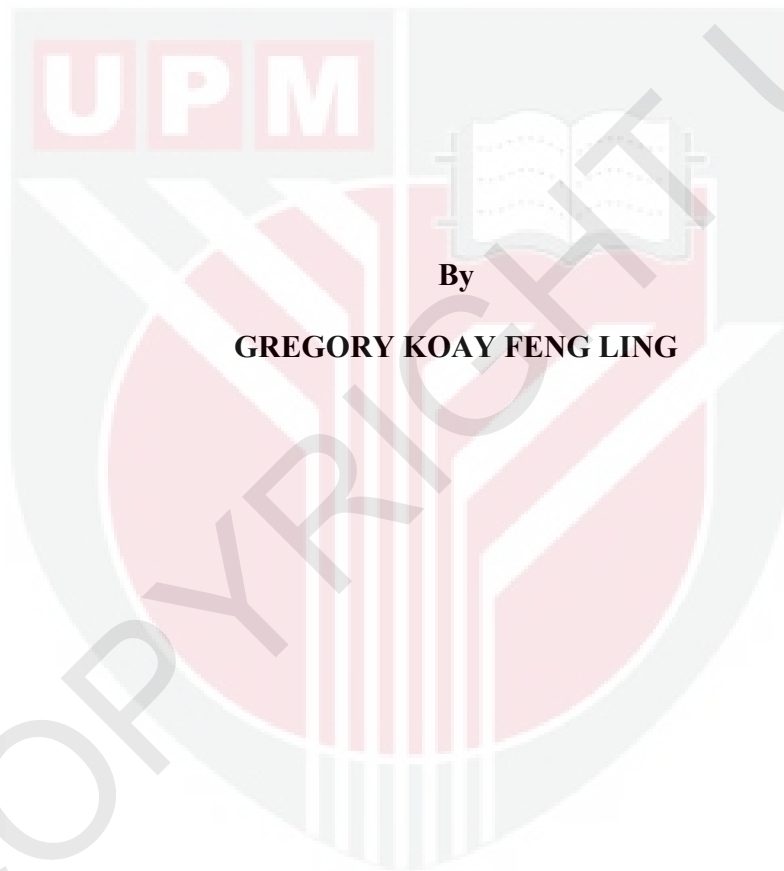


**CRYSTALLIZATION OF PALM OIL-BASED 9,10-DIHYDROXYSTEARIC
ACID EMPLOYING ISOPROPYL ALCOHOL AS SOLVENT**



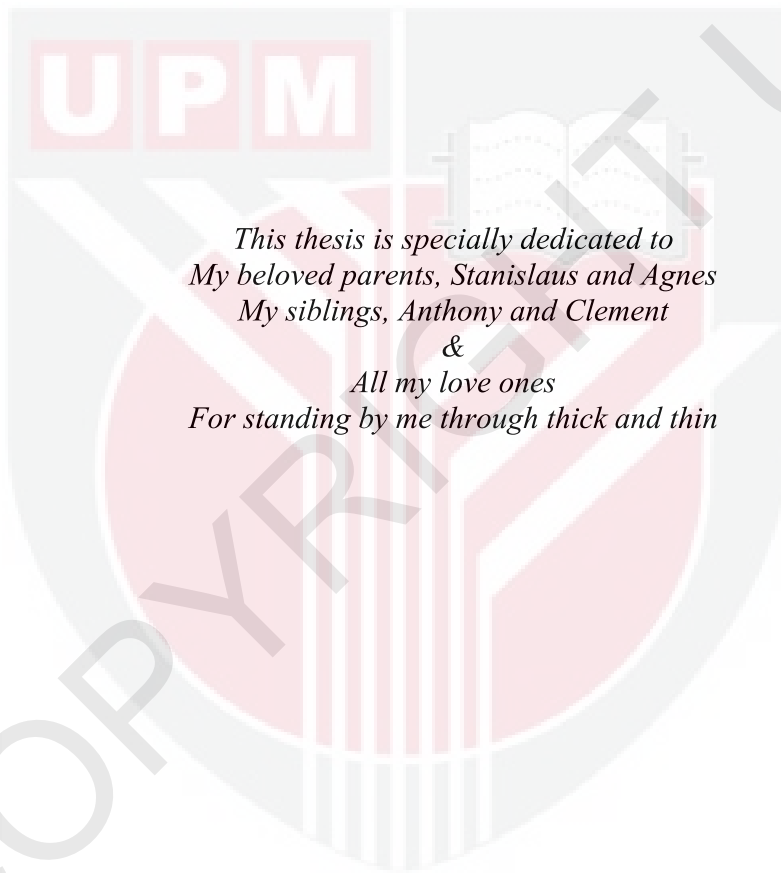
By

GREGORY KOAY FENG LING

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of Master of Science**

April 2007

FK 2007 23



*This thesis is specially dedicated to
My beloved parents, Stanislaus and Agnes
My siblings, Anthony and Clement
&
All my love ones
For standing by me through thick and thin*

