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# The effect of sunflower wax and carnauba wax on the storage stability and sensory evaluation of palm-based chocolate spread

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**SUMMARY:** This study investigates the storage properties and sensory evaluation of palm-based chocolate spreads formulated with sunflower wax (SFW) and carnauba wax (CW) in a wide range of temperatures. Hardness, spreadability and stability assessments of chocolate spreads with 3.5% SFW and 3.5% CW were made over a 24-week storage period. The hardness, spreadability and stability of the chocolate spreads were analyzed by means of Texture Analyzer, observation and LUMiFuge Stability Analyzer, respectively. A sensory evaluation of these chocolate spreads was conducted using a 7-point hedonic scale. The hardness of both chocolate spreads was influenced by low temperatures of 5-10 °C during the storage study. Decreasing and increasing trends were observed for samples stored at 5 and 10 °C, respectively. However, both palm-based chocolate spreads remained spreadable at 5 °C even after 24 weeks of storage. Both chocolate spreads demonstrated stability at 40 °C after 24 weeks of storage. The sensory evaluation revealed that chocolate spread with 3.5% CW was the most widely accepted by sensory panelists. Therefore, chocolate spread with 3.5% CW has the potential to be utilized over a wide range of temperature applications of 5-40 °C.

KEYWORDS: Carnauba wax; Palm-based chocolate spread; Sensory evaluation; Storage study; Sunflower wax

**RESUMEN:** *Efecto de las ceras de girasol y de carnauba sobre la estabilidad durante el almacenamiento y la evaluación sensorial de la crema de chocolate a base de palma*. Este estudio investiga las propiedades durante el almacenamiento y la evaluación sensorial de las cremas de chocolate a base de palma formuladas con cera de girasol (SFW) y cera de carnauba (CW) para lograr una amplia aplicabilidad de temperatura. La dureza, la capacidad de untar y la estabilidad de las cremas de chocolate con 3,5 % de SFW y 3,5 % de CW se llevaron a cabo durante un período de almacenamiento de 24 semanas. La dureza, la capacidad de untar y la estabilidad de las cremas de chocolate se analizaron utilizando el Analizador de Textura, la observación y el Analizador de Estabilidad LUMiFuge, respectivamente. La evaluación sensorial de estas cremas de chocolate se realizó utilizando una escala hedónica de 7 puntos. La dureza de ambas cremas de chocolate se vio influenciada por la baja temperatura de 5 °C-10 °C durante el almacenamiento. Se observaron tendencias decrecientes y crecientes para las muestras almacenadas a 5 °C y 10 °C, respectivamente. Sin embargo, ambas cremas de chocolate a base de palma siguieron siendo untables a la temperatura de 5 °C, incluso después de 24 semanas de almacenamiento. Además, ambas cremas de chocolate con 3,5 % de agua destilada fue la más aceptada por los expertos en análisis sensorial. Por lo tanto, la crema de chocolate con 3,5 % de agua destilada tiene el potencial de ser utilizada para aplicaciones en un amplio rango de temperaturas de 5 a 40 °C.

**PALABRAS CLAVE**: Cera de carnauba; Cera de girasol; Crema de chocolate a base de palma; Estudio de almacenamiento; Evaluación sensorial.

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### **1. INTRODUCTION**

Chocolate spread is a product commonly used in confectionery. Its desirability is largely attributed to properties imparted by oils and fats, which contribute to its ability to be spread smoothly and maintain stability. The desirable physical properties dependent on the oils and fats' chemical structure are related to the fatty acid composition (FAC) and consequently, triacylglycerol (TAG) composition. A common criterion for chocolate spread is that it should be stable for 6-12 months at room temperature of about 25-28°C (Shamsudin, 2004). During this period, phase separation (oiling out) should not occur at room temperature and the chocolate spread should be readily spreadable without difficulty.

In order to obtain a chocolate spread that is stable and spreadable at a wide range of temperature (between 5 and 45 °C), the incorporation of oils and fats without modification may not work. At 45 °C, chocolate spread will experience phase separation (oiling out), while at a low temperature of 5 °C, the chocolate spread will solidify. These scenarios of phase separation and solidification are usually seen in temperate countries. Phase separation at higher temperatures of 35 °C and above is normally seen in tropical countries. In this regard, the oleogelation method might be the solution to these undesirable occurrences. Oleogelation is a method in which the gelling agent traps liquid vegetable oils to form a three-dimensional (3D) network system to structure food products. Öğütcü and Yilmaz (2014) reported that by employing oleogelation, the saturation level of the products' fatty acids remains unchanged. Oleogelation addresses temperature-related stability concerns.

