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Thermo-Physical Properties of Fats and Oils

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***Abstract*— Thermo-physical properties of butter, hydrogenated fat, palm oil, coconut oil, sunflower oil, and groundnut oil were examined. Experimental results revealed the highest slip melting point (38oC) and specific gravity for hydrogenated fat whereas the lowest slip melting point (-18oC) and hardness values for sunflower oil. However, sunflower oil showed the highest refractive index. The consistency of butter and hydrogenated fat was reported acceptable plastic and spreadable. Palm oil was found soft and spreadable. The melting curve of palm oil, butter, and hydrogenated fat showed two endothermic peaks. However, coconut oil, sunflower oil, and groundnut oil showed only one peak. The solid fat content (SFC) of butter, hydrogenated fat, palm oil, and coconut oil were found within the recommended range for baking performance. Melting enthalpy was strongly correlated to hardness ($r = 0.8604$, $P < 0.001$) of fat and oil samples. Correlation coefficient (r) of 0.8977 ($P < 0.001$) was found between SFC and hardness. The results strengthen the premise that hardness of the fat is not dependent solely on the SFC and as the results demonstrated that the melting enthalpy also plays a key role in influencing the hardness of fats and oils.**

***Index Terms*— Fats and oils, SFC, hardness, melting enthalpy, thermal properties, physical properties.**

I. INTRODUCTION

Fats and oils are widely used food components. They are the indispensable ingredients in baked products and influence their microstructure and physical properties. The precise physical and thermal properties of the fat are crucial in baking because of the fact that fats are directly incorporated into the dough. The quality and physical properties of many food products tenaciously rely on upon the physical properties and thermal behavior of fats and oils. Fats and oils in baking contribute to product characteristics such as tenderness, moisture, mouthfeel, lubricity, flavor, structure, and shelf life. Shortenings which are often produced by partial hydrogenation of oils are commonly used in the baking industry [1]. However, during partial hydrogenation of oil, trans fatty acids are formed and research has proved the direct connection of trans fatty acids with many health problems [2].

Physical properties of fats and oils are amongst the most imperative properties that specify freshness and quality of the fats and oils as well as their functionality in food products. The melting profile of fats and oils influences the incorporation of air, rheology, mouthfeel, shelf life and other quality parameters of food products, especially in baked products.

Fats and oils are multicomponent mixes of triacylglycerols (TAGs) with specific properties that are considered significant in foods. The most noteworthy attributes of physical properties of fats and oils are associated with the solid-liquid phase change; i.e. melting point as well as the proportion of solid content in fats and oils at a particular temperature. Onset and offset melting temperature, melting range, peak melting point, melting enthalpy and solid fat content. These parameters of fats and oils can be studied by Differential Scanning Calorimetry (DSC). These properties are essential to understanding the functionality of fats and oils in the food products. In general, fats and oils can exhibit tremendously complex thermal behavior depending on the chemical composition and protocol for the DSC experiment [3]. According to Tan and Che Man [3] as the thermal profile of fats and oils endow with large information about the nature of transition and so can be used as a significant probe for "fat and oil". The objective of this study was therefore to investigate thermo-physical properties of fats and oils used for food products.

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II. METHODOLOGY

Six different types of edible fat and oil samples were examined both from plant and animal origin. Palm oil was obtained from Ruchi Soya Industries Ltd., Gurgaon. Butter (Amul), hydrogenated fat (Dalda), sunflower oil (Nature Fresh), groundnut oil (Dhara), and coconut oil (Parachute) were procured from local retailers.

A. Physical Properties

Density was determined using a 25 ml pycnometer, previously calibrated with distilled water, following the standard method of AOAC (2000) and specific gravity was calculated by the formula: Specific gravity = Density of oil/Density of water.

The viscosity measurement was carried out by Brookfield Viscometer (RV Model). The spindle was selected such that during measurements the torque was between 10 to 100%. The temperature of samples was maintained at 25±1˚C throughout the test. Refractive index was determined using an Abbe Refractometer and white light according to AOAC Standard Method (2000). The slip melting point (SMP) was measured according to the Official Method of AOCS (1989). A column of fat was tempered at 10±1˚C for 24 h in an open capillary tube. The tube was then heated slowly in a water bath until the fat column started to rise due to hydrostatic pressure. The temperature at which this occurs was determined as SMP using averaging of three replicates.

