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# Effect of storage temperature on texture, polymorphic structure, bloom formation and sensory attributes of filled dark chocolate

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**Abstract**

The effects of 18 and 30°C storage temperatures on texture, polymorphic structure, bloom formation and sensory attributes of dark chocolate, stored for 8 weeks were studied. Results showed that storage at 18°C for 8 weeks, significantly retarded changes in filled chocolates; the chocolates were free from bloom during the storage period. In contrast, at 30°C there was an increase in the rate of fat migration and rate of change of C36 and C50, and also a decrease in texture and the polymorph structure in the coating changed to β and β′ polymorphs. However, the chocolates bloomed in the third week of storage (2 cycles). Sensory evaluation indicated that, storage at 18°C is better than 30°C, and desiccated coconut gives a pleasant flavour to the chocolate. 

*Keywords:* Storage temperture; Texture; Polymorphic structures; Bloom formation; Sensory attributes; Filled dark chocolate

## 1. Introduction

In chocolate confections filled with a lipid substance, such as peanut butter, lauric (hard butter), and non-lauric, low-melting point lipids tend to migrate into the chocolate coating over time. Lipids most likely to migrate are those with the lowest melting point and greatest fluidity (fatty acids, or triglycerides with short chains and/or unsaturated fatty acids; Talbot, 1990). This migration can cause the chocolate to become sticky and soft and the filling harder (Ziegleder, 1997), and influence the structural integrity of the coating. In addition, if chocolate is invaded to a significant degree by incompatible lipids, such as lauric or non-lauric fats, this will adversely alter the phase behaviour of chocolate lipids, resulting in "bloom" or whitening formation (Paulicka, 1973; Timms, 1984). All of these will reduce consumer acceptability of the product. Fat migration can occur at a considerable rate at room temperature (17–23°C), and this accelerates as the temperature increases (Wootton, Weeden & Munk, 1970; Wacquez, 1975). Migration decreases with increases in the solid fraction of the lipids. Wootton et al. (1970) indicated that the storage of chocolate coated biscuits, under moderate summer conditions (27–32°C), resulted in deterioration of the products. Fat migration in chocolate has been demonstrated by Talbot (1990, 1995, 1996) and Ziegleder (1997) and Ziegleder, Moser and Geier-greguska (1996a, b, 1998)

To date there is limited information on the use of desiccated coconut formulation as base filling centre in chocolate; however, desiccated coconut is a pleasant flavour for most people. Therefore, this study was carried out to determine the effect of fat migration of desiccated coconut (DCN) blended with palm mid-fraction (PMF), on texture, polymorphic structure, bloom formation and sensory attributes of the chocolate at different storage tempertatures.

## 2. Material and methods

### 2.1. Material

PMF was supplied by Soctech Edible Oil Sdn Bhd. Cocoa butter (CB) and cocoa liquor was obtained from KL-Kepong Sdn Bhd; desiccated coconut was obtained from a general store. Petroleum ether (bp 40–60°C),

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acetone high performance liquid chromatography (HPLC) grade and acetonitrile (HPLC grade) were obtained from Merck. Triglyceride standards were obtained from Sigma Chemicals.

### 2.2. Preparation and storage of chocolate samples

The fat migration was stimulated in real products by using layers of cream filling and dark chocolates, approximately 100 g each, in a plastic container (10×5×5 cm length×width×height), respectively; 30% PMF + 15% DCN + 55% icing sugar were carefully blended to form a homogeneous paste to fill up the filling section. For the coating section, dark chocolate was prepared by using cocoa liquor (42%), CB (8.4%), sugar (41.6%), skimmed milk powder (8%) and lecithin (0.4%). All samples were held for 2 months at 18 and 30°C for further testing.

