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Thermal and Crystallinity Properties of Tengkawang Seeds Fat (*Shorea mecistopteryx* Ridley)

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***Abstract*—Objectives: Tengkawang (*Shorea mecistopteryx* Ridley) is one of the typical Kalimantan plants that can produce fat from the seeds. The tengkawang seeds fat is not yet fully known for its physico-chemical characteristics, especially its polymorphism. The purpose of this study was to determine the thermal and crystallinity properties of tengkawang seeds fat through thermal analysis using differential scanning calorimeter (DSC) and crystallinity analysis with x-ray diffractometer (XRD). Thermal analysis showed that tengkawang seeds fat has four peaks on the thermogram i.e. glass transition (0.43ºC), crystallization point (14.08ºC), eutectic point (23.55ºC), and melting point (37.22ºC). Crystallinity analysis showed that tengkawang seeds fat had high crystallinity at three peaks on the diffractogram. It was concluded that the tengkawang seeds fat has polymorphism. There are three crystalline forms, namely α, β' , and β. The α crystalline was formed at an angle of 6.88º and a temperature of 14.08 ºC. The β' crystalline was formed at an angle of 19.35º and a temperature of 23.55 ºC. The β crystalline was formed at an angle of 24º and a temperature of 37.22ºC.**

Keywords:* Shorea mecistopteryx *Ridley, polymorphism, DSC, XRD

I. INTRODUCTION

Tengkawang name of a fruit derived from the type of Shorea, included in the family Dipterocarpaceae and is known as illipe nut or borneo tallow nut [1]. Several types of tengkawang that grow in Kalimantan, including *Shorea stenoptera*, *Shorea mecistopteryx*, *Shorea seminis*, and *Shorea lepidota*. The type of *Shorea Mecistopteryx* produces fruit every year, while other types of Shorea produces every 3-5 years [2].

Tengkawang seeds can produce high-value vegetable fat which is one of Indonesia's export commodities [3]. Tengkawang seed fat is also called cacao butter substitute because it has physico-chemical properties similar [4]. Long carbon chain fatty acids contained in tengkawang seed fat and cocoa butter show more than one crystal form called polymorph. Polymorphs are caused by the structure of triacylglyceride molecules (TAG), diacylglycerides (DAG), monoacylglycerides (MAG), phospholipids, and additional types of additional groups that differ between the carboxyl groups of adjacent molecules causing a change in the angle of the slope of the crystal chain [5]. The form of fat polymorph is also influenced by external factors, such as melting temperature because it can increase the distance between atoms or between molecules in a crystal [6]. In addition, the rate of cooling temperature also affects the level of crystal nucleus formation and crystal size [7].

The use of tengkawang seed fat in pharmaceutical and cosmetic products is generally through the process of fusion. This will affect the stability of the polymorph in the fat. Therefore, the thermal characterization and crystallinity characterization of tengkawang seed fat is carried out to improve standards and quality as one of the raw materials that will produce quality products.

II. MATERIAL AND METHOD

A. Material

The material used in this research is tengkawang seed fat (*Shorea mecistopteryx* Ridley) obtained from Balai Besar Penelitian Ekosistem Dipterokarpa, Badan Penelitian, Pengembangan dan Inovasi, Kementerian Lingkungan Hidup dan Kehutana in Samarinda City Jl. A. Wahab Syahranie No. 68, Sempaja, Samarinda, East Kalimantan.

B. Method

Analysis using Differential Scanning Calorimeter (DSC)

A sample of 7.626 mg of tengkawang seed fat was added to the pan sample and one pan reference. Heating is performed starting at a temperature of -100 ºC to 100 ºC with a heating speed of 3 ºC / min, and a heat flow rate of 30 ml/min. After sample preparation, scanning and analysis are performed.

Analysis with X-Ray Diffractometer (XRD)

The sample weight used is 1.6 grams. This analysis was carried out at a diffraction angle range of 2θ 0-45º with a shift speed of 0.800º / sec using CuKα1 radiation = 1.54060 nm; Kα2 = 1.54439 nm) at a voltage of 40 kV and a current of 30 mA.

