UNIVERSITI PUTRA MALAYSIA

BEHAVIOUR OF PALM OLEIN DURING LOW TEMPERATURE STORAGE AND IDENTIFICATION OF PALM OLEIN CLOUD

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BEHAVIOUR OF PALM OLEIN DURING LOW TEMPERATURE STORAGE AND IDENTIFICATION OF PALM OLEIN CLOUD

BY

PHYO ZAW SWE

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Especially dedicated to my beloved parents....

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THE BEHAVIOUR OF PALM OLEIN DURING LOW TEMPERATURE STORAGE AND IDENTIFICATION OF PALM OLEIN CLOUD

BY

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March, 1994

Chairman : Associate Professor Yaakob Bin Che Man, Ph.D.

Faculty : Food Science and Biotechnology

Palm olein, one of the world's most consumable oil, faces problems such as poor low temperature stability and formation of cloud upon storage. In order to study the behaviour of the oil during low temperature storage and identify the components of cloud, the oil was crystallized at 12.5°C over the period of 12 to 24 hours. The behaviour of the triglycerides present in the crystallized oil were monitored by three independent analyses: carbon number analysis (CN) by gas liquid chromatography (GLC), fatty acid composition and content by fatty acid methyl esters (FAME)-GLC, and glyceride composition and content by reverse phase high performance liquid chromatography (RP-HPLC). At 18 hours of storage, the triglyceride types determined by CN analysis that had the maximum concentration (44.49%) was C50 while C52 exhibited the lowest value of 41.10%. In FAME analysis, palmitic acid (C16) had the highest concentration of 41.67% after the oil had been stored for 15 hours while oleic (C18:1) exhibited the lowest value of 41.52%. Triglyceride analysis by HPLC showed that palmitic-oleic-palmitic (POP) concentration increased to the highest value of 33.53% at 18 hours of storage while palmitic-oleic-oleic (POO) concentration decreased to the lowest value of 23.98% which represent 19.96% increased and 12.77% decreased, respectively.

The second aspect studied was the separation of cloud from palm olein and identification of the glyceride that made up the cloud. The cloud from palm olein was separated from the mother (liquid) oil by crystallizing the oil at 10°C for 4 hours followed by brief centrifugation. Oils from three different sources were used as samples. Isolated clouds were identified by using the three analyses mentioned above. Clouds from all three sample oils were found to comprise of 1,3-dipalmito-glycerol and 1-palmito-3-oleo-glycerol. The physical properties of the cloud was determined by X-ray diffraction (XRD) and differential scanning calorimetry (DSC) analyses. A polarized optical microscope was employed to observe the crystal morphology. The results indicated that the cloud crystals had a mix structure of ß-a and ß-b polymorphic forms, a melting point of 70.3°C, crystallization temperature of 53.8°C, the heat of fusion and crystallization were 129.84J/g and -129.24J/g respectively. The most common crystal size ranged from 70μm to 80μm.

Abstrak tesis yang dikemukakan kepada Senat UPM sebagai memenuhi syarat untuk Ijazah Sarjana Sains.

TABIAT OLEIN KELAPA SAWIT SEMASA PENYIMPANAN PADA SUHU RENDAH DAN PENGENALPASTIAN KELADAK OLEIN KELAPA SAWIT

OLEH

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Mach, 1994

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Olein kelapa sawit, salah satu minyak yang paling banyak digunakan di dunia, mengalami masalah-masalah seperti kestabilan yang rendah dan pembentukan keladak apabila disimpan. Bagi mengkaji tabiat minyak semasa penyimpanan suhu sejuk dan mengenalpasti komponen-komponen keladak, minyak dihablur pada 12.5°C bagi jangka masa dari 12 hingga 24 jam. Tabiat trigliserida yang terdapat didalam minyak yang dihablurkan dianalisis dengan tiga analisis yang berasingan: analisis nombor karbon (CN) dengan kromatografi gas cecair (GLC), komposisi dan kandungan asid lemak dengan metil ester asid lemak (FAME) GLC, dan komposisi, dan kandungan gliserida dengan fasa berbalik kromatografi cecair berprestasi tinggi (RP-HPLC). Bagi penyimpanan pada 18 jam, jenis trigliserida ditentukan dengan analisis CN yang menunjukkan

kepekatan maksima (44.49%) ialah C50 sementara C52 menunjukkan nilai terendah iaitu 41.10%. Dalam analisis FAME, asid palmitik (C16) menunjukkan kepekatan tertinggi dengan 41.67% selepas minyak disimpan selama 15 jam sementara oleik (C18:1) menunjukkan nilai terendah iaitu 41.52%. Analisis trigliserida dengan HPLC menunjukkan bahawa kepekatan palmitik-oleik-palmitik (POP) meningkat kepada nilai tertinggi iaitu 33.53% pada 18 jam penyimpanan sementara kepekatan palmitik-oleik-oleik (POO) menurun kepada nilai 23.98%. Angka-angka ini mewakili peningkatan 19.96% dan penurunan 12.77%, masing-masing.