Consistency was measured by Texture Analyzer TA-XT2 (Stable MicroSystems), controlled by a computer. Before measurements samples were melted and tempered for 24 hours at room temperature (25 ^{o}C). The compression values were converted to yield value, in accord to Haighton [4].

B. Thermal properties

The heating thermogram was determined by differential scanning calorimetry (DSC) according to the AOCS (2004). A Perkin-Elmer Model DSC 4000 equipped with a liquid nitrogen-cooling unit was used for the thermal analysis of the samples. The data processing system used was the Pyris Series Thermal Analysis System software. About 6 mg of sample was placed in a small aluminum sample pan. The pan was then placed on the sample platform of the DSC. An empty aluminum pan was placed on the reference platform. For determination of the melting point a linear heating rate of 5 ^{o}C/min over a temperature range of -30 ^{o}C to 80 ^{o}C was used and holding time at both extreme temperatures were 10 min. The following parameters for result assessment were used: onset and offset melting temperature (T_{on} and T_{off}), temperature range (T_r), peak melting temperature (T_p), and melting enthalpies (ΔH_m). The SFC was obtained from the melting curve. The SFC as a function of temperature was calculated from the partial areas at different temperatures (−20 °C to 40 °C, intervals of 5 °C).

B. Texture Analysis

The texture profile analysis of the crystallized samples was measured using a double compression test with a Texture Analyser TA-XT2 (Stable MicroSystems) as measured by Kanagaratnam et al. [5]. Before analysis, samples (30 ml) were melted at 70 °C for 10min, followed by chilling at −20 °C for 90 min, and then tempered at 5 ^{o}C and 20 °C for 24 h. The hardness of samples was determined with a cylindrical probe with a diameter of 6 mm and cell load of 5 kg. The probe penetrated the sample at a constant, optimized pre-test speed of 1 mm/min; test speed of 10 mm/min and post-test speed of 2 mm/min to a distance of 12 mm. The hardness of the shortening was indicated by the maximum force detected during compression. The measurements were performed in triplicate for each sample.

C. Statistical analysis

Both the means and standard deviations (SD) were calculated using the SPSS (version 16.0) statistical software. The one-way analysis of variance (ANOVA), with the Tukey's multiple comparisons, was performed to test the significance.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Physical properties

The fats and oils do not exhibit sharp melting point as these are blends of triglycerides and each triglyceride has its own melting point, yet slip melting point (SMP) provides the information about the melting point. The SMP is defined as the temperature at which a sample rises in an open capillary upon heating under defined conditions. **Table 1** shows the physical properties of fats and oils. The sunflower oil showed the lowest SMP at a negative scale of -18 ^{o}C because of its higher percentage of unsaturated fatty acids. Hydrogenated fat had the highest SMP of 38 ^{o}C. Palm oil had SMP of 32 ^{o}C in between the range of butter (35 ^{o}C) and hydrogenated fat (38 ^{o}C). Refractive index of sunflower oil was maximum which indicates it's most unsaturated nature and the refractive index of coconut oil was least which showed its most saturated nature of the fatty acid composition. Refractive index of hydrogenated fat and palm oil were same which implies their similar proportion of unsaturation. The specific gravity of hydrogenated fat was maximum while specific gravity of palm oil butter was least which reflects that molecules of hydrogenated fat were more closely packed and molecules of palm oil and butter were loosely packed.

TABLE 1
PHYSICAL PROPERTIES OF FATS AND OILS

Sample	SMP (^{o}C)	RI	SG (g/cm$^{3)}$	V (cP)	CN (g/cm^{2})
SO	-18^{d}±0.6	1.348^{b} ±0.2	0.918^{a}±0.4	48.78^{a}±0.9	40.80^{c}±1.9
GO	2.5^{a}±0.9	1.343 ^{c} ±0.1	0.915^{a}±0.2	57.00^{b} ±0.8	44.24^{b}±2.7
CO	24 ^{d} ±0.6	1.330^{d} ±0.1	0.918 ^{c} ±0.3	42.81^{b} ±0.6	38.41^{a}±1.2
PO	32 ^{c}±0.5	1.339 ^{a} ±0.2	0.911^{b} ±0.5	81.00 ^{c} ±0.6	150.03^{a}±4.3
B	35 ^{d} ±0.6	1.334^{d} ±0.1	0.911^{d} ±0.5	81.06 ^{c} ±0.7	366.73^{b}±5.6
HF	38 ^{b}±0.5	1.339^{b} ±0.1	0.922^{d} ±0.4	82.00^{d} ±0.8	291.71^{d}±5.3

Mean* ±S. D.
*Mean of triplicate determinations.
$^{a-d}$Values in the same column followed by different letters are significantly different (P < 0.05).