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

CRYSTALLIZATION OF PALM OIL-BASED 9,10-DIHYDROXYSTEARIC ACID EMPLOYING ISOPROPYL ALCOHOL AS SOLVENT

By

GREGORY KOAY FENG LING

April 2007

Chairman: Associate Professor Luqman Chuah Abdullah, PhD

Faculty: Engineering

9,10-dihydroxystearic acid (DHSA) was derived from commercial grade palm oil based crude oleic acid. It was derived through the epoxidation of the unsaturation present in the oleic acid with performic acid in the presence of sulphuric acid and hydrogen peroxide, followed with in-situ hydrolysis of the epoxide with hydrogen donor such as water. DHSA promises great potential in the personal care industry. It has the prospective to act as a multipurpose intermediate in the synthesis of fine chemical products such as decorative cosmetics. However, the impurities present in DHSA could lead to skin irritation and thus the crude DHSA produced need to endure purification process in order to be qualified as an additive for the cosmetic industry. Solvent crystallization was chosen as the path in preparing DHSA crystals of at least 80% purity and of high desirability.

The objective of this research was to study the effects of temperature and of solvent quality and quantity on the solvent crystallization of DHSA. On detailing the effects of temperature, four aspects were scrutinized: weight based crystal yield, particle size

distribution (PSD), quality of purified DHSA crystals and crystallization operation efficiency. PSD, crystal morphology and quality of purified DHSA crystals were scrutinized when detailing the effects of solvent quality and quantity.

In the course of carrying out the research work, a crystallization unit was fabricated. The detailed design and operation of the crystallization unit were described. This research, through gas chromatography (GC), PSD, Fourier Transform – Infra Red (FT-IR) and scanning electron microscopy (SEM) analysis, revealed that solvent crystallization of DHSA was best carried out with the cooling temperature of 20°C, 80% isopropyl alcohol (IPA) concentration and $\frac{\text{crude dihydroxystearic acid}}{\text{solvent}}$ ratio of 1.0:1.0.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**PENGHABLURAN ASID 9,10-DIHIDROKSISTEARIK BERASASKAN
MINYAK SAWIT MENGGUNAKAN ISOPROPIL ALKOHOL SEBAGAI
PELARUT**

Oleh

GREGORY KOAY FENG LING

April 2007

Pengerusi: Profesor Madya Luqman Chuah Abdullah, PhD

Fakulti: Kejuruteraan

Asid 9,10-dihidroksistearik (DHSA) dihasilkan daripada asid oleik mentah yang berasaskan minyak sawit bergred dagangan. Ia dihasilkan melalui proses mengepoksidasikan ketidaktepuan yang wujud dalam asid oleik dengan asid performik. Proses ini dijalankan dalam kehadiran asid sulfurik dan hidrogen peroksida. Seterusnya, proses hidrolisis dijalankan secara *in-situ* keatas epoksida yang terhasil dengan penderma hidrogen seperti air. DHSA mempunyai potensi yang baik dalam industri penghasilan barangan penjagaan diri. Ia mempunyai prospek untuk menjadi pengantara pelbagaiguna dalam sintesis bahan kimia halus seperti bahan kosmetik untuk tujuan perhiasan diri. Namun, bahan cemar yang hadir boleh mengakibatkan kesan sampingan. Oleh itu, DHSA mentah perlu dinyahcemarkan supaya ia boleh digunakan sebagai aditif dalam industri kosmetik. Proses penghabluran dengan bantuan pelarut telah dipilih untuk menghasilkan hablur DHSA berketulenan 80% dan ke atas serta mempunyai permintaan yang tinggi dalam pasaran.

Objektif kajian ini ialah untuk mengkaji kesan suhu dan kesan kualiti dan kuantiti bahan pelarut ke atas penghabluran DHSA. Dalam mengkaji kesan suhu, empat aspek telah diperinci: jumlah penghasilan hablur, taburan saiz zarah (PSD), kualiti hablur DHSA yang telah dituliskan dan kecekapan operasi penghabluran. PSD, morfologi hablur dan kualiti hablur DHSA diperinci semasa mengkaji kesan kualiti dan kuantiti bahan pelarut.

