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SOLID FAT CONTENT MEASUREMENT AS A POSSIBLE MEANS OF DETECTING ADULTERATION IN PALM OIL*

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RINGKASAN

Sifat pencairan dari minyak kelapa sawit dan campuran 'stearin' dengan minyak kelapa sawit yang mempunyai berbagai-bagai kandungan telah dianalisa berdasarkan kandungan Lemak Pejalnya sepanjang suhu 5°C sehingga 55°C dengan menggunakan 'pulsed NMR spectrometer'.

Hasil percubaan menunjukkan bahawa semua mempunyai sifat yang sama kecuali apabila stearin dimasokkan ke dalam minyak kelapa sawit telah meninggikan nilai kandungan Lemak Pejalnya di sepanjang suhu yang dikaji. Kesan ini adalah lebih besar untuk stearin keras dibandingkan dengan stearin lembut.

Ini mungkin satu cara yang baik untuk mengesan 'adulteration' dalam minyak kelapa sawit.

INTRODUCTION

Palm Oil is traditionally traded on the basis of its free fatty acids, water and dirt contents (HARTLEY, 1977). There are occasions when the oils received are exceptionally hard even if the various characteristics fall within the acceptable values and these lead to the suspicion of either (1) stearin being added intentionally (adulteration) or (2) unloading incompletely melted palm oil which has 'fractionated' during shipment. The palm oil industry is becoming more concerned with this problem of adulteration (BERGER, 1979). Recently the possibility of using SFC measurement to estimate the stearin content of palm oil has been discussed (NEWPORT INSTRUMENT, 1979). The work described in this paper is to suggest a possible means of detecting such adulteration by solid fat content (SFC) measurement and the melting behaviour of the oil.

EXPERIMENTAL

Special prime bleach (SPB) palm oil was used in this study. Two stearin fractions were obtained by solvent fractionation. 160 ml of acetone was added to 20 g of molten palm oil in a 250 ml flask. The solution was homogenised and kept in a 40°C water bath for 30 min before being cooled at 0.5°C/min to 22°C and left overnight at that temperature. A hard stearin fraction was obtained upon separation. To obtain the soft stearin, the homogenised solution was similarly treated but cooled at 0.5°C/min to 7°C instead of 22°C and left overnight at 7°C.

A Bruker P 20 Minispec pulsed NMR spectrometer with an analogue computation unit was used. Mixtures of triolein and tristearin of various ratios were melted and thoroughly homogenised before being transfered into NMR tubes (10 mm o.d. x 100 mm) to a height of 15 mm. The transfering action was carried out in an oven at 70°C. These reference samples were then slowly cooled at 5°C/hr to room temperature and left overnight before being used for calibrating the digital readout unit. Eleven such reference samples were prepared with solid contents ranging from 0% to 100% at 10% intervals. The pulse width, sensitivity and gain of the spectrometer were adjusted to optimum conditions to give the best linear calibration curve between the measured SFC values and percentage of solid in the prepared reference samples.

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The palm oil samples (oil and mixtures) were melted and thoroughly homogenised before being filled in the NMR tubes. A number of tubes was required for each sample. The sample was first kept at 70°C for 30 min. and chilled at 0°C for 90 min. One tube was then held at each measuring temperature for 30 min. prior to measurement (OH, 1978). The transfer of the tubes from melting to the chilling temperature and subsequently to the measuring temperatures was carried out in a proper sequence such that every tube was given the same length of chilling and holding time. The melting, chilling and holding of the samples were carried out in aluminium conditioning blocks which were pre-equilibrated in thermostated baths.

RESULTS

The composition and properties of palm oil and its fractions are given in *Table 1*. *Figure 1* shows the different melting behaviours of palm oil and the stearin fractions. When palm oil is mixed with hard stearin or soft stearin fraction, its melting behaviour is rather different. This is especially so for mixtures with high levels of adulteration as shown in *Figures 2* and *3* and *Table 2*.

The designation for these mixtures is explained below: the percentage of adulteration in palm oil is indicated by a number directly in front of the symbol P which denotes palm oil. This is preceded by a prefix which for hard stearin is S, for soft stearin is D. For example S5P is a mixture of 5% hard stearin in palm oil.

DISCUSSION

Palm oil and its fractions behave rather differently during the process of melting *(Figure 1)*. The hard stearin having higher SFC values melts at first gradually but more rapidly at a later stage. Complete melting occurs only at high temperature. Palm oil having much lower SFC values melts in a rather different manner. Upon heating its SFC values decreases rapidly at first but more slowly on approaching total melting. The soft stearin which is softer than the hard stearin but harder than palm oil has its melting behaviour intermediate between those of the two.