SMP- Slip melting point; RI- Refractive index; SG- Specific gravity; V- Viscosity; CN- Consistency
SO-Sunflower oil; GO-Groundnut oil; CO-Coconut oil; PO-Palm oil; B-Butter; HF-Hydrogenated fat.

Viscosity values estimate oil's relative thickness or resistance to flow. It may be considered the integral of the interaction forces between molecules. A high viscosity means that the liquid will not flow easily and vice versa. As expected, the viscosity of hydrogenated fat was maximum among semi-solid fats. There was no significant difference between viscosity values of butter and palm oil. But among oils unexpectedly viscosity of coconut oil was least. It might be because of the unique chemical composition of coconut oil. It is composed of medium chain triglycerides (MCTs) while generally fats and oils consist of long-chain triglycerides (LCTs). MCTs have a lower molecular weight than conventional oils. This gives MCTs a lower viscosity than conventional oils [6]. The statistics presented in Table 1 shows the yield values of fats and oils. 'Yield value' is the predominantly used criterion to predict the consistency of fats. The consistency of fats is a necessary aspect of baked products. It denotes those features of the food texture that refer to flow and deformation. The highest yield value referred to butter (366.73 g/cm^2), followed by hydrogenated fat (291.71 g/cm^2), and palm oil (150.03 g/cm^2). No considerable difference was noted among the consistency of SO, GO, and CO oils. According to Haighton's [4] classification, the yield value of fats between 200-800 g/cm^2 was categorized as plastic fats that can be spreadable. Since the butter and hydrogenated fat of this study had yield values between this ranges, they also could be classified as plastic and best consistency for excellent spreadable products. Palm oil also lies into the soft and spreadable category. Consistency data showed that sunflower oil, groundnut oil, and coconut oil were very soft, just pourable, and not shape retaining.

A. Thermal Properties

Differential Scanning Calorimetry (DSC) is the most used thermoanalytical technique for the study of thermal behavior of fats and oils. DSC is a technique that is used to measure the function of the differential heat flow with temperature for the compounds showing thermal transitions such as melting and crystallization [7]. Evaluation by DSC bestows direct measures of the energy involved in the melting process of fats and oils. Fat melting, contributes to volume expansion, characterizing an endothermic effect. The peak is an interpretation of change in the differential heat flow, which is caused by the changes in the samples associated with absorption or evolution of heat.

DSC-melting curves of fat and oil samples are displayed in **Fig. 1.** The peak melting temperatures are mentioned in **Table 2**. The melting behavior of all samples was monitored on heating at 5 °C/min. The peak melting temperatures ranged from -19.39 °C to 35.01 °C, with the butter being the highest. The DSC melting curve recorded for butter, palm oil, and hydrogenated fat showed well distinguishable two endothermic events. According to Kaisersberger [8] melting range and DSC curve shape result from combined effects of fatty acid composition, polymorphism and thermal history.

Melting curves were broad and overlapped. The melting peaks were multiple and irregular with shoulder, fusion and merging peaks along with one or two major endotherms. Fredrick et al. [9] stated that the melting profiles provided an indication of the polymorphic transitions. Garti et al. [10] reported that the first peak of heating thermogram represented to melting of the α form, intermediate peak corresponded to the melting of the β′ form and the last peak represented the melting of the β form.

TABLE 2
MELTING PROFILE OF FATS AND OILS

Sample	T_o (°C)	T_f (°C)	T_r (°C)	T_p (°C)	ΔH_m (J/g)
SO	-27.77	-5.39	22.38	-19.39	9.24
GO	-27.32	7.84	35.16	0.54	38.13
CO	10.19	32.15	21.96	26.75	239.45
PO	-10.51	36.31	46.82	28.07	12.30
B	-25.82	38.05	63.87	35.01	36.40
HF	-15.13	40.70	55.83	34.66	65.98

T_o- Onset temperature; T_f- Offset temperature;
T_r- Range of temperatures; T_p- Peak temperature;
ΔH_m- Melting enthalpy; SO- Sunflower oil;
GO- Groundnut oil; CO-Coconut oil; PO-Palm oil;
B-Butter; HF- Hydrogenated fat.