Dalam pada menjalankan kerja kajian ini, seunit mesin penghablur telah dibina. Rekabentuk dan cara mengoperasikan mesin penghablur ini telah diterangkan secara terperinci. Analisis kromatografi gas (GC), PSD, Penukaran Fourier – Infra Merah (FT-IR) dan penelitian mikroskopi dengan elektron (SEM), menunjukkan bahawa penghabluran DHSA paling sesuai dijalankan pada suhu 20°C dengan alkohol isopropil berkepekatan 80% dan DHSA mentah:bahan pelarut bernisbah 1.0:1.0.

ACKNOWLEDGEMENTS

First and foremost, I would like to thank my advisor and chairman of the supervisory committee, Assoc. Prof. Dr. Luqman Chuah Abdullah, my supervisory committee members Assoc. Prof. Dr. Thomas Choong Shean Yaw, Dr. Salmiah Ahmad and Ir. Parthiban Siwayanan for constantly guiding and encouraging me throughout this research. Thanks for your advice and suggestion. Without you, this thesis could not have reached its final form.

I would also like to extend my thanks to all the staff members of Department of Chemical and Environmental Engineering, UPM, of School of Graduate Studies, UPM and of Advanced Oleochemical Technology Division, MPOB, especially for the professional and technical assistance rendered.

Last but not least, I thank my parents, siblings, all my family members and all my love ones for their continuous prayers, love and support throughout my course of study.

I certify that an Examination Committee has met on 18 April 2007 to conduct the final examination of Gregory Koay Feng Ling on his Master of Science thesis entitled “Crystallization of Palm Oil-Based 9,10-Dihydroxystearic Acid Employing Isopropyl Alcohol as Solvent” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

Azni Idris, PhD

Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Sa’ari Mustapha, PhD

Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Tinia Idaty Mohd Ghazi, PhD

Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Mohd Sobri Takriff, PhD

Associate Professor
Faculty of Engineering
Universiti Kebangsaan Malaysia
(External Examiner)

HASANAH MOHD. GHAZALI, PhD

Professor/Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 21 JUNE 2007

This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

Luqman Chuah Abdullah, PhD

Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Thomas Choong Shean Yaw, PhD

Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Salmiah Ahmad, PhD

Deputy Director General (Services)
Malaysian Palm Oil Board
(Member)