The production of chocolate spreads with a widerange temperature stability using the oleogelation method, facilitated by the direct dispersion of waxes, has gained attention in recent research. Wax consists of wax ester, fatty acid, fatty alcohol and n-alkane. Sunflower wax (SFW) for example, consists of 75.0 -85.0% wax ester (Endlein and Peleikis, 2011) while carnauba wax (CW) consists of 96.0 -97.0% wax ester (Doan *et al.*, 2017). Wax with a higher percentage of wax ester is suitable as a gelling agent to trap oil in a three-dimensional (3D) network to behave like a solid fat (Manzocco *et al.*, 2017). The FDA has approved natural waxes as ingredients and additives in food (Rocha *et al.*, 2013). The approved limit for carnauba wax is 5000 mg/kg (Codex A., 2024).

Despite advancements in production techniques, comprehensive storage studies evaluating the spreadability and stability of such chocolate spreads remain limited in the literature. Limited information could be obtained from a literature review on storage studies of chocolate spread using the oleogelation method from wax. Fayaz et al. (2017) reported that the firmness of chocolate spread with beeswax (BW) and propolis wax which replace part of the palm fraction in these products showed an increasing trend during a 23-day storage period at 20 °C. However, the firmness of monoglyceride-containing chocolate spread in a similar study by Fayaz et al. (2017) was slightly reduced during the storage study under similar temperature and storage conditions. It was also reported that chocolate paste containing shellac wax to replace part of the palm fraction did not undergo phase separation during a 30-day storage study at 30 °C (Patel et al., 2014). On the contrary, Said et al. (2019) produced three types of chocolate spread (without oleogelation process) each using palm olein (POo), corn oil and olive oil in addition to the palm fraction found in the basic chocolate bars used. It was reported that the firmness of the three chocolate spreads stored at 28 °C for 28 days was significantly reduced compared to chocolate spread stored at 10 °C. Nevertheless, chocolate spread with olive oil showed a significant change during the storage study at 28 °C, while chocolate spread with POo showed a significant change at 10 °C.

While sensory evaluation is important in assessing the overall quality of food products, limited research exists on the sensory attributes of chocolate spreads containing wax-based oleogels. In a sensory evaluation for chocolate spread consisting of different fat systems, 50% hydroxypropylmethyl cellulose-based oleogels (HPMC), xanthan gum, olive oil and sunflower oil (SFO), the spread was acceptable according to panelists compared to the control chocolate spread (without oleogels). Chocolate spread with this oleogel had a creamy texture and appearance as well as imported cocoa flavor (Bascuas et al., 2021). Scores for sensory evaluation other than the chocolate spread, such as sausages containing rice bran wax oleogels, could not match the properties of normal sausages (control sample) in terms of firmness and elasticity. Nevertheless, the oleogel

sausages were able to match the adhesion, cohesiveness and elasticity properties of the control sausages (Wolfer *et al.*, 2018).

In this study, palm-based chocolate spreads containing 3.5% sunflower wax (SFW) and 3.5% carnauba wax (CW) were selected for a storage study over six months as they were found to be stable at 40 and 45 °C after 24 h of storage. Hardness, spreadability and stability tests of the chocolate spreads were conducted every two weeks for six months. Sensory evaluation was also performed on the chocolate spreads containing 3.5% SFW and 3.5% CW at 25 °C.

# 2. MATERIALS AND METHODS

#### 2.1. Materials

POOo (Fry-O) was manufactured by Mewah Oils Sdn. Bhd. (Selangor, Malaysia), SFO (Naturel) was manufactured by Lam Soon Edible Oils Sdn. Bhd. (Selangor, Malaysia), SFW was manufactured by Shri Balaji Driers (Karnataka, India) and CW was distributed by Anizz Legacy Resources (Selangor, Malaysia).

Sugar (Prai) was manufactured by Malayan Sugar MFG. Co. Bhd. (Pulau Pinang, Malaysia) while cocoa powder was manufactured by K.L Kris Food Industries Sdn. Bhd. (Selangor, Malaysia). Soy protein, emulsifier and flavoring were manufactured by LLC Co. (Missouri, United States of America), EvaChem Sdn. Bhd. (Selangor, Malaysia) and Guangdong Huana Chemistry Co. Ltd. (Guangdong, China) as well as Matrix Flavors Fragrances Sdn. Bhd. (Selangor, Malaysia). Vanillin was bought from Yummy Bakery Sdn. Bhd. (Selangor, Malaysia) and milk powder was bought from Promac Enterprises Sdn. Bhd. (Selangor, Malaysia).

#### 2.2. Methods

#### 2.2.1. Preparation of chocolate spread

Chocolate spread was produced according to the method by Shamsudin (2004). An oil blend of 50% POOo:50% SFO was selected as the base oil to be blended with 3.5% SFW and 3.5% CW, respectively, to form an oleogel structure. The base oil of 50% POOo:50% SFO was selected as this oil blend showed 0.0% SFC at the temperature range of 5-45 °C. Base oil comprising 50% POOo:50% SFO as well as 3.5% SFW and 3.5% CW was chosen based on the study by Norazura Aila *et al.* (2022). The storage study of chocolate spreads with 3.5% SFW and 3.5% CW was conducted at 5, 10, 25, 40 and 45 °C for six months. Chocolate spreads with 3.5% SFW and 3.5% CW at 5, 10 and 25 °C were stored in an incubator, while at 40 °C and the samples were stored in an oven. Stability, spreadability and hardness tests of the stored samples were conducted every two weeks.