Fig. 1 demonstrated that the endothermic peak of coconut oil was the sharpest and is located at a higher temperature, indicating a small melting range. The melting curve of coconut oil has single tall endotherm peak without any shoulder peak whereas all other fats and oils samples showed a distinct tall endotherm peak with small shoulder peaks. The melting peaks of the solid and semi-solid samples (butter, hydrogenated fat, and palm oil) were wider than those of the liquid samples (groundnut oil, coconut oil, and sunflower oil). There is a broadening of the curves in hydrogenated fat and moving to higher temperatures regions. Formation of trans and positional isomers due to hydrogenation resulted in the formation of non-intersoluble triglycerides, increasing the melting range of the samples. Palm oil had a very characteristic curve, showing clearly its two component fractions, separated by an exothermic peak; these are the low-temperature endotherm (olein fraction) and the high-temperature endotherm (stearin fraction). The endotherm region at lower temperature contained two small fusion peaks. Butter also showed two peaks. It is important to note that coconut oil, groundnut oil, and sunflower oil had a beta (β) crystal forming tendency. Beta crystals are most stable hence these oils showed only one endothermic event. Whereas, palm oil and butter have a beta prime (β′) crystal forming tendency which is less stable and finally leads to stabilize into beta type (β) crystals as temperature increases.

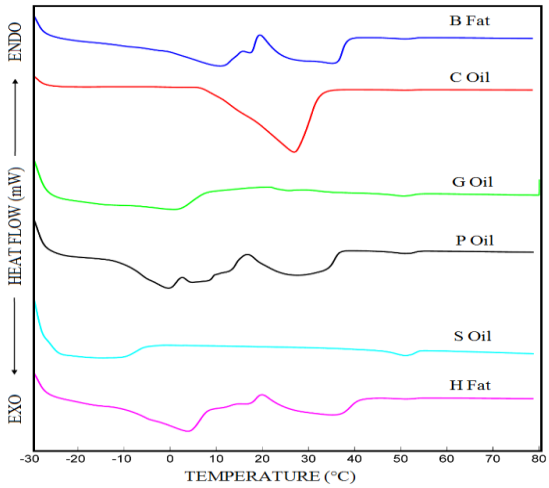


Fig. 1 Typical DSC thermograms of fats and oils. Butter (B Fat), Coconut oil (C Oil), Groundnut oil (G-Oil), Palm oil (P Oil), Sunflower oil (S-Oil) and Hydrogenated fat (H Fat)

Generally, thermal characterization of fats and oils can be explained by different transitions temperatures obtained from DSC. Five DSC parameters, namely onset temperature (T_o), offset temperature (T_f), temperature range (T_r) (the difference between T_o and T_f), peak temperature and melting enthalpy (ΔH_m) were established for the major thermal curve in all fat and oil samples. Complete comparisons of these five DSC parameters are summarized in **Table 2** for melting curves.

The sample with the lowest peak points was sunflower oil (-19.39 °C) while butter had the highest (35.01 °C). The ‘‘onset’’ temperature is a typical parameter, which represents the beginning of the melting process defined as the intersection of the tangent at the first leg of the main peak with the base line. Integration of the total peak area is used to determine enthalpy. Melting enthalpies (ΔH_m) were found to be significantly different from each other. The data obtained showed that ΔH_m was the least for sunflower oil (9.24 J/g), followed by palm oil (12.30 J/g), butter (36.40 J/g), groundnut oil (38.13J/g), hydrogenated fat (65.98J/g). ΔH_m was notably high for coconut oil (239.45J/g). These results were expected and in accordance with Dian et al. [11]. The highest ΔH_m observed in coconut oil may be probably attributed to the high portion of medium-chain saturated fatty acids. The ΔH_m in sunflower oil was the lowest as very little energy was needed to melt the liquid oil, attributed the presence of high unsaturated fatty acid (UFA) compositional nature. Dian et al. [11] investigated that unsaturation of fatty acids ultimately related to fat’s softness. And fat’s softness affects textural characteristics of the final product.

