Development and Characterisation of Thermo-Resistant Milk Chocolate Containing Cocoa Butter Emulsion

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Abstract: Aimed at the production of thermo-resistant milk chocolate, a method of using cocoa butter emulsion was applied and the product was assessed for its heat stability, melting properties, texture and sensorial characteristics. Milk chocolate was produced by conventional lab scale method and different percentage (0-4%) of the formulated cocoa butter emulsion (water:cocoa butter:lecithin) was mixed into tempered milk chocolate. Results indicate improvement in heat stability of chocolate. Even though it did not significantly increase its melting point, the chocolate structure was strengthened and was able to hold the molten fat within its network. This holding capacity increased with the increased of emulsion content. At 3-4% cocoa butter emulsion content, the chocolate was able to retain its shape up to 40°C. Addition of cocoa butter emulsion at 1 to 3% did not significantly change the hardness of chocolate but at 4% emulsion content the chocolate became significantly less hard and slightly crumbles on cutting. Higher emulsion content also lowered the glossiness of chocolate, reduced its pleasant odor and smoothness. The acceptability score was however did not differ significantly between chocolates containing cocoa butter emulsion (1-4%) and chocolate without the emulsion.

Key words: Chocolate ∙ Cocoa Butter ∙ Emulsion ∙ Thermo-Resistant

INTRODUCTION

Cocoa butter in chocolate starts to soften at about 28°C with consequent loss of strength as temperatures rises and generally melts at 33.8°C when solid cocoa butter becomes liquid [1]. Improvement of heat stability of chocolate will allow wider distribution and consumption of chocolate especially in tropical countries. The introduction of water into chocolate mass was one of the earliest and popular method in developing heat stable chocolate. A stable network structure formed when bonds between solid particles created through uniform reaction with moisture in chocolate mass. This non-fat skeleton resists the deformation of the mass even when fat subsequently melt at high temperatures. However, homogenizing of water into a chocolate mass causes rapid thickening of the chocolate at temperature where it is normally still liquid. This behavior is caused by the formation of layers of syrup on the surface of sugar particles in chocolate, which increases the friction between them [2]. Since it is no longer liquid it is impossible to use the mass for depositing into moulds or enrobing. The composition must be ground and the obtained powder is pressed into shape by compression molding [3]. Gidey and Dove [4] described the use of 2-10% cocoa butter emulsion (lecithin:water:cocoa butter) dispersed into chocolate mass to avoid rapid thickening. Upon mixing with the liquid chocolate mass, the emulsion particles melted and the water phase released and formed rigid network with other non-fat chocolate particles. A homogeneous distribution of emulsion before the particles were melted was crucial because a premature release of the water would cause a sudden viscosity increase. The heat resistant structure was however only developed after a storage period of 1 or 2 weeks at temperatures between 15°C and 28°C. Other almost similar techniques of using water in oil emulsions to produce heat stable chocolate are as described by Kealey and
Quan [5], Takemori et al. [6] and Ducret et al. [7]. The heat resistance in chocolate for all these methods was however, reported to develop after more than 24 hour storage. Simburger [8] has described an instantaneous heat resistance development for chocolate mass containing water in oil emulsion or increased water content by applying microwave heat treatment on water containing chocolate mass after it has been shaped but before, during and/or after cooling. It was claimed that the product obtained by this process could be subjected to temperature of 40 to 50°C without losing their form. The use of cocoa butter emulsion also reduces calories in chocolate [9, 10, 11]. The objective of the current study is to apply different percentage (0-4%) of the formulated cocoa butter emulsion namely water(49%):cocoa butter (49%):lecithin(2%) into milk chocolate and to determine the respective heat stability, melting properties, texture and sensorial characteristics of finished product.

MATERIALS AND METHODS

Materials: Cocoa butter emulsion and milk chocolate were made using commercially sourced ingredients. Cocoa liquor (natural Malaysian cocoa liquor) and cocoa butter (deodorized, pure prime press of Malaysian origin) were purchased from Barry Callebaut Malaysia Sdn. Bhd., Klang, Selangor. Whole milk powder (New Zealand) was purchased from Promac Enterprises (Malaysia) Sdn. Bhd., Klang, Selangor. Lecithin (L-phosphatidylcholine from soya bean) was purchased from Akashi Biosystems (Malaysia) Sdn. Bhd. Shah Alam. Sugar and vanillin powder was purchased from local supermarket.

