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MORPHOLOGICAL STUDIES OF DHSA/DHSA-OCTYL ESTER/RBD PALM KERNEL OLEIN/MEDIUM CHAIN TRIGLYCERIDES SYSTEM.

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Key Words: Dihydroxystearic Acid, octyl dihydroxystearate, RBD palm kernel olein, medium chain triglycerides, ternary phase diagram.

Abstract

Dihydroxystearic acid (DHSA) and octyl dihydroxystearate (DHSA-octyl ester) have been previously prepared from palm oleic acid and preliminary results showed that these compounds are suitable in personal care and cosmetics products. The objective of this research is to study the phase behavior in ternary system of DHSA/DHSA-octyl ester/RBD Palm Kernel Olein (RBDPKOo) & MCT at 85°C. From the ternary phase diagram, results showed that all ratios of were completely in two-phase region with various concentrations of RBDPKOo and MCT. The phase changes were observed through polarizing light while the formation of texture was confirmed using polarizing microscope combination with heating. Needles and spherulite textures were found in this system.

Abstrak

Asid dihidroksistearik (DHSA) dan oktil dihidroksistearat (DHSA-oktil ester) telah disediakan oleh penyelidik terdahulu menggunakan asid oleik kelapa sawit dan hasil awal menunjukkan sebatian ini sesuai untuk bahan kosmetik dan hias diri. Objektif penyelidikan ini adalah untuk mengkaji kelakuan fasa sistem ternari DHSA/DHSA-oktil ester/RBD Palm Kernel Olein (RBDPKOo) & MCT pada 85°C. Berdasarkan gambarajah fasa ternari, hasil menunjukkan untuk semua nisbah DHSA/DHSA-oktil ester yang digunakan, menunjukan kewujudan kawasan dua fasa pada berbagai kepekatan RBDPKOo dan MCT. Kajian perubahan fasa dilihat melalui cahaya terkutub dan pembentukan tekstur disahkan dengan mikroskop terkutub bersama pemanasan. Tekstur jarum dan sferulit telah ditemukan dalam sistem ini.

Introduction

Dihydroxystearic acid (DHSA) and octyl dihydroxystearate (DHSA-octyl ester) are new oleo chemicals derivatives that have been successfully prepared from palm oleic acid via catalytic and enzymatic reaction respectively [1-3]. Previous study showed that the polymorphic structure formation could be produced in the mixture of DHSA and DHSA-octyl ester using medium chain triglycerides are resulted in excellent spreading properties in make up application [6]. Preliminary study indicated that these compounds are suitable in cosmetics as thickener or gelling agent for oily properties [5]. DHSA ester has a variety of uses; such as plasticizers, mold release agents, cosmetics (hair products, lipsticks and lotion) and as chemical intermediate compound [5]. The irritancy test confirmed that DHSA at below 5% is found to be non irritant. The aim of the present study is to find out the phases behavior and the texture formation when oil is added to the DHSA/DHSA-octyl ester.

Materials and Methods

Materials

Purified DHSA and octyl dihydroxystearate (DHSA-octyl ester) were obtained from Advanced Oleo chemical Technology Centre (AOTC) at Malaysian Palm Oil Board. The DHSA and DHSA-octyl ester characteristics are shown in Table 1. Figures 1 and 2 show the structures of both compounds. An oily phase, medium chain triglycerides (MCT) and RBD Palm Kernel Olein (RBDPKOo) was obtained from Southern Acid Sdn Bhd.

Preparation of ternary system of DHSA/DHSA-octyl ester/RBDPKOo&MCT

DHSA and DHSA-octyl ester were mixed at various weight ratios ranging from 0% to 100%. 0.5g of the mixture was placed in a 10ml sample tube and placed in a water bath maintained at 85°C to melt the mixture.

Approximately 0.01% by weight (or one drop) of (50/50) RBDPKOo and MCT was added into the mixture and homogenized using a vortex mixer (Heildoph, reax top). The samples were melted again and centrifuged at 4000 rpm for 10 minutes at 25°C (Mistral 1000). Each sample was repeatedly heated up and centrifuged for at least 3 times. The sample was then allowed to equilibrate in water bath at 85°C for at least one hour. The anisotropic region of the mixture was inspected visually through crossed polarizer. Additional RBDPKOo and MCT were then added to DHSA/DHSA-octyl ester mixture and the procedure was repeated until 100% RBDPKOo&MCT.

Polarized light microscopy and image analysis

The anisotropic region of the mixture was confirmed by using polarizing microscope in combination with heating (Olympus AX70).

Table 1: Characteristics of Pure DHSA and DHSA-octyl ester [1].