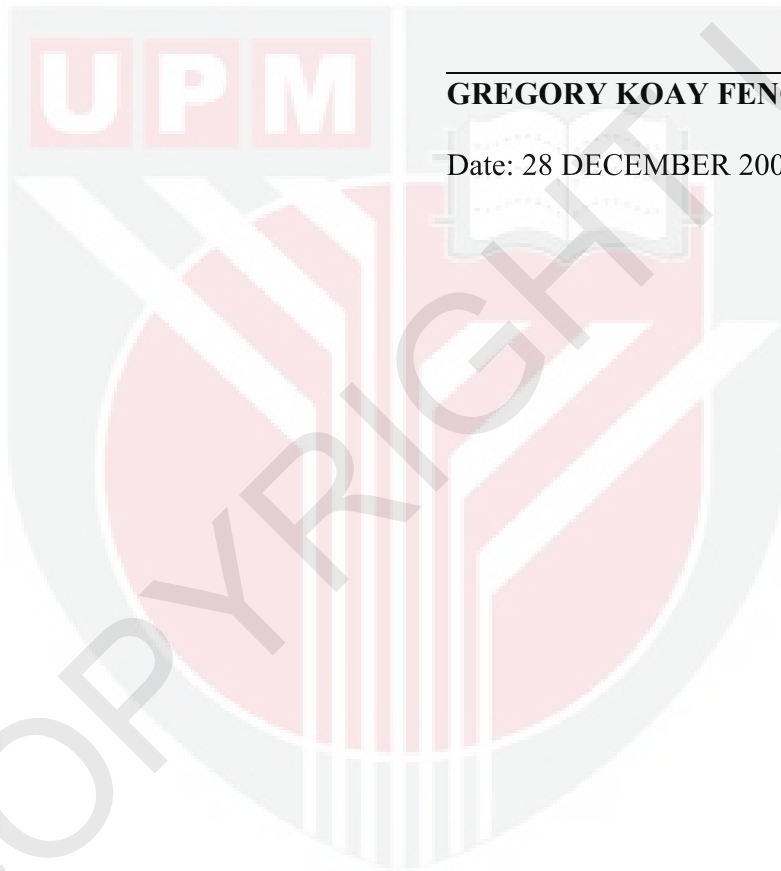
AINI IDERIS, PhD

Professor/Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 17 JULY 2007

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



GREGORY KOAY FENG LING

Date: 28 DECEMBER 2006

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LIST OF ABBREVIATIONS

AOTD	Advanced Oleochemical Technology Division
BTSFA	Bis(trimethylsilyl)trifluoroacetamide
CSD	Crystal size distribution
CSTR	Continuous stir tank reactor
DHSA	Dihydroxystearic acid
DMF	N,N-Dimethylformamide
D [3,2]	Surface weighted mean diameter
D [4,3]	Volume weighted mean diameter
FFA	Free fatty acids
FT-IR	Fourier Transform – Infra Red
G	Growth rate
GC	Gas chromatography
HCOOH	Performic acid
H ₂ O ₂	Hydrogen peroxide
H ₂ SO ₄	Sulphuric acid
IPA	Isopropyl alcohol
J	Nucleation rate
J_i^+	Adsorption rate of component i
J_i^-	Desorption rate of component i
KBr	Potassium bromide
KCl	Potassium chloride

KNO ₃	Potassium nitrate
L_D	Lag time
L_s	Steady state geometric mean crystal diameter
$L'(t)$	Geometric mean size of fat crystals' size distribution with time
MPOB	Malaysian Palm Oil Board
N	Population density
NaCl	Sodium chloride
Na ₂ SO ₄	Sodium sulphate or sodium sulphate anhydrous
PKO	Palm kernel oil
PSD	Particle size distribution
RI	Reflective index
R _t	Retention time
SEM	Scanning electron microscopy
TAG	Triacylglycerols
T_c	Crystallization temperature
TMS	Trimethylsilyl
T_m (K) or T_m	Melting point or melting temperature
X_i	Average mesh size
XRD	X-ray diffraction
a_{eq}	Chemical activity at equilibrium state
a_{ss}	Chemical activity at supersaturated state
d_v	Volume diameter
d_s	Surface diameter

d_{sv}	Surface volume diameter
d_d	Drag diameter
d_f	Free falling diameter
d_{St}	Stokes' diameter
d_a or d_p	Projected area diameter
d_c	Perimeter diameter
d_A	Sieve diameter
d_F	Feret's diameter
d_M	Martin's diameter
d_R	Unrolled diameter
h_i	Induction time
k_i	Volume percent
n	Number density per volume of crystals or total volume percent, 100
s	Standard deviation
s_{max}	Maximum fraction of solid fat
$s(t)$	Fraction of solid fat at time t
x	Solubility of solute at temperature T (K)
x_{eq}	Chemical activity of ideal solution at equilibrium state
x_{ss}	Chemical activity of ideal solution at supersaturated state
x_i^l	Molar fraction of component i at arbitrary point
$x_{i,eq}^l$	Molar fraction of component i at equilibrium liquid state
x_i^s	Concentration of component i at solid surface

x_j^s	Concentration of component j at solid surface
ΔG_c	Activation free energy of nucleation
ΔG_d	Activation free energy of diffusion
ΔH	Change in enthalpy
ΔH_m	Molar heat of melting
ΔT	Supercooling value
λ	Nominal induction time
μ	Maximum increase rate in fraction solid fat
μ_{eq}	Chemical potential at equilibrium state
μ_{ss}	Chemical potential at supersaturated state
v	Growth rate
σ	Supersaturation or crystal/melt interface free surface energy
τ_c	Time constant associated with crystallization

CHAPTER 1

INTRODUCTION

1.1 Oleochemicals

Oleochemicals are chemicals derived from biological oils and fats via the splitting of triglycerides into their constituent fatty acid derivatives and glycerol or via modification of oils and/or fats. The oils and/or fats may be of vegetable (*e.g.* castor oil, coconut oil, corn oil, cottonseed oil, groundnut oil, linseed oil, olive oil, palm kernel oil (PKO), palm oil, rapeseed oil, sesame oil, soybean oil, sunflower oil, etc.), animal (*e.g.* butter, lard and grease, edible and non edible tallow, etc.) or marine (*e.g.* fish oil) origin. Oleochemicals are analogous to petrochemicals. In other words, similar chemicals may be synthesized from petroleum. The most basic oleochemicals are fatty acids, fatty methyl esters, fatty alkyl esters, fatty alcohols, fatty amines and glycerol (Ahmad *et al.*, 2000a).