Production of Cocoa Butter Emulsion: Cocoa butter was melted at 55°C and homogenized with equal volume of water and 2% lecithin was added to the mixture. The mixture was thoroughly homogenized for 3 minute at 9500rpm using a high shear mixer (IKA, Germany). The emulsion sample was then sealed and refrigerated. The solid state emulsion was then finely ground prior to the molding of chocolate.

Production of Milk Chocolate: The milk chocolate was formulated to contain a total fat content of 33.5% inclusive of cocoa butter and milk fat, 6% fat free cocoa solid, 17% fat free milk solid and 43% sugar. Mixing of ingredients with small doses of cocoa butter fraction was carried out using a laboratory scale mortar and pestle mill (Pascal, UK) at 45°C. Subsequently the mixture was refined using a three roll refiner (Pascal, UK) to attain chocolate particle of 40µm in size. The size was measured using portable digital micro-screw meter (Mitutoyo, Japan). The refined chocolate mass was then transferred back into mortar and pestle mill and the remaining fraction of cocoa butter was added to the mixture and conched for 6 hour at 55°C. Lecithin was added and the conching continued for another two hours. Finished chocolate was tempered manually on marble slab at 28 to 29°C. The tempered chocolate was molded and cooled in a chiller at 16°C for 30 minutes. This chocolate, free of cocoa butter emulsion, was used as control sample. Chocolates, each containing 1, 2, 3 and 4% cocoa butter emulsion respectively, were prepared by mixing the cocoa butter emulsion into the tempered liquid chocolate. The chocolates were molded and cooled. The chocolates were removed from mould, packed in airtight plastic container and stored at 24°C.

Moisture Content Analyses: The moisture content of cocoa butter emulsion and chocolate samples was determined using HR73 Halogen moisture analyzer (Metler Toledo, Malaysia). Chocolate and emulsion samples were separately cut into small pieces and then each milled using a dry kitchen blender into fine solid particles. 5mg of the respective sample was placed on the pan and heated to 105°C until constant weight was achieved. Measurement was determined in triplicate and the mean value calculated.

Fat Content Analyses: The fat content of chocolate was determined using the Soxhlet procedure [12] where 3-4g melted chocolate sample was subjected to acid digestion and a 4 hour Soxhlet reflux in petroleum ether of boiling range 40-60°C. Analysis was carried out in triplicate.

Heat Resistance Test: The heat resistance of chocolate was tested by exposing unwrapped chocolate samples to a series of temperatures starting from 28°C, with 1°C temperature increment to a maximum of 40°C. Chocolate samples were placed on a tray and stored in an incubator (IFE 550, Memmert, Germany) for 2 hours at each temperature before sensory assessment was made by experienced panelist on surface appearance, stickiness, hardness and shape keeping property. Photograph of tested sample was taken using a digital camera (Panasonic DMC-FS7).

Melting Profile: The melting profile of chocolate and fat
samples was measured using a scanning calorimeter DSC-7 (Perkin Elmer, Norwalk, Connecticut, USA) based on method by Md. Ali and Dimick [13]. The sample was melted in the oven at 50°C. A sample weighing 3-5mg was hermetically sealed in a DSC aluminum pan. The sample was heated to 60°C for 30 min and then cooled at 0°C for 90 min. It was then transferred into an incubator and maintained at 26°C for 40 hours for stabilization. The sample was cooled again at 0°C and held for 90 min before being transferred to a DSC chamber and held at-25°C for 5 min on the DSC head. The melting profile of sample was measured at a heating rate of 20°C/min to a maximum of 50°C. Measurement was made in triplicate and the mean value was used.