#### 2.2.2. Hardness of chocolate spread

Chocolate spreads of 110 g containing oleogelators were placed in a 6 cm (diameter) x 6 cm (height) container. These chocolate spreads were stabilized at 25 °C for 24 hr. The samples were then stored at 5 °C (incubator, Sanyo), 10 °C (incubator, Sanyo), 25 °C (incubator, Protech SD350), 40 °C (oven, Memmert UF110) and 45 °C (oven, Memmert, UF40) for six months and were tested every two weeks. The hardness of these chocolate spreads was measured using Texture Analyzer (TA.XT Plus, Stable Micro System, England) following the method from the library of texture analyzer regarding chocolate spreads with modification. Chocolate spreads were penetrated with a 5-mm cylinder probe to a depth of 23 mm and rate of 1 mm/sec. The maximum force  $(g_f)$  used was recorded as the hardness value. Three replicates were conducted for hardness.

#### 2.2.3. Kinetic model for hardness of chocolate spread

Changes in the quality or deteriorative parameter can be represented by a general mathematical model as below:

$$- dA/dt = kAn$$

in which dA/dt is the change in response A with time, A is the quality or deteriotive parameter measured, t is the time, k is rate constant and n is the power factor or order of reaction.

Following this, the derivatization of the Arrhenius model to fit various orders of reaction were used to fit with the hardness data as follows:

1)  $t_{E} = (A_{E} - A_{O})/k$  (Zero-order reaction)

2)  $t_{E} = (\ln A_{O} - \ln A_{E})/k$  (First-order reaction)

3)  $t_{\rm E} = (1/A_{\rm E} - 1/A_{\rm O})/k$  (Second-order reaction)

in which  $t_E$  is shelf life,  $A_E$  is exceed hardness (in this case, 2105 g<sub>p</sub> based on unspreadable hardness of commercial chocolate spread),  $A_O$  is initial hardness and k is rate constant.

# 2.2.4. Spreadability of chocolate spread (by observation)

Chocolate spreads of 110 g containing oleogelators were placed in a 6 cm (diameter) x 6 cm (height) container. These chocolate spreads were stabilized at 25 °C for 24 hr. The samples were stored at 5 °C for six months and were tested every two weeks. The spreadability of the chocolate spreads was observed according to their ability to spread on white paper using a butter knife before 30 sec. Three replicates were conducted for spreadability.

#### 2.2.5. Stability test of chocolate spread

The stability of the chocolate spreads with oleogelators during storage was analyzed using a centrifugal stability analyzer according to Sivaruby et al. (2013) with slight modification. These chocolate spreads were tempered until 24 weeks at 25, 40 and 45 °C. Stability measurements were taken every two weeks using LUMiFuge Model LF111 LUM (LUM Ltd., Berlin, Germany). The samples were gently filled into rectangular polycarbonate cells using syringes with needles to prevent any air pockets. The loaded cells were placed horizontally in the centrifuge. The selected centrifugal analysis parameters were rotational speed of 500 rpm for a period of 50 min at experimental temperatures of 25, 40 and 45 °C. This system measures near infrared (NIR) transmission profiles continuously during centrifugation. LUMiFuge software, SEPView 5.1 was used to calculate the integral of every transmission curve over a chosen length (the sample length). Zero transmission indicates stable samples.

#### 2.2.6. Sensory evaluation of chocolate spread

An acceptance test was conducted using 30 untrained panelists (Handiati *et al.* 2019) and a 7-point structured hedonic scale for the attributes of glossiness, spreadability, color, smoothness, sweetness, oily taste and overall acceptability.

#### 2.3. Statistical analyses

The results of the hardness, spreadability and stability test were analyzed with Minitab version 16.0 (Pennsylvania, USA) using two-way Analysis of Variance (ANOVA) with factors of duration and temperature. All tests were carried out in triplicate. For sensory evaluation, one-way ANOVA was conducted with 30 panelists. Significant differences ( $p \le 0.05$ ) among the samples were determined using the Tukey's *post-hoc* test.