DSC is an easy and fast technique, which is highly practical and useful for determination of Solid Fat Content (SFC). At the application temperature, SFC of fats can be determined from the DSC melting curves by partial integration. Partial areas are obtained according to the procedures described by Menard and Sichina [12]. The functional performance and textural quality of fats and oils are estimated mainly by the proportion of solid and liquid contents. It is generally accepted that the partial area under the melting peak (endothermic event) is equivalent to the percentage of solid fat remaining at the selected temperature [13]. The ratio of solid to liquid fat; that is, the amount of fat that is crystallized, was calculated using the DSC technique at 10, 15, 20, 25, 30 and 35 °C. The SFC of butter, hydrogenated fat, coconut oil, and palm oil at different temperatures calculated from the DSC data are given in **Fig. 2.** SFC of sunflower oil and groundnut oil could not be estimated at room temperature (25 °C) because of their very low melting point.

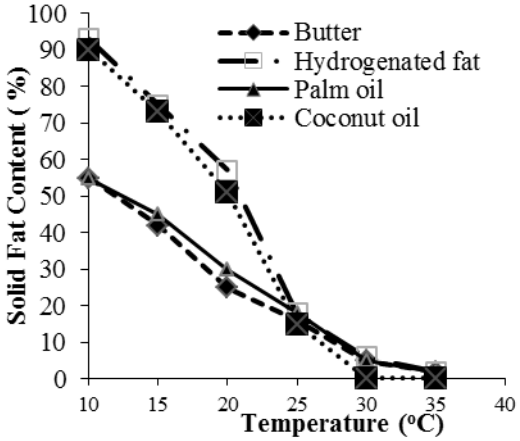


Fig. 2 Solid fat content (SFC) of fats and oils

Woerfel [14] reported that the solid profile has a good relation with hardness characteristics of plastic fats and influences the structure of the final product. The value of percentage solid up to 25 °C represents the hardness of a specific fat while between 25 °C to 33 °C demonstrates the flavor releasing potential during melting. The difference in SFC between 25 °C and 35 °C explains heat tolerance power of fats and oils while SFC at 35 °C determines mouthfeel [15]. Low SFC at all temperatures shows plastic behavior. SFC in ranges of 15–25% at usage temperature (23 °C) is recommended for shortening to have excellent baking performance [16]. SFC at room temperature (25 °C) should be 15−35% for desirable spreadability as plastic fats [17].

B. Texture Analysis

Texture analysis is primarily concerned with the mechanical properties, as they relate to its sensory properties detected by humans. Texture analyzer performs this test by applying a controlled force to the product and recording its response in the form of force, deformation and time. Hardness is one of the most important macrostructural properties of fat systems and is widely used to characterize their functionality.

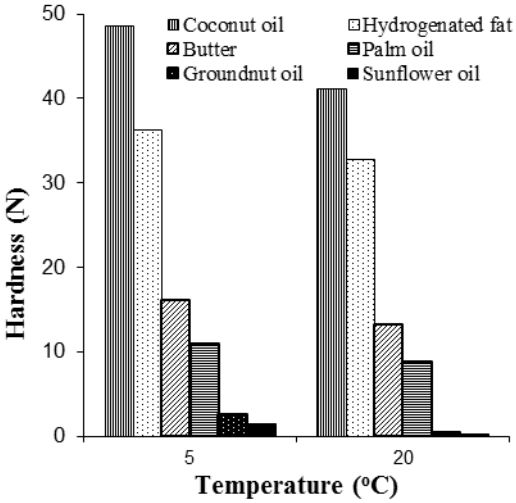
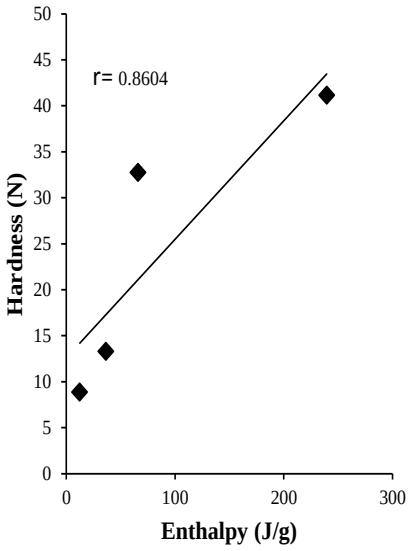