### 2.3. Triglyceride profile of fats used

Triglyceride profiles were determined by HPLC according the AOCS (1989) method. Mobile phase was acetone: acetonitrile 75:25; the sample was diluted in acetone to make up to 10% sample concentration; a volume of 10 μl was injected into a water C18 column (3.9×300 mm); the flow rate was 1.5 ml/min. The value was expressed as a percentage. All samples were analysed in duplicate.

### 2.4. Determination of thermal behaviour of the fats used

Solid fat content (SFC) profiles were evaluated according to the IUPAC (1987) method using a Bruker Nuclear Magnetic Resonance (NMR). Tubes (10 mm diameter) were filled, with samples at 60°C for 30 min, 0°C for 90 min, 26°C for 40 h and 0°C for 90 min. SFC was determined at 5, 10, 15, 20, 25, 30 and 35°C; measurements were made in triplicate. The final SFC was calculated as an average of these three readings. Melting point was determined by the Differential Scanning Calorimeter (DSC) method as described by Md. Ali and Dimick (1994). Samples (3±0.01mg) were tempered in DSC pans with the same methods as for SFC determination. DSC melting curves were recorded at a heating rate of 10°C/min from −20 to 50°C. Melting point was obtained from the DSC thermogram. Analysis was carried out in duplicate

### 2.5. Determination of texture

Hardness or degree of softening was measured by determining the maximum penetration force for chocolate. A Texture Analyser Model TA-XT2I (UK) was used to measure the depth of penetration of samples using the following parameters: Product height = 10 mm, penetration depth = 6 mm; probe needle PN2, temperature 20°C, pre-speed 1.0 mm/s, test speed = 1.1mm/s, post speed = 10.0mm/s and the duration time of the test took approximately 1–2 min.

### 2.6. Determination of polymorphic structure

The polymorphic forms of the fat mixture crystals in the samples were determined using a FR 592 Diffractis X-ray generator (Delft, Holland). The samples were held at 0°C for 90 min. For crystal stabilization, the samples were then incubated at 26°C for 40 h and at 0°C for 90 min prior to measurement. The samples were placed at 20°C within a single-compartment cell using a custom-made temperature controlled holder maintained at 20°C by an external circulation thermostatted bath. A Kodak (Eastman Kodak Co., Rochester, NY) diagnostic film direct exposure (Cat 155 8162) was used and the spacings on X-ray film were measured with an Enraf-Nonius Guiner viewer capable of reading the nearest 0.001 nm under illuminated magnification.

### 2.7. Determination of bloom formation

Bloom was determined using the Cambell (1967) method. Chocolate was placed in a humidity chamber (KATO, Japan) at 80% relative humidity. The complete cycle for the bloom was carried out by exposing the chocolate to 30°C for 8 h, followed by 20°C for 16 h. The number of complete cycles required for bloom was recorded.

### 2.8. Sensory evaluation

Sensory attributes of filled chocolate, i.e. colour, texture, flavour and overall acceptability were evaluated using a 7-point hedonic scale (1 = dislike extremely, 4 = moderate, 7 = like extremely) by 25 tranined panellists selected from post-graduate students and staff of the Faculty of Food Science and Biotechnology, Universiti Putra Malaysia (Larmond, 1977).

### 2.9. Statistical analysis

All measurements and/or experiments were conducted in duplicates. The statistical analyses for determining analysis of variance and degree of significance were carried out using the Statistical Analysis System (SAS, 1997).

## 3. Results and discussion

### 3.1. Triglyceride profile

Table 1 shows that CB was composed of three high molecular weight triglycerides, C50, C52 and C54. PMF

comprises C50 and C52 triglycerides. Modification of palm oil by double fractionation yields a PMF with concentrated levels of palmitic oleic palmitic (POP) and stearic oleic stearic (POS) (Pease, 1985). On the other hand, coconut oil consists of a wide range of triglycerides from C32–C38 with substantial amounts of the shorter chain fatty acids series (Wong, 1991). On blending 15% desiccated coconut with 30% PMF, POP C50 of the PMF decreases by 16%, because new triglycerides had invaded, e.g. trialaurin (C36) and C38.