When palm oil is mixed with stearin (up to 20% by weight) its melting curve is displaced upward towards higher SFC values. An upward displacement of the melting curve would mean that the mixture would have correspondingly higher melting

TABLE 1: FATTY ACID COMPOSITION, MELTING RANGE AND SLIP POINT OF PALM OIL AND ITS FRACTIONS

Acid	Palm Oil	Hard Stearin	Soft Stearin
C14	1.3	1.4	1.1
C16	45.4	72.9	59.4
C18	4.3	4.7	5.5
C18:1	38.9	17.2	29.6
C18:2	10.1	3.8	4.4
Melting Range/C	22–49	57–59	40–51
Slip Point/C	35	57	42
I.V.*	53.2	22.3	34.6

* I.V. = Iodine value (calculated)
= 0.9(%C18:1) + 1.8(%C18:2)

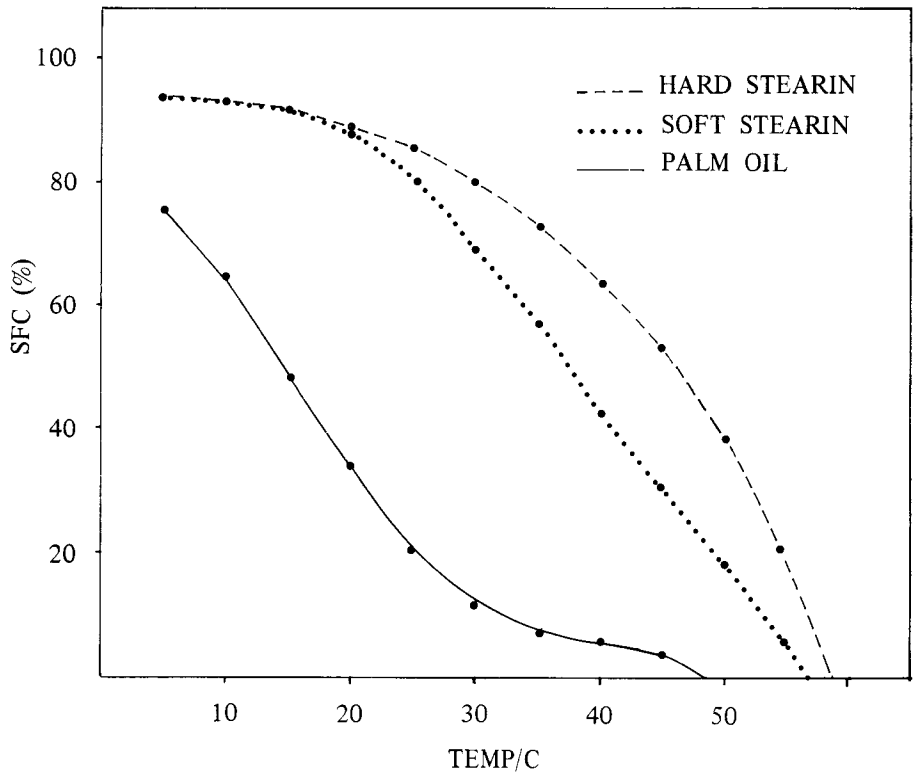

Figure 1: Melting Behaviours of Palm Oil and Its Fractions.

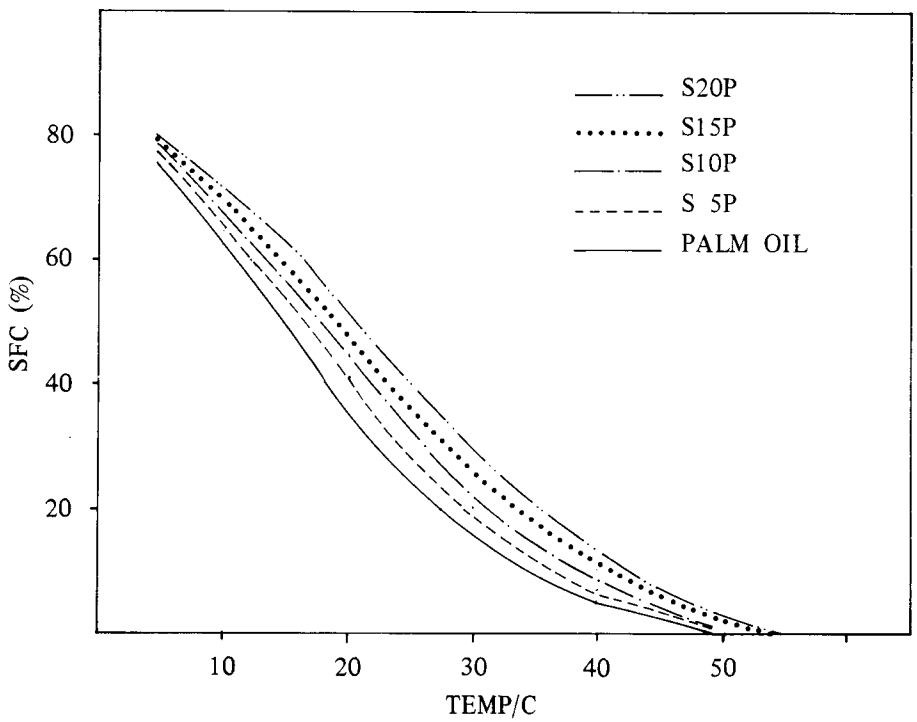

Figure 2: Melting Behaviours of Hard Stearin – Palm Oil Mixtures.