Aspek kedua yang dikaji ialah pemisahan keladak dari olein kelapa sawit dan pengenalpastian gliserida yang menjadikan keladak. Keladak dari olein kelapa sawit telah dipisahkan dari minyak asal dengan penghabluran minyak pada 10°C selama 4 jam diikuti dengan pengemparan yang singkat. Minyak dari tiga sumber digunakan sebagai sampel. Keladak yang diasingkan telah dikenalpasti dengan menggunakan tiga analisis seperti di atas. Keladak dari ketiga-tiga sampel minyak didapati mempunyai 1,3 dipalmito-gliserol dan 1-palmito-3-oleo-gliserol. Ciri-ciri fizikal keladak telah ditentukan dengan analisis pembelauan sinaran-X (XRD) dan kalorimeter pengimbas pembezaan (DSC). Mikroskop optik terkutub telah digunakan untuk memerhati morfologi hablur. Keputusan menunjukkan bahawa keladak mempunyai struktur campuran dari bentuk polimorf ß-a dan ß-b, titik cair 70.3°C, suhu penghabluran 53.8°C, haba pelakuran dan penghabluran 129.84J/g dan -129.24J/g masing-masing. Saiz hablur yang paling banyak ialah dalam julat 70μm to 80 μm.

CHAPTER 1

INTRODUCTION

Natural and processed fats and oils, whether of animal or vegetable origin, play a significant role in the economy of many countries including both oil-producers and oil users. These materials are used extensively, but not exclusively in the food industry.

Palm oil is derived from the mesocarp of the fruits of the oil palm species *Elaeis guineensis* mainly of the hybrid *Tenera* which yields 4-5 tonnes palm oil (PO) per hectare, plus about 0.5 tonne of palm kernel oil (PKO) and 0.6 tonne palm kernel meal (PKM) (PORIM, 1989). Palm oil is now the world's second most important vegetable oil after soybean oil, taking up about 15% of the world's vegetable oils production and exports (Business Times, 1989). Palm oil share in the world oil and fat supplies has been steadily increasing (Lal and Gasper, 1991). World palm oil production has trebled since 1980 and is projected to increase through this decade with potential to outstrip soybean oil production by the year 2000 (Mabbett, 1993). Malaysia now dominates the world market, processing a full 99% of its production at home and exporting all but 10% of the processed oil (Mabbett, 1993). The country accounts for 60% of the world's palm oil production (Mabbett, 1993).

Palm oil has characteristics that makes it a versatile ingredient in food products. By simple fractionation, palm oil can be resolved into a liquid, a solid and an intermediate fractionated palm olein (known as palm midfraction) and various grades of palm stearin can be obtained (Loke and Teah, 1989). Moreover, the application of hydrogenation and interesterification processes increases the range of palm oil products available for use by the food manufacturing industry.

From the nutritional point of view, palm oil is not only cholesterol free but is also a rich source of possible anti-cancer agents such as beta-carotene (pro-vitamin A) and vitamin E (Temple and Basu, 1988). Unrefined palm oil is the richest known natural source of the pro-vitamin A pigment, beta-carotene. However, the refined palm oil (as is the case with most other refined oils) has its carotene destroyed by heat.

The characteristics of palm oil for edible use have the properties in common such as bland flavour, pale colour and glossy appearance and good stability in use and storage. While palm oil usage in the solid fat market is substantial, its share in the liquid oil market is limited because of its poor cold stability and notorious cloud formation (Loke and Teah, 1989). These and other properties are determined by the glycerides and the fatty acid compositions of palm oil.

Palm olein is the liquid fraction obtained by fractionation of palm oil after crystallization at a controlled temperature. The storage quality of palm olein depends greatly on efficiency of the fractionation procedure. It is fully liquid in warm climates, has a narrower range of glycerides, and blends perfectly with any seed oil (Loke and Teah, 1989). Refined, bleached and deodorized (RBD) palm olein, the major form of palm oil consumed and exported by Malaysia, contains 46% saturated fatty acids (myristic, palmitic and stearic); 43% mono-unsaturated (oleic) and 11% poly unsaturated acids (linoleic) (Gunstone, 1986). Due to the contents of its saturated fatty acids, palm olein cannot meet the 'cold test' in U.S.A and other temperate countries, which specifies that a salad oil must remain clear after standing 5.5 hours in an ice bath (AOCS Method Cc 11-53, 1984). One of the possible solutions to this issue is by blending palm olein with the more unsaturated oils (Loke and Teah, 1989), modifying composition toward greater unsaturation of palm oil (Tan et al., 1981) or by adding the crystal inhibitors (Weiss, 1967).