Table 1
Triglyceride composition of chocolate and filling fats[a]

| CNP (%) | CB | PMF | CNO | 30%PMF + 15%DCN |
|---|---|---|---|---|
| C26 | – | – | 0.3 | 0.10 |
| C28 | – | – | 0.8 | 0.32 |
| C30 | – | – | 2.7 | 0.99 |
| C32 | – | – | 11.1 | 3.78 |
| C34 | – | – | 15.3 | 6.75 |
| C36 | – | – | 19.4 | 6.16 |
| C38 | – | – | 16.2 | 4.85 |
| C40 | – | – | 11.7 | 2.76 |
| C42 | – | – | 8.6 | 1.82 |
| C44 | – | – | 4.8 | 1.50 |
| C46 | – | 0.3 | 2.8 | 1.45 |
| C48 | 0.7 | 4.1 | 2.6 | 1.32 |
| C50 | 19.7 | 72.3 | 2.1 | 50.14 |
| C52 | 45.7 | 21.5 | 1.6 | 9.50 |
| C54 | 33.2 | 2.0 | 0.8 | 2.14 |

[a] CNP, carbon number profile; CB, cocoa butter; PMF, palm mid fraction; CNO, coconut oil; DCN, desiccated coconut.

### 3.2. Thermal behaviour

Figs. 1 and 2 represents then melting point and SFC characteristics of CB, PMF, coconut oil (CNO) and PMF + CNO, respectively Cocoa butter shows a sharp SFC profile from 20–30°C and melting point compared to PMF. Cocoa butter is hard and brittle at temperatures up to 27°C; most melting occurs over the narrow range of 27–33°C and is essentially complete at 35°C, whereas PMF and CNO completely melted at 31.2 and 25°C, respectively (Fig. 2). This melting profile is a key to the performance and suitability of cocoa butter for confectionery applications (Wainright, 1996). CNO is much softer (as a base for cocoa butter substitute (CBS)); it is inferior to palm kernel oil-based CBS. Higher amounts of short-chain fatty acids, but relatively lower amounts of unsaturated fatty acids gives a low