1.2 Fatty Acids

In chemistry, a fatty acid is a carboxylic acid. A fatty acid is characterized by the number of carbon atoms in the carbon chain, ranging generally from caproic acid with six carbon atoms to behenic acid with 22 carbon atoms. Most of the natural fatty acids have an even number of carbon atoms. Fatty acids without double bonds are called saturated fatty acids. They are generally solids (Dieckelmann and Heinz, 1988).

Saturated fatty acids also do not contain other functional groups along the carbon chain. The term 'saturated' refers to hydrogen, in that all carbons (apart from the carboxylic acid [-COOH] group) contain as many hydrogen atoms as possible.

Fatty acids with double bonds are unsaturated and generally liquids (Dieckelmann and Heinz, 1988). One or more alkene functional groups exist along the carbon chain, with each alkene substituting a singly-bonded '-CH₂-CH₂-' part of the chain with a doubly bonded '-CH=CH-' part. The two hydrogen atoms that are bound to the doubly bonded carbon atoms can occur in either *cis*- or *trans*- configuration.

1.3 Palm Oil and Palm Kernel Oil as Sources of Fatty Acids

In tallow, C₁₆ and C₁₈ fatty acids dominate; in coconut oil, C₁₂ and C₁₄ acids are more prevailing (Dieckelmann and Heinz, 1988). Thus, tallow and coconut oil are traditionally used as feed stocks for oleochemicals of chain lengths 12, 14, 16, 18 and 18:1. Prior to 1985, tallow was an important raw material for the oleochemical industry. However, during the period 1985-1995, the significance of tallow was greatly reduced. This was due to the following three factors:

- Minimal increase in the world's production (about 25% increase in 10 years);
- Suspected relationship between human disease and materials of animal origin and thus, the reluctance of manufacturers to use tallow;

- The negative perception among consumers on the use of animal derived oleochemicals for consumer products, particularly in personal care products.

For the ensuing years, no increase or even a decrease in tallow production and use was foreseen and it was expected that tallow would play a less important role in the oleochemical industry in the future.

In a similar fashion, prior to 1985, coconut oil was the most important source of C₁₂ and C₁₄ fatty acids for the oleochemical industry. However, during the period of 1985-1995, the global production of coconut oil increased by only 10% and the production was not expected to increase significantly in the foreseeable future. Thus, the significance of coconut oil for the oleochemical industry was also expected to be diminished somewhat in the future (Ahmad *et al.*, 2000a).

For the corresponding period, both palm oil and PKO enjoyed steady and heartening growth in Malaysia. In 1985, the production of crude palm oil and crude PKO stood at 4,134,463 and 511,908 tons per annum respectively and by the year 1994, the production increased to 7,810,546 and 1,036,538 tons per annum respectively. There was almost a 90% increase for crude palm oil production and over 100% increase for crude PKO production. For the ensuing 10 years, the production increase was almost 80% for crude palm oil and almost 60% for crude PKO. In 2004, the production of crude palm oil stood at 13,976,182 tons per annum while the production of crude PKO

stood at 1,644,445 tons per annum (MPOB, 2005). The annual production of oil palm products in Malaysia for the period 1975-2004 is shown in Table 1.1.

Table 1.1: Malaysian annual production of oil palm products for 1975 – 2004 (Malaysian Palm Oil Board – MPOB, 2005)

Year	Crude palm oil (tons)	Crude PKO (tons)
1975	1,257,573	108,260
1980	2,573,173	222,285
1985	4,134,463	511,908
1990	6,094,622	827,233
1995	7,810,546	1,036,538
2000	10,842,095	1,384,685
2004	13,976,182	1,644,445

This mass production not only put Malaysia in the global map as both the world top producer and exporter of palm oil but also as the world third major producer and top exporter of 17 biological oils and fats besides elevating the status of palm oil as the world second most produced biological oils and fats, ranking right behind soybean oil (MPOB, 2005).

Insignificant increase in tallow production allowed the emergence of palm oil as a new source of C₁₆ and C₁₈. As the centre of growth of oleochemicals gradually shifts to the ASEAN region, palm oil would equally replace tallow and its usage would therefore markedly increase (Yusof *et al.*, 1996; Ahmad *et al.*, 2000a). Coconut oil used to enjoy a unique position in the oleochemical industry due to its fatty acid composition, particularly lauric acid which is highly regarded in the cosmetics and detergent industry. However, this position is threatened with the emergence of PKO as the sole alternative source of commercially available lauric oils. For Malaysian oleochemical

manufacturers, PKO is preferred as coconut oil is more expensive and its supply is limited (Dieckelmann and Heinz, 1988; Ahmad *et al.*, 2000a).