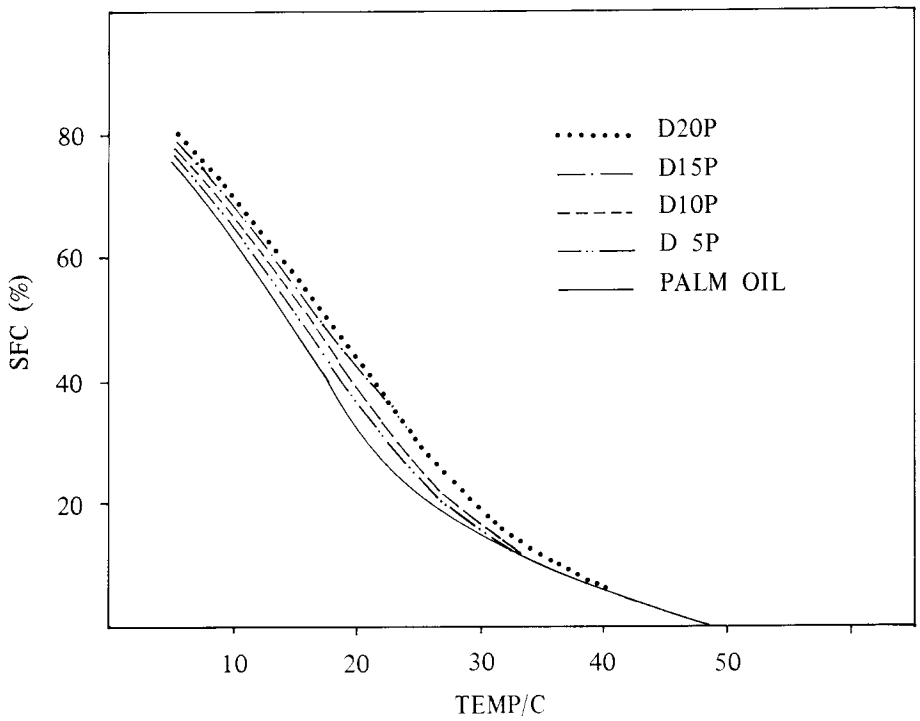

Figure 3: Melting Behaviours of Soft Stearin – Palm Oil Mixtures.

TABLE 2: SOLID FAT CONTENT (%) OF PALM OIL, STEARINS AND MIXTURES OF THE TWO

Temperature (°C)	S10P	S20P	Hard Stearin	D10P	D20P	Soft Stearin	Palm Oil
10	68.8	74.2	93.2	63.8	68.3	92.8	61.8
20	45.0	53.5	88.8	39.0	37.1	86.2	31.8
30	23.5	38.0	78.5	18.0	15.2	69.8	13.0
40	10.1	18.2	63.0	4.8	5.2	42.8	5.0
50	–	7.8	36.8	–	–	17.0	–

range. The effect is larger for the hard stearin and the displacement is observed over the entire temperature range (5° – 55°C). For soft stearin the displacement is smaller and is limited only to temperatures below 30°C. In general displacement increases with the amount of hard stearin present in the mixtures *(Table 2)*. However this is not always the case with additions of soft stearin.

Like any other natural product, palm oil do fluctuate to a certain extent. The sensitivity of this method of detecting adulteration depends on the range of the SFC values of palm oil that is acceptable to both the oil producer as well as the consumer. Further measurements on a large number of representative palm oil samples have to be carried out in order to establish a standard procedure of such detection.

The results however show that the effect of stearin fractions on the melting behaviour of palm oil could probably be used as a means of detecting the presence of these fractions, especially when hard stearin is added to palm oil either intentionally or otherwise.

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SUMMARY

The melting behaviours of (i) palm oil and (ii) mixtures of stearin and palm oil of various compositions were monitored over the temperature range of 5° to 55°C by solid fat content (SFC) measurement using a pulsed NMR spectrometer. The results showed that they all have similar behaviours except that the incorporation of stearin into palm oil has increased its SFC values over the temperature range studied. The effect is larger for hard stearin than it is for soft stearin. This offers a possible means of detecting adulteration in palm oil.

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