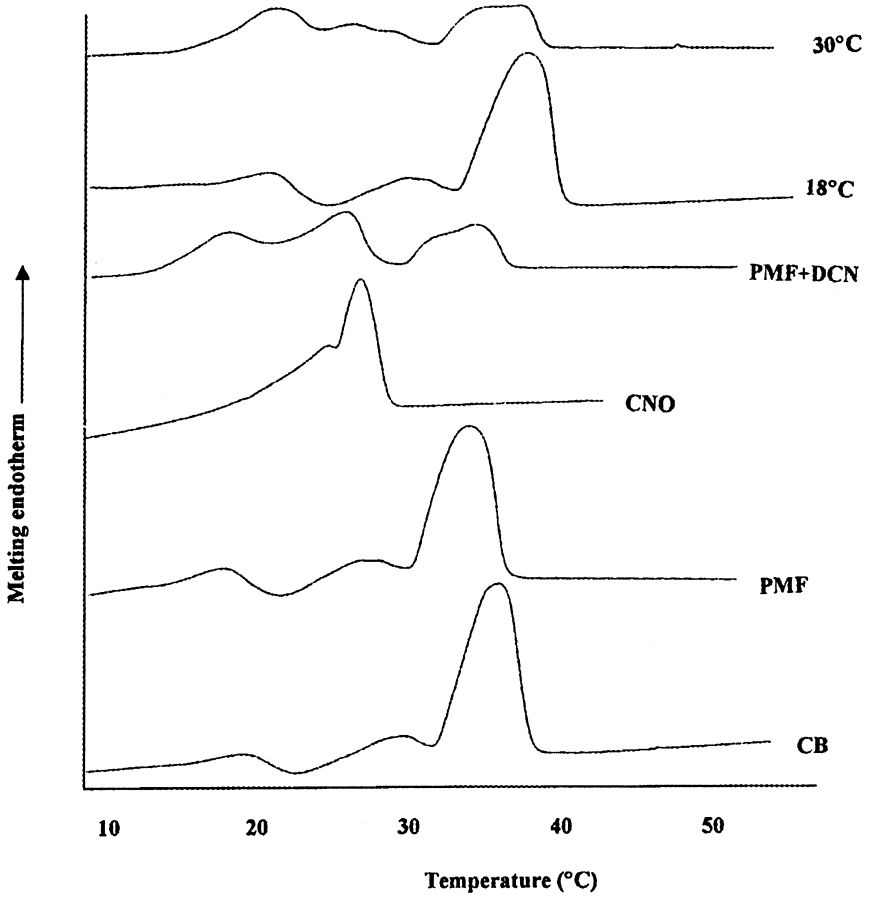


Fig. 1. Melting point differential scanning calorimeter of CB, PMF, PMF + DCN and dark chocolate filled with PMF + DCN stored at 18 and 30°C for 8 weeks. PMF, palm mid-fraction; DCN, Desiccated coconut; CB, cocoa butter.

melting stearin (Smp; 30°C), especially suitable for wafer and other fillings (Wong, 1991). Blending of 15% desiccated coconut to 30% PMF, results in eutectic effects at 25–30°C, due to incompatibility between their triglyceride compositions (Herzing, 1989).

### 3.3. Rate of change of C36 and C50

The linear curve fitting displayed in Fig. 3,was used to provide the rate of movement of C36 (LLL) and C50 (POP) from the filling centre into the chocolate layer by the following expression:

$$Y = k\mathrm{t} + b$$

$$\frac{\mathrm{d}y}{\mathrm{d}t} = k$$

where $Y$ is the triglyceride (%), $k$ is the rate of movement % week$^{-1}$ of the triglyceride, $t$ is the storage time/week, $b$ is the intercept. Ziegleder et al, (1998) noted that the rate of fat migration decreases with the decrease in storage temperature. At 18°C, the rate of movement of C36 and C50 was 0.13 %/week and 0.15 %/week, respectively. However, at 30°C, the rates of movement of C36 and C50 were dramatically increased in the chocolate layer to 0.53 %/week and 2.17 %/week, respectively. Guiheneuf, Cousens, Wille and Hall, (1997) indicated that the amount of lipids migrated is relatively higher at 28°C than 19°C. Hence, they suggested that the mechanism of migration involves both, diffusion of the liquid triacylglycerols and capillary attraction of the hazelnut oil into the chocolate matrix.

### 3.4. Texture

Chocolates, filled with PMF + DCN stored at 30°C, were substantially softer than those stored at 18°C (Fig. 4). This was due to the fact that at 30°C, PMF and CNO mixture was completely melted, having (0%) SFC, leading to marked migration of the filling fats into the chocolate layer. PMF and CNO, which have substantially different fatty acids compositions, would show more pronounced eutectic behaviour. In general, the more dissimilar the fatty acid composition of two blended fats, the stronger will be the melting point depression and the softer the fat blend (Herzing, 1989).

Incompatibility occurs between three blended fats, (CB/PMF/CNO triglycerides). However, a new liquid phase appears which results in the softening of the chocolate layer. According to Dimick and Manning (1987), an increase in the amount of liquid phase results in softer chocolate.