#### **3. RESULTS AND DISCUSSION**

#### 3.1. Hardness of chocolate spread

The hardness of chocolate spreads with different oleogelators during the storage study on day 1 (Week 0) to week 24 is shown in Figures 1 and 2, while the predicted shelf life of chocolate spreads at 5, 10 and 25 °C based on the unspreadable condition is shown in Table 1. Based on 2-way ANOVA, statistically, a storage period of 0-24 weeks was not a significant factor (p > 0.05), storage temperature of 5-45 °C was a significant factor (p < 0.05), whilst the interaction between time and temperature was also a significant factor (p < 0.05) for both chocolate spreads with 3.5% SFW and 3.5% CW. In general, at 5 °C, the hardness of chocolate spread containing 3.5% SFW showed a decreasing trend. Even so, they were statistically similar (p > 0.05) upon storage at week 4 until week 24. Significant differences (p < 0.05) for Week 0 and week 2 compared to week 4 until week 24 might be due to the stabilization period. Doan et al., 2016 and Toro-Vazquez et al., 2007 reported that microplatelet crystalline particles of the oleogel re-organized their molecular packing within their network and caused structural changes during storage. On the other hand, a study by Öğütcü and Yilmaz (2015) reported that the hardness of an oleogel containing 3% SFW in hazelnut oil showed an increased hardness after storage of 30 days, plateau upon storage from day 31 to day 60 and decreased after storage of 90 days at 4 °C. However, similar oleogels containing 7 and 10% SFW showed fluctuating as well as increasing trends, respectively, for the same storage period at 4 °C. All of these trends showed that there might not be a specific trend for the oleogel studied by Öğütcü and Yilmaz (2015), which depended on the percentage of wax incorporation and temperature.



FIGURE 1. Hardness of chocolate spread with 3.5% sunflower wax (SFW) at a) 5 °C b) 10 °C c) 25 °C d) 40 °C and e) 45 °C during storage study of week 0 (day 1) – week 24. The results are expressed as mean ± standard deviation (n = 3).



FIGURE 2. Hardness of chocolate spread with 3.5% carnauba wax (CW) at a) 5 °C b) 10 °C c) 25 °C d) 40 °C and e) 45 °C during storage study for week 0 (day 1) – week 24. The results are expressed as mean ± standard deviation (n = 3).

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Sample	Temperature (°C)	Reaction Order	R2	$\frac{\mathbf{A}_{_{0}}}{(\mathbf{g}\mathbf{f})}$	A <sub>e</sub> (gf)	k	T <sub>E</sub> * (Years)
3.5% SFW	5	1	0.6775	1722.20	2105	0.0118	13.16
	10	1	0.9160	515.18	2105	0.0206	6.32
	25	0	0.6674	58.04	2105	0.8147	52.34
3.5% CW	5	1	0.8799	1896.80	2105	0.0251	6.26
	10	1	0.8856	471.50	2105	0.0303	4.23
	25	0	0.5604	32.27	2105	0.8085	53.40

TABLE 1. Predicted shelf life of choc	plate spread containing 3.5% SFW	/ and 3.5% CW at 5, 10 and 25 °C
---------------------------------------	----------------------------------	----------------------------------

SFW: Sunflower wax, CW: Carnauba wax, R2: R-squared,  $A_0$ : Initial hardness,  $A_e$ : Exceed hardness, k: Constant,  $t_E$ : Shelf life \* Based on unspreadable condition.

The hardness of chocolate spread with 3.5% SFW showed no clear trend at all storage temperatures. In general, the initial hardness of the sample (week 0) was higher at lower temperatures. For storage temperatures of 25, 40 and 45 °C, no significant changes (p > 0.05) were recorded from week 0 to week 24. The hardness of this chocolate spread during storage for 24 weeks at all experimental temperatures was lower than that of the unspreadable condition of commercial chocolate spread at 5 °C, with a value of 2105 g<sub>c</sub>

Similar to the hardness of chocolate spread with 3.5% SFW, the hardness of chocolate spread with 3.5% CW showed no clear trend at all storage temperatures. Generally, this hardness was not significant from week 0 to week 24 for temperatures of 25, 40 and 45 °C. Chocolate spread with 3.5% CW at the low temperature of 5 °C showed a significant change (p < 0.05) during the storage at week 0, although this chocolate spread showed a non-significant decrease (p > 0.05) starting at week 2 until week 18. The hardness for chocolate spread with 3.5% CW decreased slightly at week 20 and remained without significant differences (p > 0.05) until week 24. A similar explanation can be found for this storage trend when the oleogel re-arranged the molecular packing of its network, which produced structural changes. At 10 °C, there was a significant change (p < 0.05) in the hardness of the chocolate spread with 3.5% CW from week 0 to week 8. However, there was a non-significant increase (p > 0.05) starting at week 10 until the end of the storage test. In general, the hardness of the chocolate spread with 3.5% CW at temperatures of 5 and 10 °C at each week of storage was significant (p < 0.05) compared to the hardness at temperatures of 25, 40 and 45 °C at each week of storage. A study by Öğütcü and Yilmaz (2015) reported that the firmness of oleogels consisting of 3, 7 and 10% CW in hazelnut oil showed decreasing, increasing and fluctuating trends, respectively, during 90 days of storage at 4 °C. However, oleogels containing 3, 7 and 10% CW with virgin olive oil showed unchanged, fluctuating and fluctuating trends, respectively at 4 °C during 90 days of storage (Öğütcü and Yilmaz 2014). Similar to the chocolate spread containing 3.5% SFW, the hardness of chocolate spread with 3.5% CW at 25, 40 and 45 °C is less than 60  $g_f$  and can be considered as no hardness. The hardness of this chocolate spread at 24 weeks of storage, at all experimental temperatures was lower than that of the unspreadable condition of commercial chocolate spread at 5 °C with a value of 2105 g<sub>c</sub>