**RESULT AND DISCUSSION**

**Production of Cocoa Butter Emulsion-Milk Chocolate:**

Milk chocolate was produced by conventional lab scale method of mixing, refining and conching. 0 to 4% cocoa butter emulsion was homogenized into tempered milk chocolate. However, a homogeneous distribution of cocoa butter emulsion within tempered chocolate was difficult to achieve due to thickening of chocolate mass during molding especially at higher percentage of emulsion. This was probably resulted from a premature release of water from the emulsion into chocolate mass as cocoa butter melted at warm chocolate temperature. Addition of more than 4% cocoa butter emulsion into tempered chocolate mass was not possible due to a resulted thick texture of the mass during molding. The cocoa butter emulsion was found to contain 47.87% moisture and 51.36% fat. Addition of 1 to 4% cocoa butter emulsion had resulted in the increase of moisture from 1.9 to 3.15% and 34.4 to 38.48% of fat content in chocolate (Table 1). Chocolate samples were kept for 7 days before analyses were conducted in order to allow formation of heat stable structure.

**Heat Resistance Testing:** The samples were 7 days old before heat test was conducted and were kept for 2 hours at respective tested temperatures before subjectively examined for their shape keeping ability, surface appearance, stickiness and texture. The image is illustrated in figure 1. All chocolates were solid and very firm when stored at 28°C. Control chocolate formulation softened slightly at 29°C but chocolates with cocoa butter emulsion continued to be hard and firm. At 30°C control chocolate and chocolate with 1% cocoa butter emulsion were slight soft, but the texture remained firm which gave smooth clean cut with knife. At this temperature, chocolate with 2% emulsion was slightly harder than 1% emulsion and with 3% and 4% emulsion the texture appeared to be hard and firm. The progression of softening at increasing temperature from 29 to 32°C parallels with increasing percentage of emulsion in the chocolate formulation. However there was sign of fat separation where chocolate surface appeared to be very slightly oily and started to become sticky. The chocolate had become predominantly soft at 32°C but with increasing cocoa butter emulsion, the texture was firmer.

**Statistical Analysis:** The experiment was carried out in 3 replicates. Data were analyzed statistically using Minitab-14 software for analysis of variance (one-way ANOVA) and Tukey multiple comparison test at a significance level of 0.05. The correlation between sensory attributes and the amount of cocoa butter emulsion in chocolates was analyzed by Pearson correlation at p<0.05.
Table 1: Moisture and fat content of cocoa butter emulsion and chocolates with 0, 1, 2, 3 and 4% cocoa butter emulsion (cbe)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Moisture content (%)</th>
<th>Fat content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cocoa butter emulsion</td>
<td>47.9 ± 0.78 a</td>
<td>51.4 ± 0.82 a</td>
</tr>
<tr>
<td>+ 0% cbe</td>
<td>1.5 ± 0.48 a</td>
<td>34.4 ± 0.53 a</td>
</tr>
<tr>
<td>+1% cbe</td>
<td>1.9 ± 0.40 ab</td>
<td>35.0 ± 0.54 ab</td>
</tr>
<tr>
<td>+2% cbe</td>
<td>2.3 ± 0.37 abc</td>
<td>35.2 ± 0.02 abc</td>
</tr>
<tr>
<td>+3% cbe</td>
<td>2.5 ± 0.27 bcd</td>
<td>35.8 ± 0.02 bcd</td>
</tr>
<tr>
<td>+4% cbe</td>
<td>3.2 ± 0.42 c</td>
<td>38.5 ± 0.21 c</td>
</tr>
</tbody>
</table>

Mean values from triplicates analysis ± standard deviation. Values followed by different superscripts in the same column differ significantly (p<0.05) (n=3).

Fig 1: Image of chocolate samples with different percentage of cocoa butter emulsion compared to normal chocolate when stored at different temperatures.

The shape of control chocolate started to deform at 34°C and collapsed by 36°C. Chocolates with cocoa butter emulsion however, able to retain their shape up to the highest temperature tested (40°C) even though with a soft texture. Major observation was made with chocolate containing 3% and 4% cocoa butter emulsion. These chocolates were able to give a clean cut, without sticking to knife even though at 40°C. Chocolate with 4% emulsion was however crumbled slightly on cutting, but the texture was harder than the rest.