### 3.5. Polymorphic structure

The polymorphic structures of CB, PMF, PMF + DCN and chocolates stored during the 8 weeks at 18°C and 30°C are shown in Tables 2 and 3, respectively. In CB, two strong spacings near 4.55 and 4.5 A, were found, which indicate the formation of β polymorph. The same findings were reported by Riiner (1970), Chapman, Akehurst and Wright, (1971), Timms (1984) and Sabariah, Md. Ali and Chong (1998). Chocolate stored at 18°C contained only β at all times. At 30°C and 2 weeks of storage, β was more dominant than β. A mixture of β′ and β crystals was observed during

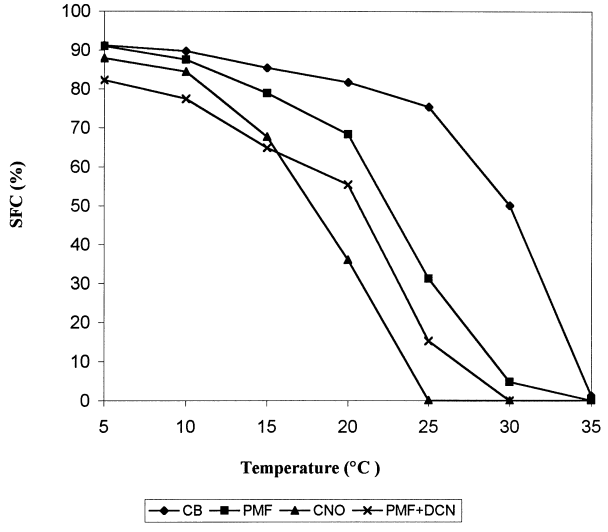


Fig. 2. Solid fat content (%) of CB, PMF, CNO and PMF + DCN. CB, cocoa butter; CNO, coconut oil; PMF, palm mid-fraction; DCN, desiccated coconut. Each value in the table represents the means ± S.D. of four measurements.

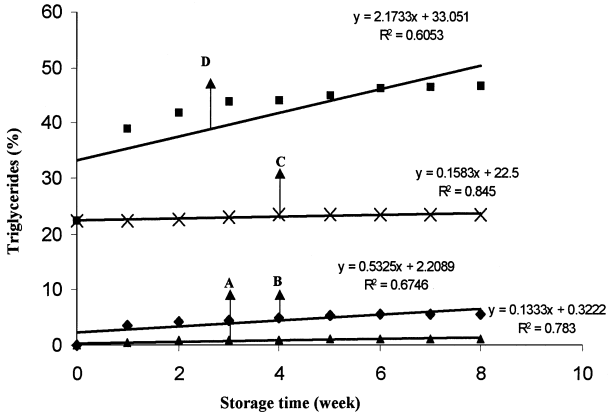


Fig. 3. The rate of change of trilaurin (C36; LLL) and C50 (POP) triglycerides in the dark chocolate filled with PKS + DCN, stored for eight weeks at 18 and 30°C. A, Rate of change in C36 (LLL) of chocolate stored at 18°C; B, Rate of change in C36 (LLL) of chocolate stored at 30°C; C, Rate of change in C50 (POP) of chocolate stored at 18°C; D, Rate of change in C50 (POP) of chocolate stored at 30°C; LLL, Trilaurin; POP, Palmitic-Oleic - Palmitic. PMF, palm mid fraction, DCN = desiccated coconut. Each value in the table represents the means ± S.D. of four measurements.

the last 6 week's storage period. According to Timms (1984) and Kheiri (1982) PMF fat is stable in β′-2-polymorph In our study, we found that this could be due to an increase of the percentage of POP and undesirable glycerides such as POO, PPP and PPO from PMF and C36, from coconut oil, in the chocolate layer. Riiner, Chapman, Akehurst & Wright (1971) and Timms observed that β formation is more likely in the binary and ternary mixtures where Malaysian CB and CBE were most dominant.