The potential shelf life of chocolate spreads with different oleogelators at 5, 10 and 25 °C was also calculated using kinetic modelling (Table 1) based on unacceptable commercial chocolate spread hardness of 2105  $g_f$  at 5 °C. The shelf life based on the unspreadable hardness of commercial chocolate spread was not conducted for 40 or 45 °C, as hardness was not deemed the appropriate quality parameter at these temperatures. The best reaction order was selected based on the R<sup>2</sup> value, which was close to 1. The shelf life of chocolate spread containing 3.5% SFW at 5, 10 and 25 °C based on hardness kinetic are 13.16 years, 6.32 years and 52.34 years with the best reaction order of 1, 1 and 0, respectively. This means that chocolate spread containing 3.5% SFW will be unspreadable only after 13.16 years when stored at 5 °C, unspreadable after 6.32 years when stored at 10

°C and unspreadable after 52.34 years when stored at 25 °C. The shelf life of chocolate spreads containing 3.5% CW at 5, 10 and 25 °C based on hardness kinetic were calculated to be 6.26 years, 4.23 years and 53.41 years with reaction order of 1, 1 and 0, respectively. Both of the chocolate spreads (containing 3.5% SFW and 3.5% CW) had similar reaction orders at 5, 10 and 25 °C.

#### 3.2. Spreadability of chocolate spread

Figures 3 and 4 show the spreadability of chocolate spreads with different oleogelators at a temperature of 5 °C (low temperature). The spreadability test was not performed at other experimental temperatures, namely at 10, 25, 40 or 45 °C, as chocolate spread at these temperatures are easily spreadable. According to Norazura Aila et al. (2023), significant changes were observed in the crystal structure of SFW oleogel when the percentage of wax increased to 3.5% from 0.5% at 10 °C, when the densest structure was found in the oleogel with 3.5% SFW. Based on this trend, 3.5% SFW oleogel at 5 °C will present a denser structure compared to that at 10 °C. Chocolate spreads containing 3.5% SFW and 3.5% CW at 5 °C showed solid behavior but they can still be spread within 30 seconds at all weeks of storage, from day

1 to week 24. By using similar chocolate spread formulation, the base oil of 50% POOo and 50% SFO without wax was able to be spread easily at 5 °C after 24 h of storage (Norazura Aila et al. 2022). This indicated that the base oil might interfere less with fat crystallization at 5 °C. Chocolate spread containing 3.5% SFW showed a rougher texture after storage for 4 weeks and above compared to day 1 and week 2. This might be due to aggregation and clustering among the microplatelet particles and consequently, reinforced structure of the oleogels over storage time as reported by Doan et al. (2016). The same trend can be observed for chocolate spread containing 3.5% CW in terms of texture at 5 °C. Upon cooling, the precipitation of oleogels resulted in the formation of solid nuclei (Doan et al. 2018) and subsequently in strong interactions of crystals as well as supramolecular entities that trap liquid oil in a three-dimensional network when the onset of crystal growth occurs (Yuping et al. 2005). Although the texture was slightly rougher, this chocolate spread can still be spreadable at 5 °C compared to the commercial chocolate spread.

#### 3.3. Stability of chocolate spread

Table 2 shows the stability of chocolate spreads with different oleogelators in a storage study of

Week/	25 °	С	40 °	С	45 °C	
Temperature and sample	3.5% SFW	3.5% CW	3.5% SFW	3.5% CW	3.5% SFW	3.5% CW
Week 0 (Day 1)	Stable	Stable	Stable	Stable	Stable	Stable
Week 2	Stable	Stable	Stable	Stable	Stable	Stable
Week 4	Stable	Stable	Stable	Stable	Stable	Stable
Week 6	Stable	Stable	Stable	Stable	Stable	Stable
Week 8	Stable	Stable	Stable	Stable	Stable	Stable
Week 10	Stable	Stable	Stable	Stable	Stable	Stable
Week 12	Stable	Stable	Stable	Stable	Stable	Stable
Week 14	Stable	Stable	Stable	Stable	Stable	<2.0%
Week 16	Stable	Stable	Stable	Stable	<2.0%	Stable
Week 18	Stable	Stable	<2.0%	Stable	<2.0%	Stable
Week 20	Stable	Stable	Stable	Stable	<2.0%	Stable
Week 22	Stable	Stable	Stable	Stable	Stable	Stable
Week 24	Stable	Stable	Stable	Stable	<5.0%	<2.0%

TABLE 2. Stability of chocolate spread with 3.5% SFW and 3.5% CW during the six-month storage study at 25, 40 and 45 °C

CW: Carnauba wax, SFW: Sunflower wax.