The factors that causes cloud in palm olein, often seen especially on supermarket shelf, is somehow overlooked by many researchers. Knowledge of chemical make-up of clouds is absolutely necessary in order to eliminate cloud. More emphasis can therefore be given on improving the quality of palm olein relating to cold stability and cloud formation.

At present, there is no report available either on behaviour of palm olein in low temperature storage or on the identity of cloud. Therefore, the objectives of this study are outlined as follows:

1. To monitor the triglyceride behaviour of palm olein during low temperature storage.
2. To isolate the cloud and identify its components.
3. To study polymorphism and thermal behaviour of the cloud.

CHAPTER 2

LITERATURE REVIEW

Utilization of Palm Oil

Palm oil and palm kernel oil are both derived from the oil palm fruit and have many applications with potential applications currently being explored. The use of palm oil can be categorized into edible and non-edible purposes.

Edible Purposes

Palm oil may be used in most of the major edible products made from vegetable oil. These are: (1) domestic margarine (2) non-domestic margarine (3) low calorie spread (4) vanaspati (5) vegetable shortening and (6) edible oil.

Domestic Margarine

Most margarine is produced and used in developed countries. It has been produced in a number of different forms, not merely in the image of butter. Massiello (1978) lists 10 kinds of household margarine: regular stick, polyunsaturated and high polyunsaturated stick, whipped stick (regular, premium) and whipped soft (tub, liquid, diet imitation) and vegetable oil spreads.

Domestic margarine in these countries should be spreadable at a very low temperature. The physical characteristics of margarine correlate well with solid fat index (SFI) measurements of the oil from which it is made. Soft margarine has SFI values at 10°C from 21 to 24 (Weiss, 1983). Table 1 shows the comparison of SFI values of natural fats. From the table, it can be seen that palm oil is not suitable in soft margarine processing because of its much higher SFI value (34).

Table 1

SFI Values of Natural Fats

Fat	Melting point (°C)	SFI value				
		10°C	21.1°C	26.7°C	33.3°C	37.8°C
Butter	36	32	12	9	3	0
Cocoa butter	29	62	48	8	0	0
Coconut oil	26	55	27	0	0	0
Lard	43	25	20	12	4	2
Palm oil	39	34	12	9	6	4
Palm kernel oil	29	49	33	13	0	0
Tallow	48	39	30	28	23	18

(Source: Weiss, 1983)

Consumers are health conscious and they would normally go for a margarine that is high in polyunsaturated fatty acids. Solid fat content (SFC) of palm oil is high and polyunsaturated fat content is relatively low (about 10%) (Moolayil, 1976). Therefore, palm oil can be used only in limited amounts in domestic margarine in developed countries. As the above mentioned limitations apply to a lesser extent only in the developing countries, higher percentage of palm oil can be used in producing domestic margarine in these countries.

On the other hand, palm oil is highly suited as a component in margarine production because of attributes such as it crystallizes in beta-prime (ß') form, has a semi-solid consistency similar to butter, has a high level of palmitic acid, it reduces excessive C18 level of most seed oils, has an entirely natural fatty acid composition, has no trans- or positional isomers, has 10% natural linoleic acid (EFA), has no linolenic acid and no flavour reversion (Pantzaris, 1988). Several patents have been issued which use interesterification reactions for processing margarine oils. Carlile and van Selm (1976) randomize palm oil to be blended with hydrogenated soybean oil and liquid vegetable oil for tub margarine. They claim that unrandomized palm oil crystallizes slowly and ultimately causes the margarine to become hard and brittle. Randomized palm oil remains soft and spreadable. Wieske et al. (1976) co-randomized palm oil, coconut oil, and hydrogenated soybean oil, and blended this with liquid oil to make a soft tub margarine. Fromhold (1974) prepared a similar product with different proportions of the various component oils. Co-randomized palm oil, palm stearin fraction, and soybean oil, and blending it with uninteresterified palm oil, palm olein fraction, and soybean oil could also be used to make margarine in print form (Kattenberg and Verburg, 1977). Others like Read (1975), Kattenberg and Poot (1977) co-randomized a blend of palm with hydrogenated soybean oil to make a tub margarine free from oiliness, graininess, and waxiness. Wieske (1977) co-randomized palm and soybean oil hard fats and