A survey conducted by Mohtar (1998) from January 1996 to October 1997, the 93 samples provided by ten companies indicated that various types and grades of palm fatty acids were produced in Malaysia. The composition of the products depended on the raw materials used (whether they were crude or refined, hydrogenated or un-hydrogenated, palm oil or PKO based) and the purification processes used after the splitting.

1.4 Hydroxyl Fatty Acids

Hydroxyl fatty acids have great interest to the industry because of their different behavior compared with ordinary fatty acids. Hydroxyl fatty acids and their derivatives have many applications, *e.g.* as additives in lubricants and cosmetic products, as emulsifiers, as polyols for polyurethanes and as surfactants for detergents. Their uses as a corrosion inhibitor in lubricants (Borggrete, 1990) and as auxiliaries for the textile industry (Meffert and Wilk, 1988) have been investigated. Currently, the main source of hydroxyl fatty acids supplied by nature is castor oil. Because castor oil is not available in Malaysia, other means to produce the acids have been initiated. Oleic acid is a valuable oleochemical available from many natural sources including palm oil and is an important raw material for preparing hydroxyl fatty acids. In Malaysia, palm oil emerges as an alternative to castor oil, owing to its oleic acid composition (Roila *et al.*,

1998; Siwayanan *et al.*, 2004; Ahmad *et al.*, 2000a). Table 1.2 shows that high content of oleic acid is found in palm oil.

Table 1.2: Fatty acid composition of palm oil (Salmiah and Kang, 1998)

Fatty acids	Weight percent
C ₆	-
C ₈	-
C ₁₀	-
C ₁₂	0.2
C ₁₄	1.1
C ₁₆	44.0
C ₁₈	4.5
C _{18:1}	39.2
C _{18:2}	10.1
Others	0.8

Palm oil based hydroxyl fatty acids can be used to substitute castor oil in many of the latter's commercial applications. Its derivatives can be used in lipstick formulations to extend shelf life, as feedstock for the production of emulsifiers and surfactants. In particular, it can be used as one of the starting materials for the production of GEMINI surfactants (surfactant having two long hydrocarbon tails and two head-groups). It is also known to form organogels with various organic liquids. These organogels are widely utilized as regulators for viscoelastic properties of greases and cosmetic products (Ahmad *et al.* 2000b).

1.5 Dihydroxystearic Acid (DHSA)

MPOB has successfully discovered a methodology in producing a dihydroxy fatty acid named as 9,10-DHSA from palm oil based oleic acid. Due to the development of

oleochemical industry in Malaysia, palm oil based unsaturated fatty acids (especially oleic acid) is easily available and provides an easy source of raw materials for this discovery. However, it should be noted that this discovery is not only applicable to palm oil based oleic acid but to oleic acids derived from other types of oils and/or fats as well (Roila *et al.*, 1998). The said DHSAs are specialty chemicals, derived from epoxidation of oleic acid with either peracetic or performic acid followed with hydrolysis with hydrogen donor such as water (Roila *et al.*, 1998; Siwayanan *et al.*, 2004; Koay *et al.*, 2005; Sumaiya *et al.*, 2005). Figure 1.1 shows the DHSAs formation process from oleic acid.