Parameter	DHSA	DHSA-Octyl ester
Hydroxyl value, *mgKOH/g*	309.3 ± 3.9	246.1 ± 3.5
Acid Value, *mgKOH/g*	180.3 ± 1.2	1.8 ± 0.1
Iodine Value, *mgKOH/g*	1.1 ± 0.2	0.3 ± 0.1
Saponification Value, *mgKOH/g*	178.5 ± 1.0	138.5 ± 0.7
Melting Point ^{0}C	90.6 ± 0.9	69.8 ± 0.2

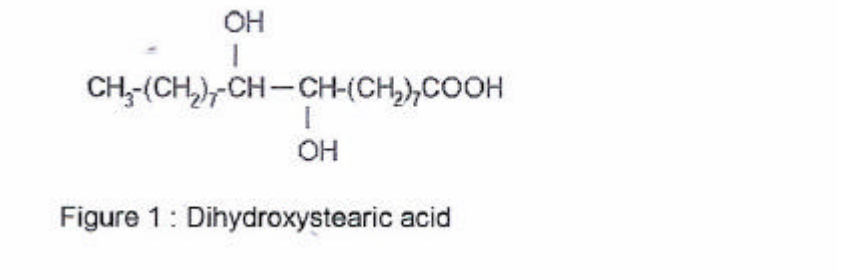


Figure 1 : Dihydroxystearic acid

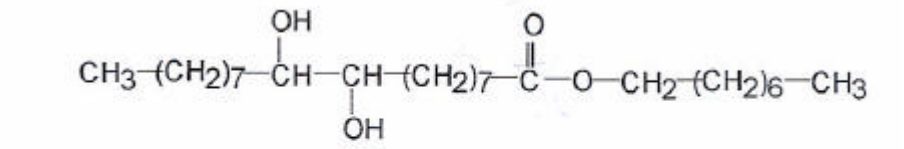


Figure 2 : Octyl Dihydroxystearate

Results and Discussion

Ternary Phase Diagram

The ternary phase diagram of DHSA/DHSA-octyl ester/RBDPKOo&MCT was shown in Figure 3. The result shows that all ratios starting from 0 to 100 of DHSA/DHSA-octyl ester/RBDPKOo&MCT was completely in two phase region. It was observed that percentage of RBDPKOo &MCT up to 50% showed mild separation and above this ratio the separation was very clear.

Polarized microscope

Further observation related to the crystal texture or texture formation of the mixture was closely studied separately. Several ratios of DHSA and DHSA-octyl ester mixture were prepared and titrated with up to 50%.

The two-phase region at 50% RBDPKOo&MCT was selected for the crystal texture observation under microscope. This is the right ratio for sample characterization because percentage higher RBDPKOo&MCT than 50% produced poor texture. Table 2 summarizes the mixture ratios and types of crystal formation. Needles, spherulite and the mixture of these two types of crystals were found at various ratios of. DHSA alone (100:0) exhibited a large densely packed spherulite and DHSA-octyl ester alone (0:100) a less densely packed needles is formed. In between these ratios, from 10:90 to 40:60 of DHSA/DHSA-octyl ester, the needles are increase and denser. While the ratio 50:50 of DHSA/DHSA octyl-ester, the small needles are aggregating into cluster form. With further increase from 60:40 to 70:30 of DHSA/DHSA-octyl ester it can be observed that the needles are broken down and scattered became small spherulite. Further increasing of DHSA from 80:20 to 90:10, the small spherulite became more prominent. Figure 4 shows the images for each sample at 50% RBDPKOo/MCT with a different ratio of DHSA/DHSA-octyl ester at 25°C.

Conclusion

Two types of crystal were found in DHSA/DHSA-octyl ester/RBDPKOo&MCT system. DHSA alone exhibited a large densely packed spherulite texture and DHSA-octyl ester alone formed less densely packed needles. A small densely packed spherulite texture was formed in 80:20 and 90:10 of DHSA /DHSA-octyl ester.