This application of cocoa butter emulsion has improved the heat stability of chocolate. However it was noted that fat separation occurred where the surface of chocolates appeared oily even though the solid status retained. This showed that the technique did not prevent the fat from melting but rather strengthening the chocolate network by preventing it from collapsing even though the fat has melted. It was also noted that the fat separation decreased with the increase in cocoa butter emulsion in chocolate. This indicated that the chocolate...
network with cocoa butter emulsion had somehow minimized the flow out of molten fat by holding it within the chocolate structure. To overcome the ‘oiling off’ of product surface at increased temperatures, Kemp and Downey [14] suggested exposing the chocolate surface to a humid atmosphere at about 96% RH and 29°C for approximately 2 minutes, before cooling, which leads to the development of a thin sugar crust on the surface. The use of amorphous sugar in chocolate ingredients had also been suggested in the development of thin sugar crust on chocolate surface to prevent the fat separation problem [15].

**Melting Profile Analyses:** The onset temperature represents the starting temperature where the fat crystals begin to melt. This involves the melting of low melting point fats, whilst the end temperature indicates the end of melting process [16]. A shift from solid to liquid phase occurred at the peak temperature [17] where maximum energy is absorbed at this point which involves the melting of high melting point fat crystals. The melting enthalphy is defined as the amount of energy needed to melt all the fat crystals. The onset temperature of cocoa butter emulsion (Table 2) was significantly higher (P<0.05) than that of pure cocoa butter but the peak and end temperatures were not affected significantly (P>0.05). The result suggest that cocoa butter emulsion starts to melt at higher temperature than pure cocoa butter, but the transformation of solid to liquid phase as well as complete meltdown of the fat did not change significantly. Chocolates with increasing cocoa butter emulsion content showed a slight increase in onset, peak and end temperature. However, statistically the difference was not significant (P>0.05). Therefore there was no evidence to say that cocoa butter emulsion increases the melting temperature of chocolate. This is consistent with the observation for heat resistant testing where a sign of fat separation started to appear on surface of chocolate at 30°C and increased with the increase of temperature. The heat stability of chocolate with cocoa butter emulsion as shown from heat resistant testing was then due to the strengthening of its network structure where it was able to hold the chocolate structure even though the fat has melted. This network structure was postulated to minimize the fat from flowing out. This holding capacity increases with increasing emulsion content. This was shown from the lower degree of oily chocolate surface at higher emulsion content.

**Hardness Analyses:** The hardness of chocolate containing different amount of cocoa butter emulsions at ambient temperature (24°C) is illustrated in Figure 2. There were no significance difference (p>0.05) between the hardness of control chocolate and that containing 1-3% cocoa butter emulsions. However, at 4% cocoa butter emulsion the hardness of chocolate decreased significantly, as shown by a lower force needed to break or penetrate the chocolate. Results showed that hardness of chocolate correlate inversely (negative Pearson correlation at p<0.05) with the amount of emulsion. The chocolate became less hard (softer) as the amount of emulsion increased. This could be related with the higher amount of moisture in the chocolate where greater moisture will aggregates sugar particles to form gritty lumps [18]. This resulted in the increase of particle size which in turn would reduce the relative strength of their particle-to-particle interactions [19]. This is evident during heat resistance testing where at 40°C the texture of chocolate with 4% emulsion crumbles on cutting.

![Fig. 2: Hardness, given as the mean force needed to break (breaking force) and penetrate (penetration force) chocolates samples containing 0, 1, 2, 3 and 4% cocoa butter emulsion, determined from three replications (n=3). Error bars are 95% confidence intervals for the mean.](image)

