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

Anuar Kassim1*, Rathidevi Nadarajan1, Zahariah Ismail2, Mohd Zaizi Desa1, Atan Mohd Sharif1, Dzulkefly Kuang1 and Md Jelas Haron1.

1Department of Chemistry, Faculty of Science, University Putra Malaysia, 43400 UPM Serdang Selangor.
2Golden Hope Research Centre, 42700, Banting Selangor.

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.

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].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>DHSA</th>
<th>DHSA-Octyl ester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydroxyl value, mgKOH/g</td>
<td>309.3 ± 3.9</td>
<td>246.1 ± 3.5</td>
</tr>
<tr>
<td>Acid Value, mgKOH/g</td>
<td>180.3 ± 1.2</td>
<td>1.8 ± 0.1</td>
</tr>
<tr>
<td>Iodine Value, mgKOH/g</td>
<td>1.1 ± 0.2</td>
<td>0.3 ± 0.1</td>
</tr>
<tr>
<td>Saponification Value, mgKOH/g</td>
<td>178.5 ± 1.0</td>
<td>138.5 ± 0.7</td>
</tr>
<tr>
<td>Melting Point, °C</td>
<td>90.6 ± 0.9</td>
<td>69.8 ± 0.2</td>
</tr>
</tbody>
</table>

**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](image)

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

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

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

Acknowledgement
The author would like to express her appreciation and thanks to the authors and the AOTD – MPOB staffs for their co-operation, guidance and encouragement to publish this paper.

References