The results are expressed with n = 3.

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Week 0 (Day 1)



Week 2



Week 4

Week 6



Week 8



Week 10



Week 12



Week 14



Week 16



Week 18



Week 20



Week 22



Week 24

FIGURE 3. Spreadability of chocolate spread containing 3.5% sunflower wax (SFW) at 5°C during storage study for Week 0 - Week 24.







Week 6



Week 8



Week 2

Week 10



Week 12



Week 14



Week 16



Week 18



Week 20



Week 22



Week 24

FIGURE 4. Spreadability of chocolate spread containing 3.5% carnauba wax (CW) at 5°C during storage study for Week 0 - Week 24.



Week 0 (Day 1)

six months at temperatures of 25, 40 and 45 °C. Chocolate spreads with 3.5% SFW and 3.5% CW were stable for six months at 25 °C. This showed that 3.5% SFW and 3.5% CW can hold oil at 25 °C for six months and were able to prevent phase separation. Figure 5a is an example of a graph that has a stable chocolate spread. No peak was present throughout the graph, indicating that there was no oil transmission signal nor was phase separation found. The high peak at the beginning of the graph indicated the beginning of the measurement. At 40 °C, chocolate spreads with 3.5% SFW and 3.5% CW were found to be stable for six months with only a slight phase separation of less than 2.0% at week 18 for chocolate spread with 3.5% SFW. Chocolate spread with 3.5% CW that was stable at 40 °C at week 22 is shown in Figure 5a. Patel et al. (2014) reported that chocolate spread with shellac wax to replace all existing oil binders and 27% palm oil (PO) showed no phase separation at 30 °C for 30 days. This showed that chocolate spreads with 3.5% SFW and 3.5% CW were more stable than chocolate spread with shellac wax at a higher temperature and centrifugal speed.

At a temperature of 45 °C, chocolate spread containing 3.5% SFW started to experience continuous oil separation from week 16, which was after 3.75 months. This phase separation can be measured by observing whether there is a peak along the transmission graph by not including the initial peak. The percentage of transmission (oiling out) can be measured based on the area of the peak. Nevertheless, the transmission peak along the total length of the sample is not large. The maximum amount of oil detected was less than 5% at week 24 (Figure 5b). Fayaz *et al.* (2017) reported that their chocolate spread containing 5% beeswax, propolis wax and monoglyceride with pomegranate oil was stable at 30 days of storage with less than 6% oiling out at 20 °C at 10000 rpm centrifuge for 15 min. Oiling out indicates the separation of the liquid phase from the solid phase of the chocolate spread. Chocolate spread with 3.5% CW was relatively stable, with the only oiling out observed at week 24 at a value of less than 2.0%. This showed that chocolate spread with 3.5% CW was more stable, as oiling out occurred in the last week of the storage study at week 24.

#### 3.4. Sensory evaluation of chocolate spread

The results from the sensory evaluation of chocolate spreads with different oleogelators (freshly prepared sample (day 7)) as well as commercial chocolate spread at temperature of 25 °C are shown in Figure 6. Chocolate spreads with 3.5% CW and 3.5% SFW were newly produced samples. The sensory attributes evaluated were glossiness, spreadability, color, smoothness, sweetness, oily taste, and overall acceptability using a 7-point hedonic scale. Statistically, glossiness, spreadability, color and smoothness attributes were similar (p > 0.05) to one another with scores ranging from 3.23–4.27, 5.50–6.10, 4.63–5.47 and 4.50–4.93, respectively. The score for the oily taste was the highest for chocolate



FIGURE 5. Example of a) stable chocolate spread (chocolate spread containing 3.5% carnauba wax (CW), 40 °C, week 22 and b) unstable chocolate spread (chocolate spread containing 3.5% sunflower wax (SFW), 45 °C, week 24).

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FIGURE 6. Sensory mean scores for glossiness, spreadability, color, smoothness, sweetness, oily taste and overall acceptability of chocolate spread containing 3.5% carnauba wax (CW) and 3.5% sunflower wax (SFW) compared to commercial chocolate spread at 25 °C using the 7-point hedonic scale. The results are expressed with n = 30

spread with 3.5% SFW, with a score value of 4.63 or equivalent to 66.1%.