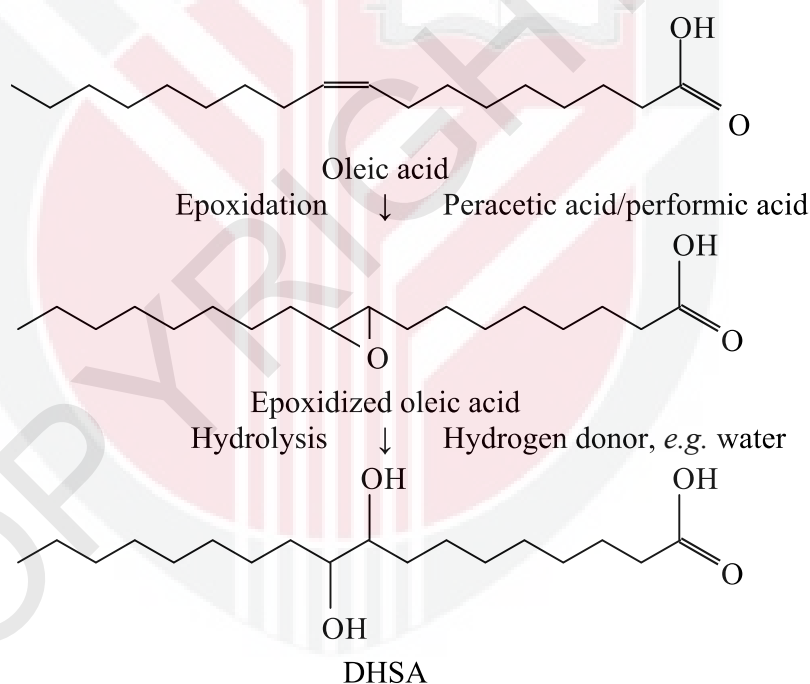


Figure 1.1: DHSAs formation process from oleic acid

Currently, palm oil based crude DHSAs are prepared in a pilot plant located in the premise of MPOB, at the rate of 500kg DHSAs per batch run (Siwayanan *et al.*, 2004). The

DHSA produced is used as primary and secondary emulsifiers, coating agents for pigments and gelling agents for oils and/or fats in the colored cosmetics formulations and research. Some of the applications and/or functionalities of DHSA, with regards to personal care finish products are tabulated in Table 1.3.

Table 1.3: Application and/or functionalities of DHSA in the personal care finish products (Awang *et al.*, 2001a; Rigano, 2003; Ismail, 2006)

Personal care finish products	Application and/or functionalities of DHSA
Lipsticks	DHSA increases the mechanical properties of lipsticks. Lipsticks with DHSA coated pigments possess higher breaking point.
Liquid foundations	DHSA gives better color development in liquid foundations. Liquid foundations with DHSA coated pigments give brighter color, implying that in order to achieve the same color brightness, less pigment is needed. DHSA also eases the spreading of liquid foundations onto the skin and thus enhances the dispersion of pigments.
Mascaras	Mascaras with DHSA coated pigments have shorter drying time, give better curling effect and increase apparent size of the eyelashes (add volume to the eyelashes).
Compact powders	DHSA, as a binding agent, increases the skin adhesion properties of compact powders. Compact powders with DHSA coated pigments give velvety skin feel and is found to be easily distributed when applied to the skin.
Metal soaps	Sodium soaps of DHSA have better detergency than stearic soap at room temperature and are more biodegradable. The soaps also exhibit corrosion inhibition with a corrosion rate of only 0.002mmyr^{-1} at 100ppm concentration.

1.6 Crystallization

A crystal is a solid in which the constituent atoms, molecules or ions are packed in a regularly ordered, repeating pattern extending in all three spatial dimensions. The process of forming a crystalline structure is referred to as crystallization.

With regards to chemical engineering and separation process in particular, crystallization is a solid-liquid separation process in which mass transfer of a solute occurs from the liquid solution to a pure solid crystalline phase. Crystallization is a process where solid particles are formed from a homogeneous phase, essentially for the purpose of purifying solid compounds. This process can occur in the freezing of water to form ice, in the formation of snow particles from a vapor, in the formation of solid particles from a liquid melt or in the formation of solid crystals from a liquid solution. In crystallization the solution is concentrated and usually cooled (cooling in one of the four most common methods of achieving supersaturation, the other three being solvent evaporation, drowning and chemical reaction) until the solute concentration becomes greater than its solubility at that temperature. Then the solute comes out of the solution forming crystals of approximately pure solute (Geankoplis, 1993; Mullin, 1993; Mullin, 2001).

In commercial crystallization, not only the yield and purity of crystals are important but the sizes and shapes of the crystals as well. It is often desirable that crystals be uniform in size. Size uniformity is desirable to minimize caking in the package, for ease of

pouring, for ease in washing and filtering and for uniform behavior when used (Myerson, 1993; Mullin, 1993; Mullin, 2001).

Crystallization cannot occur without supersaturation. In other words, the solution should contain more solute molecules than it would under ordinary conditions. A main function of any crystallizer is to cause a supersaturated solution to form. A classification of crystallizing equipment can be made based on the methods used to bring about supersaturation, as shown in Table 1.4.

Table 1.4: Classification of crystallizing equipment (Geankoplis, 1993)

Crystallizer type	Modes bringing about supersaturation
Tank-crystallizers and batch-crystallizers	Cooling of solution with negligible evaporation
Evaporator-crystallizers and crystallizing evaporators	Evaporation of solvent with little or no cooling
Vacuum-crystallizers	Combined cooling and evaporation through adiabatic evaporator

In tank crystallization, which is an old method still used in some specialized cases, hot saturated solution are allowed to cool in open tanks. After a period of time, the mother liquor is drained and the crystals removed. Nucleation and the size of crystals are difficult to control. The crystallization is essentially just ‘allowed to happen’. Crystals contain considerable amounts of occluded mother liquor and labor costs are very high. In some cases the tank is cooled by coils or a jacket and an agitator is used to improve the heat-transfer rate. However, crystals often build up on these surfaces. This type of crystallization has limited application and is sometimes used to produce some fine

chemicals and pharmaceutical products, where the product value can justify the high operating costs (Geankoplis, 1993).