### 3.6. Bloom formation

Table 4 shows the effect of storage temperature at 18 and 30°C on the bloom cycle of the chocolate layer. No bloom was observed at 18°C storage temperature; at 30°C, the onset of the bloom was observed after one week, which took around four cycles to bloom. The bloom was found to be due to recrystallization of the PMF, CNO and CB triglycerides mixture (Laustsen, 1991). PMF crystallizes out in β′-2 form because it is usually contaminated with PPO and other unsaturated glycerides from palm olein (Kheiri, 1982), and the presence of CNO triglycerides in the filling with PMF increases the probability of β′ formation. At 30°C, migration increases into the chocolate layer as time increases until equilibrium. Lovegren, Gray and Feuoge (1976) and Cruickshank and Biol 1979 have studied the action of 10% olive oil on the polymorphic behaviour of cocoa butter and have found that the transformation rates were markedly increased by an increase in liquid content; bloom is more likely to occur when the chocolate is stored at relatively high temperature (25–32°C;Talbot, 1995).

### 3.7. Sensory evaluation

Results of sensory evaluation are shown in Table 5. The colour and texture of the chocolates stored at 30°C were significantly ($P < 0.05$) less preferred than the

Table 2
X-ray diffraction pattern of dark chocolate filled with PMF + DCN stored for 8 weeks at 18°C by X-ray diffractometer after stabilization (at 26°C for 40 h)[a]

| Storage time (week) | Short-spacing (A)[b] | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 5.3 | 5.2 | 4.6 | 4.5 | 4.4 | 4.2 | 4.1 | 4.0 | 3.9 | 3.8 | 3.7 | 3.6 | Polymorphic form |
| 1 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 2 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 3 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 4 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 5 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 6 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 7 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| 8 | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| CB | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| PMF + DCN | | | | | 3.48m | 4.20s | | | 3.99w | | 3.78s | | β′ |

[a] CB, cocoa butter; PMF, palm mid-fraction; DCN, desiccated coconut.
[b] Intensities estimated visually as: s, strong; m, medium; w, weak; vw, very weak.

Table 3
X-ray diffraction pattern of dark chocolate filled with PMF + DCN stored for 8 weeks at 30°C by X-ray diffractometer after stabilization (at 26°C for 40 h)[a]

| Storage time (week) | Short-spacing (A)[b] | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 5.2 | 4.6 | 4.5 | 4.4 | 4.3 | 4.2 | 4.1 | 4.0 | 3.9 | 3.8 | 3.7 | 3.6 | Polymorphic form |
| 1 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 2 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 3 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 4 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 5 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 6 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 7 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| 8 | 5.37w | 5.19vw | | 5.55s | 3.38w | 4.26m | 4.17s | 4.08m | 3.95w | | 3.78s | 3.62w | β + β′ |
| CB | 3.38m | | | 4.55s | 4.46w | 4.24vw | 4.16vw | 4.05w | 3.94m | 3.83m | 3.71m | 3.62m | β⋙β′ |
| PKS + DCN | | | | | 3.48m | 4.20s | | | 3.99w | | 3.78s | | β′ |

[a] CB, cocoa butter; PMF, palm mid-fraction; DCN, desiccated coconut.
[b] Intensities estimated visually as: s, strong; m, medium; w, weak; vw, very weak.

Table 4
The bloom cycles of the dark chocolate filled with PMF + DCN and stored for 8 weeks at different storage temperatures[a]

| Week | Storage Temperature (°C) | | | |
|---|---|---|---|---|
| | 18°C | | 30°C | |
| | Fat Bloom[b] | Cycles[c] | Fat bloom[b] | Cycles[c] |
| 0 | – | 7[a] | + | 6[a] |
| 1 | – | 7[a] | + + | 4[a] |
| 2 | – | 7[a] | + + + + | 2[b] |
| 3 | – | 7[a] | + + + + | 1[c] |
| 4 | – | 7[a] | + + + + | 1[c] |
| 5 | – | 7[a] | + + + + | 1[c] |
| 6 | – | 7[a] | + + + + | 1[c] |
| 7 | – | 7[a] | + + + + | 1[c] |
| 8 | – | 7[a] | + + + + | 1[c] |

[a] One cycle is 30°C (16 h) and 20°C (8 h). Values with the same letter within a column is not significantly different at 5% level ($P > 0.05$) PMF, palm mid-fraction; DCN, desiccated coconut.