Statistically, commercial chocolate spread and chocolate spread containing 3.5% CW received similar scores for overall acceptability with values of 5.50 (78.6%) and 4.80 (68.6%), respectively. Therefore, chocolate spread containing 3.5% CW was acceptable and comparable to commercial chocolate spread without wax. In term of sweetness, sensory panelists preferred commercial products compared to chocolate spread with oleogels. The commercial product contains 37% sugar while chocolate spread with oleogels contains 38.9% sugar. The color of chocolate spread with oleogels were preferred compared to commercial chocolate spread due to its lighter color. Sensory panelists preferred the glossiness of chocolate spread with oleogels as the waxes enhance glossiness compared to the dull surface of commercial chocolate spread. No sensory evaluation was found for chocolate spread, chocolate paste or chocolate filling for studies using wax oleogel. However, in general, products that use oleogel have several shortcomings related to the taste of the product, especially the waxy taste (Abdollahi et al. 2020). A waxy taste was also reported by Yılmaz and Öğütcü (2015) for their fat spread products that used wax oleogel. However, Blake and Marangoni (2015) found that their final product, a bakery product such as croissant using fat containing oleogel with 7.5% rice bran wax did not have a waxy taste. Chocolate spread with 3.5% CW statistically had the same oiliness and mouthfeel (p > 0.05) compared to commercial chocolate spread. This indicates that the amount of wax used does not impart a prominent waxy taste. This result will probably give an advantage to chocolate spread with 3.5% CW in terms of commercial value.

#### 4. CONCLUSIONS

Low temperatures of 5 and 10 °C affected the hardness of the chocolate spreads with 3.5% SFW and 3.5% CW from day 1 (week 0) to week 24 of storage. However, the hardness of the chocolate spreads at temperatures of 25, 40 and 45 °C in weeks 0-24 were not affected by temperature or time (p >0.05). Both chocolate spreads with 3.5% SFW and 3.5% CW can be applied at 5 °C during the storage period (week 0 - week 24). These chocolate spreads were stable throughout the storage study from week 1 – week 24 at 25 °C. Chocolate spreads with 3.5% SFW and 3.5% CW were also stable at 40 °C throughout the storage study. However, at 45 °C, chocolate spread with 3.5% SFW showed early phase separation compared to chocolate spread with 3.5% CW. Therefore, it can be concluded that chocolate spread with 3.5% CW was more stable than chocolate spread with 3.5% SFW. The sensory evaluation also showed that chocolate spread with 3.5% CW was more acceptable by sensory panelists compared to chocolate spread with 3.5% SFW.

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# DECLARATION OF COMPETING INTEREST

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# **AUTHORSHIP CONTRIBUTION STATEMENT**

N.A. Mohd Hassim: Conceptualization, Formal Analysis, Funding acquisition, Investigation, Methodology, Project Administration, Writing - original draft, Writing – review & editing. S. Kanagaratnam: Conceptualization, Methodology, Writing – review & editing. T.K. Tang: Writing – review & editing. N.S. Sofian Seng: Conceptualization, Methodology, Writing – review & editing.

# REFERENCES

- Abdollahi M, Hossein Goli SA, Soltanizadeh N. 2020. Physicochemical properties of foamtemplated oleogel based on gelation and xanthan gum. *EJLST* **122**, 1900196. https://doi. org/10.1002/ejlt.201900196
- Bascuas S, Espert M, Llorca E, Quiles A, Salvador A, Hernando, I. 2021. Structural and sensory studies on chocolate spreads with hydrocolloid-based oleogels as a fat alternative. *LWT – Food Sci Technol* 135, 110228. https://doi.org/10.1016/j. lwt.2020.110228
- Blake AI, Marangoni AG. 2015. Factors affecting the rheological properties of a structured cellular solid used as a fat mimetic. *Food Res. Int.* **74**, 284e293. https://doi.org/10.1016/j.foodres.2015.04.045