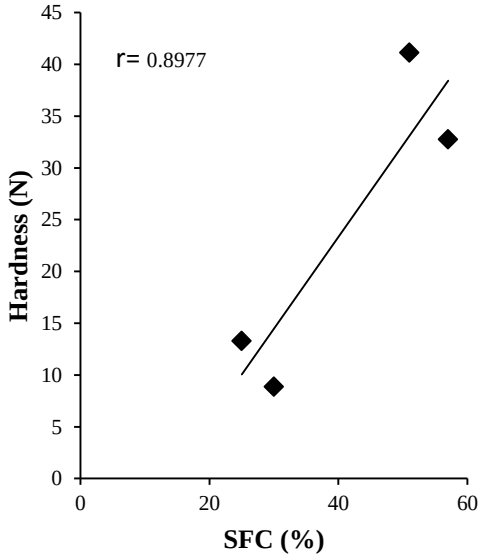
Fig. 3 Texture profile for fats and oils at 5°C and 20°C

The hardness of a fat is a critical parameter that vigorously impacts the perceived texture of a food product [19]. **Fig. 3** shows the hardness of fats and oils at 5 °C and 20 °C. Hardness (after 30 min storage at 5 °C and without storage at 20 °C) is expressed as the maximum penetration force. Hardness at an ambient temperature indicates the handling property of the fat at that particular temperature. These values showed a general decrease with increasing temperature for all samples. This is an expected result because higher temperatures are associated with lower degrees of crystallization and, therefore, softer materials. But the unexpected result is of coconut oil. Although coconut is oil but its hardness was more than the palm oil, butter, and hydrogenated fat at 5 °C and 20 °C temperatures. All other fats and oils are semi-solid at room temperature. It is because of the higher percentage of saturated fatty acid proportion in the case of coconut oil which allows it to solidify easily and do not allow melting earlier. It is in agreement with DSC thermograms results which showed the highest melting enthalpy and SFC for coconut oil up to 20 °C. Coconut oil is composed predominately of medium-chain triglycerides (MCTs) which make it unique. The hardness of sunflower oil and groundnut was measured at 5 °C after 72 h storage time because of low melting points. It was observed that at the same storage and temperature conditions groundnut oil had more hardness than sunflower oil.

Table 2 and **Fig. 3** reveals that hardness and melting enthalpy decreased in the order of coconut oil, hydrogenated fat, butter, and palm oil. However, **Fig. 2** shows that SFC decreased in the order of hydrogenated fat, coconut oil, palm oil, and butter. Butter had less SFC than palm oil at 20 °C while its hardness was more as compared to palm oil at the same temperature. It can be concluded that SFC is not the only property that determines the hardness of fats. The results are in agreement with Graef et al. [20] concluded that hardness of the fat is not dependent solely on the SFC.



(a)



(b)

Fig. 4 Effect of melting enthalpy (a) and solid fat content (b) on hardness of fats and oils

From **(Fig. 4a)** and **(4b),** it can be concluded that hardness of fats and oils is significantly affected by melting enthalpy and SFC. The correlation of hardness with melting enthalpy of fats and oils has not been reported in the literature so far to the best of our knowledge. At 20 °C temperature, correlation (r) between hardness and SFC was 0.8977 and correlation between hardness and enthalpy was 0.8604.

IV. CONCLUSIONS

The thermo-physical properties of butter, coconut oil, sunflower oil, groundnut oil, palm oil, and hydrogenated fat were examined. Coconut oil had the highest melting enthalpy and hardness as compared to other fats and oils. Moreover, the refractive index for sunflower oil was found the highest. As expected, maximum slip melting point was observed for hydrogenated fat. Consistency value of sunflower oil, groundnut oil, and coconut oil was observed just pourable. Whereas butter, hydrogenated fat, and palm oil were found spreadable. In addition, the melting curve of palm oil, butter, and hydrogenated fat show two endothermic peaks. Furthermore, SFC range for coconut oil, butter, palm oil, and hydrogenated fat was found between 15-20% at room temperature (25 °C). This fulfills one of the important requirements for baking performance. It is worth mentioning that the high correlation of melting enthalpy with hardness strengthens the premise that hardness of the fat is not dependent solely on the SFC. Results showed that the melting enthalpy also plays a key role in determining the hardness of fat and oil products. These findings have important implications for the handling and prediction of the end quality effects of fats and oils in food products.

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