[b] Fat bloom occurrence evaluated by visual observation: -, no bloom; +, weak bloom; + +, bloom; + + +, strong bloom; + + + +, intensive bloom.

[c] Cycle, counted cycles to bloom.

Table 5
Sensory characteristics of dark chocolate filled with PMF + DCN, stored for 8 weeks at 18 and 30°C[a]

| Sensory Attributes | Control | 18°C | 30°C |
|---|---|---|---|
| Colour | 5.68a±1.3 | 5.13b±1.0 | 3.56C±1.1 |
| Texture | 5.88a±1.2 | 5.96a±1.1 | 4.52C±0.9 |
| Flavour | 4.10b±0.9 | 5.55a±0.8 | 5.66a±1.3 |
| Overall acceptability | 5.11±1.1 | 5.68C±1.2 | 4.14C±0.7 |

[a] Panelists means sensory±standard deviation. Each value in the table represents the means ±S.D. of four measurements.Means within the same row with different letters are significantly different ($P < 0.05$). PMF, palm mid-fraction; DCN, desiccated coconut.

control and chocolates stored at 18°C. Fat migration adversely affects product integrity and appearance. Typical deterioration effects, frequently encountered in fat migration are softening and blooming of the coated layer and unacceptable textural change in centres, due to the loss of liquid glycerides from the filling centre (Timms, 1984; Ziegleder, 1997). All of these will reduce consumer acceptability of the product.

The flavour of the control was significantly ($P < 0.05$) less preferred than the chocolates stored at 18 and 30°C; however, the chocolate stored at 30°C scored higher for the flavour attribute but scored low in overall acceptability compared to the control and that stored at 18°C. Allen (1965) reported the presence of methyl ketones (C7, C9, C11, C13, C15) and deltalactones (C6, C8, C10, C12, C14) in volatile constituents of CNO. DCN possesses a pleasant characteristic aroma which appear to be an acceptable flavour to most people (Woodroof, 1979). This study indicates that storing chocolate filled

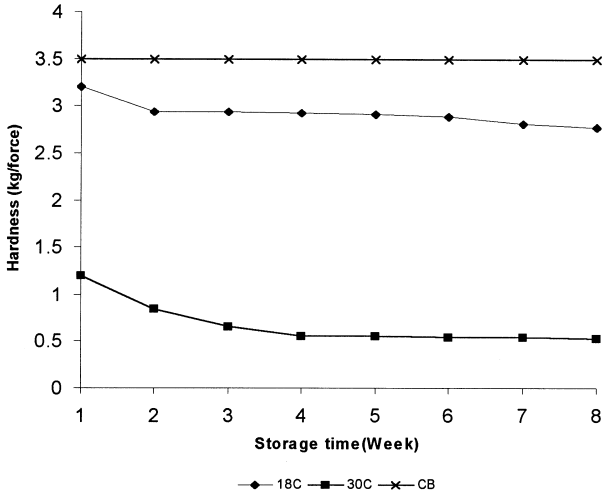


Fig. 4. Hardness of dark chocolate filled with PMF, stored for 8 weeks at different storage temperatures. PMF, palm mid-fraction; DCN, desiccated coconut. Each value in the table represents the means ± S.D. of four measurements.

with PMF + DCN fats at 18°C is the best way to retain its flavour.

From this study it can be concluded that the PMF + DCN migration at 18°C was very slow and the changes were minimal in terms of chemical composition, hardness, glossiness and polymorphic stability. PMF migration into the chocolate layer was enhanced by higher storage temperature resulting in softening effects, which leads to polymorphic transformation, which eventually leads to bloom formation. Desiccated coconut, when blended with PMF, gave a melt-away centre, to give cooling sensation in the mouth; furthermore, desiccated coconut contributed a pleasant flavour to the chocolate if stored below 20°C.

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