- Codex A. 2024. GSFA Online. Carnauba wax. https://www.fao.org/gsfaonline/additives/details. html?id=264
- Doan CD, Tavernier I, Sintang MDB, Danthine S, Van de Walle D, Rimaux T, Dewettinck K. 2016. Crystallization and gelation behavior of low- and high melting waxes in rice bran oil: A case-study on berry wax and sunflower wax. *Food Biophys.* 12, 97–108. https://doi.org/10.1007/s11483-016-9467-y
- Doan CD, Tavernier I, Okuro PK, Dewettinck K. 2018. Internal and external factors affecting the crystallization, gelation and applicability of waxbased oleogels in food industry. *Innov. Food Sci. Emerg. Technol.* **45**, 42–52. http://doi. org/10.1016/j.ifset.2017.09.023
- Doan CD, To CM, De Vrieze M, Lynen F, Danthine S, Brown A, Dewettinck K, Patel AR. 2017. Chemical profiling of the major components in natural waxes to elucidate their role in liquid oil structuring. *Food Chem.* 214, 717–725. https:// doi.org/10.1016/j.foodchem.2016.07.123
- Endlein E, Peleikis KH. 2011. Natural waxes -Properties, composition and applications. *SOFW J.* **137**, 1–8.
- Fayaz G, Goli SAH, Kadivar, M. 2017. A novel propolis wax-based organogel: Effect of oil type on its formation, crystal structure and thermal properties. J. Am. Oil Chem. Soc. 94, 47–55. https://doi.org/10.1007/s11746-016-2915-5
- Handiati YD, Praseptiangga D, Manuhara GJ, Khasanah LU. 2019. Effects of Kaempferia galanga L. essential oil incorporation on sensory and physical properties of dark chocolate bar. *IOP Conf. Ser. Mater. Sci. Eng.* 633, 012036. https:// doi.org/10.1088/1757-899x/633/1/012036
- Manzocco L, Valoppi F, Calligaris S, Andreatta F, Spilimbergo S, Nicoli, MC. 2017. Exploitation of k-carrageenan aerogels as template for edible oleogel preparation. *Food Hydrocoll.* **71**, 68–75. https://doi.org/10.1016/j.foodhyd.2017.04.021
- Norazura Aila MH, Sivaruby K, Nur Haqim I, Noor Lida H; Wan Rosnani, AI, Noor Soffalina SS. 2022. Palm-based chocolate spread for wide range temperature applications using sunflower wax, carnauba wax and bees wax. *J. Oil Palm Res.* 34, 535–545. https://doi.org/10.21894/ jopr.2021.0051

- Norazura Aila MH, Sivaruby K, Saw MH, Nur Haqim I, Rafidah AH, Muhammad Hanif M, Noor-Soffalina SS. 2023. Characteristics of sunflower wax, carnauba wax and beeswax in palm superolein blended oil. J. Oil Palm Res. 35, 694– 712. https://doi.org/10.21894/jopr.2022.0055
- Öğütcü M, Yilmaz E. 2014. Oleogels of virgin olive oil with carnauba wax and monoglyceride as spreadable products. *Grasas Aceites* **65**, 1–11. https://doi.org/10.3989/gya.0349141
- Öğütcü M, Yilmaz, E. 2015. Characterization of hazelnut oil oleogels prepared with sunflower and carnauba waxes. *Int. J. Food Prop.* **18**, 1741– 1755. https://doi.org/10.1080/10942912.2014.93 3352
- Patel AR, Rajarethinem PS, Grędowska A, Turhan O, Lesaffer A, De Vos WH, Van de Walle D, Dewettinck K. 2014. Edible application of shellac oleogels: Spreads, chocolate, cakes and pastes. *Food Funct.* 5, 645–652. https://doi.org/10.1039/ C4FO00034J
- Rocha JCB, Lopes JD, Mascarenhas MCN, Arellano DB, Guerreiro LMR, da Cunha RL. 2013. Thermal and rheological properties of organogels formed by sugarcane or candelilla wax in soybean oil. *Food Res. Int.*, **50**: 318-323. https://doi.org/10.1016/j.foodres.2012.10.043
- Said A, Atikah N, Nasir M, Abdulla C, Bakar A, Anwar W, Wan F. 2019. Chocolate spread emulsion: Effects of varying oil types on physico-

chemical properties, sensory qualities and storage stability. *J. Agribiotechnol.* **10**, 32–42.

- Shamsudin SY. 2004. *Trans* free palm based chocolate spread. MPOB TT. No. 251. *MPOB Information Series*. ISSN 1511-7871.
- Sivaruby K, Enamul HM, Miskandar MS, Spowage A. 2013. Investigating the effect of deforming temperature on the oil-binding capacity of palm oil-based shortening. *J. Food Eng.* **118**, 90–99. https://doi.org/10.1016/j.jfoodeng.2013.03.021
- Toro-Vazquez JF, Morales-Rueda JA, Dibildox-Alvarado E, Charó-Alonso M, Alonzo-Macias M, González-Chávez M. 2007. Thermal and textural properties of organogels developed by candelilla wax in safflower oil. J. Am. Oil Chem. Soc 84, 989–1000. https://doi.org/10.1007/s11746-007-1139-0
- Wolfer TL, Acevedo NC, Prusa KJ, Sebranek JG, Tart R. 2018. Replacement of pork fat in frankfurtertype sausages by soybean oil oleogels structured with rice bran wax. *Meat Sci.* 145, 352–62. https:// doi.org/10.1016/j.meatsci.2018.07.012
- Yılmaz E, Öğütcü M. 2015. Oleogels as spreadable fat and butter alternatives: Sensory description and consumer perception. *RSC Adv.* 5, 50259– 50267. https://doi.org/10.1039/C5RA06689A
- Yuping SHI, Baomin L, Richard H. 2005. Crystal morphology, microstructure, and textural properties of model lipid systems. *JAOCS* 82, 399–408. http://doi.org/10.1007/s11746-005-1084-3