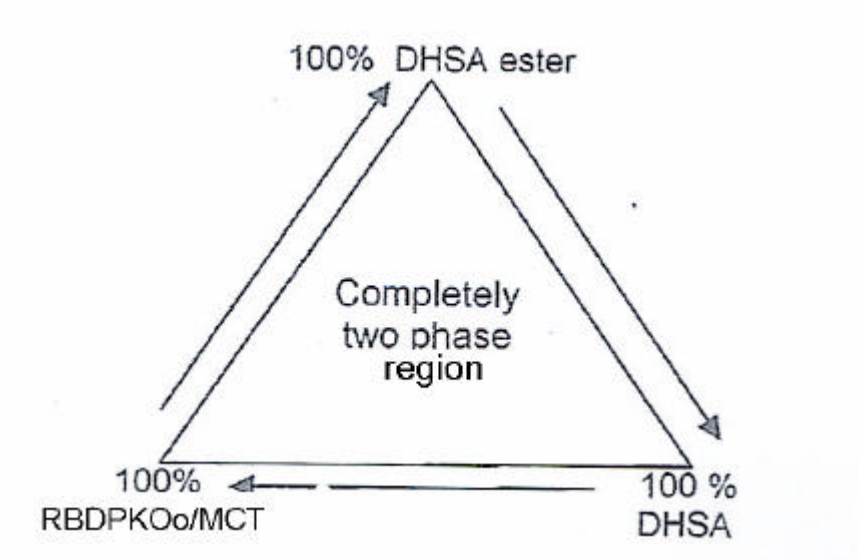
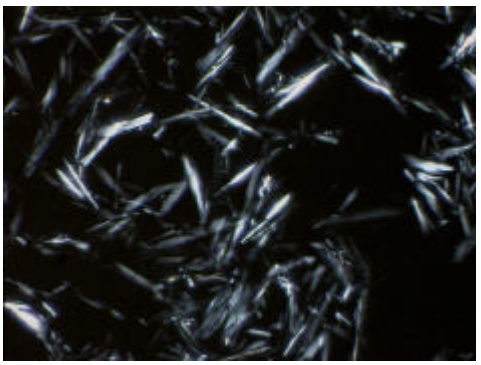


Figure 3: Phase diagram of DHSA/DHSA-octyl ester/RBDPKOo&MCT at 85°C

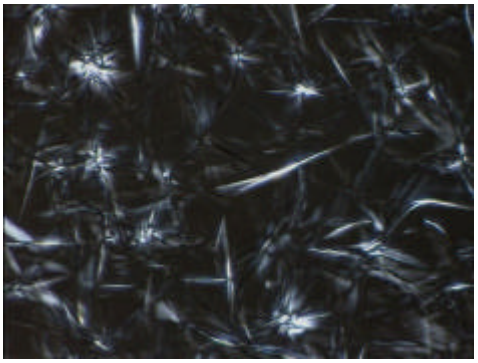
Table 2: Crystals textures of DHSA/DHSA-Octyl ester at 25°C with 50%RBDPKOo&MCT.

DHSA : DHSA-Octyl ester	Type Of Crystal
a) 0:100	Needles, less packed (less needles)
b) 10:90, c) 20:80, d) 30:70, e) 40:60, f) 50:50, g) 60:40	Needles, densely packed (more needles)
h) 70:30, i) 80:20	Small spherulite, less needles, densely packed
j) 90:10	Small spherulite, densely packed
k) 100:0	Large spherulite, densely packed

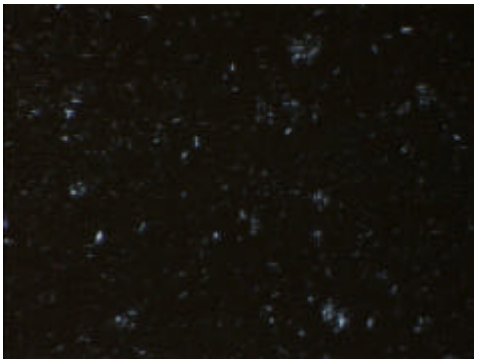
a) 0:100



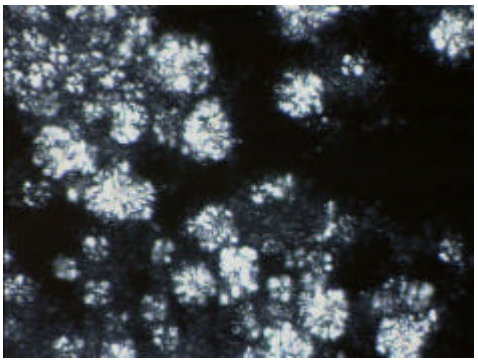
b) 10:90 c) 20:80 d) 30:70
e) 40:60 f) 50:50 g) 60:40



h) 70:30 i) 80:20



j) 90:10



k) 100:0

Figure 4: Crystal textures of DHSA/DHSA-Octyl ester at 25°C with 50% RBDPKOo/MCT, Magnification 100X; a) less densely packed needles (less needles) b-g) densely packed needles (more needles), h-i) densely packed small spherulite and less packed needles, j) densely packed spherulite, k) large densely packed spherulite.

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