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**Table 2:** Melting properties for chocolate samples with different percentage of cocoa butter emulsion, pure cocoa butter emulsion (cbe) and pure cocoa butter.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Onset temperature (°C)</th>
<th>Peak temperature (°C)</th>
<th>End temperature (°C)</th>
<th>Melting enthalphy (J/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chocolate + 0% cbe</td>
<td>29.0 ± 0.72</td>
<td>34.7 ± 0.34</td>
<td>36.6 ± 0.41</td>
<td>19.9 ± 4.47</td>
</tr>
<tr>
<td>Chocolate + 1% cbe</td>
<td>29.5 ± 0.07</td>
<td>35.8 ± 0.06</td>
<td>38.2 ± 0.59</td>
<td>23.8 ± 5.98</td>
</tr>
<tr>
<td>Chocolate + 2% cbe</td>
<td>29.4 ± 0.90</td>
<td>36.0 ± 0.37</td>
<td>40.3 ± 1.45</td>
<td>20.4 ± 7.14</td>
</tr>
<tr>
<td>Chocolate + 3% cbe</td>
<td>30.0 ± 0.47</td>
<td>35.8 ± 0.15</td>
<td>39.5 ± 2.13</td>
<td>24.5 ± 6.10</td>
</tr>
<tr>
<td>Chocolate + 4% cbe</td>
<td>30.5 ± 0.51</td>
<td>36.2 ± 0.90</td>
<td>39.7 ± 2.30</td>
<td>21.8 ± 7.84</td>
</tr>
<tr>
<td>Cocoa butter emulsion (cbe)</td>
<td>29.3 ± 0.54</td>
<td>35.0 ± 0.09</td>
<td>36.7 ± 0.30</td>
<td>87.5 ± 0.71</td>
</tr>
<tr>
<td>Pure cocoa butter</td>
<td>27.9 ± 0.61</td>
<td>34.5 ± 0.54</td>
<td>38.2 ± 1.42</td>
<td>126.7 ± 2.17</td>
</tr>
</tbody>
</table>

Mean values from triplicates analysis ± standard deviation.
Values followed by different superscripts in the same column differ significantly (p<0.05) (n=3)
Sensory Evaluation: Sensory evaluation of chocolates with different percentage of cocoa butter emulsions was determined from 20 judgments and results are illustrated in Figure 3. The addition of cocoa butter emulsion had reduced the glossiness of chocolate surface and this was very apparent with 4% emulsion where the score was significantly lower, but was still satisfactorily glossy. Below 3% addition of emulsion was however did not significantly affect the glossiness of chocolate. The addition of emulsion also reduced the pleasantness of chocolate odor but the difference was however not significant. The hardness of chocolate on the first bite was also not significantly affected by the addition of emulsion. The rate at which chocolates melted in the mouth also did not show any significant differences, as well as their melting characteristic where all chocolates melts satisfactorily in the mouth. The smoothness of chocolates were however reduced with the increased of emulsion where above 2% emulsion resulted in a slightly sandy texture. This change could be related to the higher amount of moisture content in chocolate where greater moisture aggregates sugar particles to form gritty lumps [18]. The overall acceptability of chocolate was seen to decrease with the increase in emulsion content though statistically the difference was not significant. Chocolate with 1% emulsion gave a very close acceptability score with control chocolate. Overall, the sensory evaluation study showed that the gloss, odor, smoothness and acceptability of chocolate were inversely correlated (negative pearson correlation at p<0.05) with percentage emulsion (or water content) added to chocolate. That is, higher emulsion content lowered the glossiness of chocolate, reduced its pleasant odor and its smoothness. The acceptability score was however did not differ significantly between chocolates containing cocoa butter emulsion (1-4%) and chocolate without cocoa butter emulsion [19].

CONCLUSION

Application of cocoa butter emulsion has improved the heat stability of milk chocolate. Cocoa butter emulsion did not significantly increase the melting point of chocolate; however the heat stability was resulted from the strengthening of its network structure by preventing it from collapsing even though the fat has melted. Lower degree of oily chocolate surface at higher emulsion content showed that the holding capacity of chocolate network increased with the increased of emulsion added. Addition of cocoa butter emulsion at 1 to 3% did not significantly changed the hardness of chocolate but at 4% emulsion content the chocolate became significantly less hard and slightly crumbles on cutting. Increase of cocoa butter emulsion in chocolate also resulted in increase of moisture and fat content. Sensory evaluation study showed that, higher emulsion content lowered the glossiness of chocolate and reduced the pleasant odor and its smoothness. The acceptability score was also lowered however did not differ significantly with the control chocolate.
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REFERENCES