1.7 Crystallization in Palm Oil Industry

Fractional crystallization refers to the fractionation of chemical substances based on the differences in solubility at any given temperature. In the palm oil industry, there are three principal processes used in fractional crystallization: with detergent, with solvent and with neither. Dry fractionation, carried out without solvent, is relatively cheap and widely used to tailor the properties of the oil. Detergent fractionation is a modification of the dry method. Wet fractionation, carried out with solvent, is less widespread than dry, being a more expensive process. However, its cleaner separation of palm oil components means that it is necessary to use it where a more precise composition of the fractions is required.

Wet fractionation, crystallization from a solvent, allows a cleaner separation. This means that it is possible to obtain a 'purer' solid fraction and an increased yield. Wet fractionation is much more expensive than dry or detergent fractionation. Due to high costs, relative to dry and detergent processes, the wet fractionation technique is usually reserved for high-value applications. Solvents commonly used are hexane and acetone but other solvents such as isopropyl alcohol (IPA), have been proposed (Koslowsky, 1972; Koslowsky, 1973; Smith, 2001).

1.8 Crystallization of DHSA

In MPOB, palm oil based crude DHSA is subjected to purification in which it undergoes three steps: (i.) crystallization with solvent; (ii.) solvent removal; (iii.) drying process. Some of the unwanted C_8 and C_{10} acids present, which could cause irritation to the skin, are removed during the purification process. This process is currently under development.

IPA mixture with water (80:20) is used in the solvent crystallization step. The palm oil based crude DHSA while in liquid form is mixed with IPA/water (80:20) at 1:1 w/v ratio. The mixture of palm oil based crude DHSA and IPA/water is cooled at 5°C in order for the crystallization to occur. The DHSA crystals in wetted cake form are then separated from the mother liquor using a mechanical vibrating screen. The purity of DHSA obtained after the first crystallization process is about 70%. It is necessary to carry out a second crystallization in order to achieve purity above 80%. After removal of mother liquor through the vibrating screen, the purified DHSA is washed with water. The washed purified DHSA is then dried (Siwayanan *et al.*, 2004).

1.9 Problem Statements

In recent years, oleo-based chemicals have made its presence felt among the ever blooming personal care segment, slowly but gradually chipping into the industry once dominated by petrol-based chemicals. In order to have a share in this segment which

generates multi billion worth of returns every year, MPOB has successfully formulate a compound known as DHSA, a multipurpose intermediate in the synthesis of fine chemical products such as special and decorative cosmetics.

Currently DHSA produced in MPOB has purity ranging from 55% to 70%. This DHSA contains significant amount of C₈ and C₁₀ acids. In order for DHSA to be used for cosmetic applications, these two acids need to be removed as they are known to cause irritation. Solvent crystallization has been identified as the path to rid the DHSA of impurities and the minimum purity requirement has been benchmarked at 80%.

Since MPOB is one of the pioneers in the large scale production of DHSA, not to mention that palm oil based DHSA is a relatively new product, there exist an urgent call to identify the operating parameters needed to carry out an optimal solvent crystallization process. The crystallization carried out would need to yield the maximum quantity of DHSA crystals that have desirable particle properties (for re-melting and transportation purposes) besides meeting the minimum purity requirement.

1.10 Objectives

The objectives of this research were to study:

- i) The effects of temperature on the solvent crystallization of DHSA.
- ii) The effects of solvent quality and quantity on the solvent crystallization of DHSA.

1.11 Scope of Research

Crystallization is a delicate process. It is a process influenced by quite a number of interlinked key parameters, including crystallizer design, supersaturation, growth, chemical and temperature effects, crystal size and nucleation. Tempering with any one of these parameters and the overall outcome of the crystallization would be affected. The scope of this work covered the study of the effects such as temperature and solvent quality and quantity on the crystallization process of DHSA employing IPA as solvent. These properties had strong influence in particle size distribution (PSD) and solute purity of the final DHSA crystals. The particle properties play an important role when and if re-melting and transportation of the DHSA crystals need to be carried out while the solute purity is important in determining the suitability of DHSA usage in cosmetic applications.

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