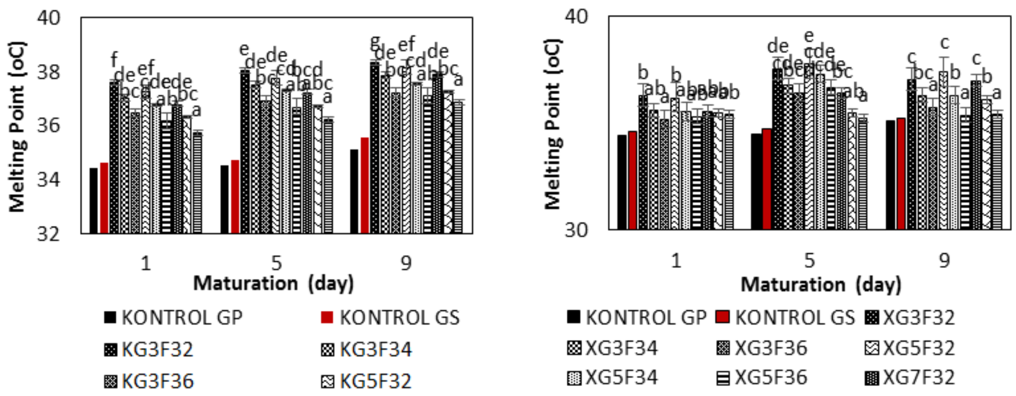


Figure 3. Melting point of chocolate during the maturation period

3.5. Hardness

Chocolate hardness is influenced by fat content, particle size, tempering quality (Afoakwa *et al*., 2008b) and moisture content (Beckett, 2008). The water content is directly proportional to the increase in the hardness of the chocolate, while the fat content has an effect on the decrease in the hardness of the chocolate. Both have a great influence on the final value of chocolate hardness. Palm sugar has a higher water content than sucrose and hydrogel has a high water absorption ability increasing the moisture content of chocolate.

In this study, the value of hardness, fat content, type of sugar and hydrocolloid content affected the hardness of chocolate. In Figure 4, it can be seen that the use of palm sugar increased the hardness value of chocolate (Saputro *et al*., 2017). The high water content in palm sugar compared to white sugar (sucrose) affected the hardness of chocolate. In addition, chocolates with the addition of hydrogel had a higher hardness value than the CGP and CPS reference chocolate (without the addition of hydrogel). Chocolate with lower fat content exhibited a higher hardness value. The length of time for the formation of crystals (maturation) also affected the increase in the hardness of chocolate. During the maturation process, there is an improvement in the formation of chocolate crystals that are not yet stable to become more stable crystals.

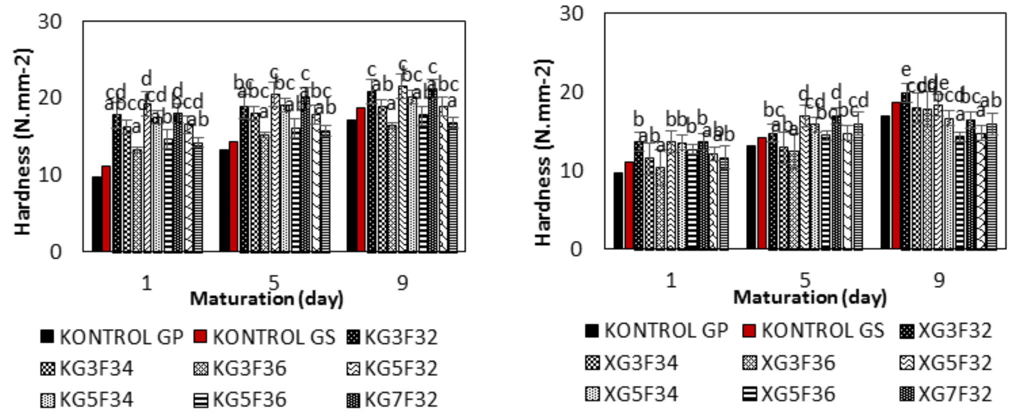


Figure 4. Hardness of chocolate during the maturation period

3.6. Particle Size

Chocolate particle size is influenced by the materials used and the processing time (Prawira & Barringer, 2009). Chocolate formulated using palm sugar has a larger particle size and has a higher level of agglomeration due to the formation of an amorphous phase of sugar containing fructose and glucose (Saputro *et al.*, 2017a; 2017b). Particle size has an inverse correlation with hardness values (Afoakwa *et al.*, 2008a). A good particle size according to Afoakwa (2016) is less than 30µm.

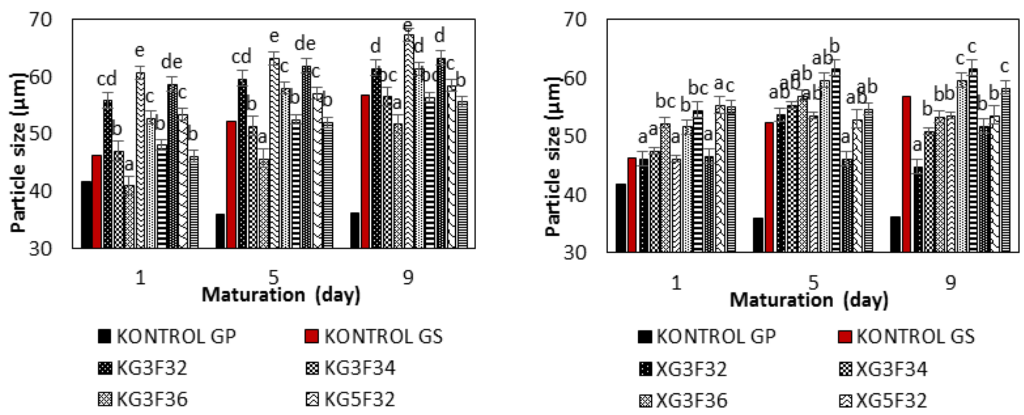


Figure 5. Chocolate particle size during the maturation period

The particle size of the GP reference chocolate was smaller because the moisture content of the chocolate formulated with white sugar (sucrose) tended to be lower (Figure 5). While the particle size of the chocolate sweetened with palm sugar had a large particle size due to the high-water content. The particle size of CXG and CKG chocolate was greater than 30µm. The high-water content results in the formation of agglomerations in the chocolate particles. CXG chocolate exhibited particle size which was inversely correlated with hardness values according to the statement of Afoakwa *et al*. (2008a). Meanwhile, CKG chocolate had particle sizes that tended to be high and had a positive correlation with the hardness value.

4. CONCLUSION

The addition of hydrogel, the period of crystal growth (maturation) and the use of different types of sweeteners affected the quality of chocolate. The high value of chocolate moisture content affected several other parameters such as hardness, particle size and melting point of chocolate. The use of different fat content affected the quality of chocolate such as hardness and melting point of chocolate. Chocolates formulated with palm sugar and hydrogel have the potential to be developed into heat -resistant chocolate products in tropical countries such as Indonesia.

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