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III. RESULTS AND DISCUSSION

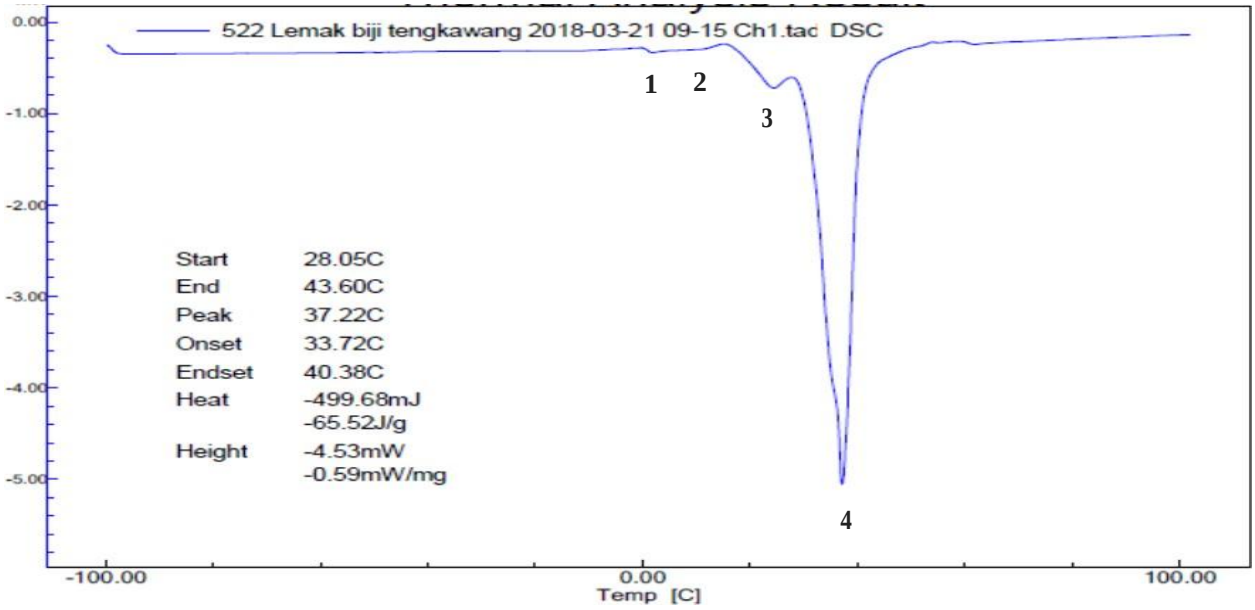

Figure 3.1 DSC Thermogram Analysis of Tengkawang Seed Fat

A. Result of Analysis Thermal using DSC

DSC analysis results have two axes, the Y-axis and the X-axis. The Y-axis represents the heat flow and the X-axis represents the temperature. Besides the peak, the thermogram also shows some specific information on tengkawang seed fat testing which can be seen in Figure 3.1.

Peak 1 shows at a temperature of 0.43 ºC glass transitions occur. Glass transitions are the beginning of a phase change called the glassy and rubbery phases. The glassy phase occurs before glass transitions, which is when the sample molecule is still in a rigid and solid-state. As the temperature increases, the heat capacity received by the sample also increases and causes the arrangement and movement of atoms in the sample to change. This indicates a change to the rubbery phase which is a state that is compatible or more flexible. The rubbery phase starts at 1.48 ºC above the glass transition temperature where the molecule will continue to move until it reaches equilibrium.

Peak 2 shows that at a temperature of 14.08 ºC crystallization process occurs. The process of crystallization is seen from the peak towards the top. The crystallization process occurs after the occurrence of glass transitions because the atoms will continue to experience movement with increasing temperature. When it reaches the right temperature, the atom will release energy that will turn into a phase of crystallization that can release heat. Therefore, the peak in the crystallization phase goes upward

Peak 3 shows that the temperature is 23.55 ºC is the eutectic point. Eutectic points generally occur in amorphous form. At a temperature of 23.55ºC, the solid phase and liquid phase have the same composition but not in a melting point temperature state.

Peak 4 shows the melting point of tengkawang seeds fat at 37.22ºC. Before experiencing the melting point phase, the sample undergoes a crystalline transition to amorphous which is accompanied by an increase in temperature, thus reaching the melting point phase which indicates the endothermic reaction. This happens because of an increase in movement between atoms which is influenced by thermal energy. The initial process of melting the sample at 33.72ºC and ending at 40.38ºC. After that, as the temperature continues to increase the molecular motion also continues to increase which causes the sample polymer to remain in the liquid state and has been amorphous.

Based on the thermogram data obtained, the reaction that occurs in tengkawang seed fat is an endothermic reaction. At the Peak 1 (glass transition), peak 2 (eutectic point), and peak 4 (melting point) downward direction, this is a parameter that the reaction that occurs is an endothermic reaction. If an endothermic reaction occurs, it is assumed that a change from one form to another is reversible or is called an enantiotropic. Changes like this are considered important because they can indicate changes in polymorphism.

B. *Result of Crystallinity Analysis with X-Ray Diffractometer*

Based on the results of XRD analysis, tengkawang seed fat is predicted to have several crystalline forms. This is based on the results of an analysis that shows several peaks at an angle of 2θ 5-24º. Some of these peaks stated that in fat Tengkawang seeds have 3 crystals. The difference in intensity and angle of 2θ on the diffractogram is due to differences in absorption and scattering by the atoms contained in the sample.

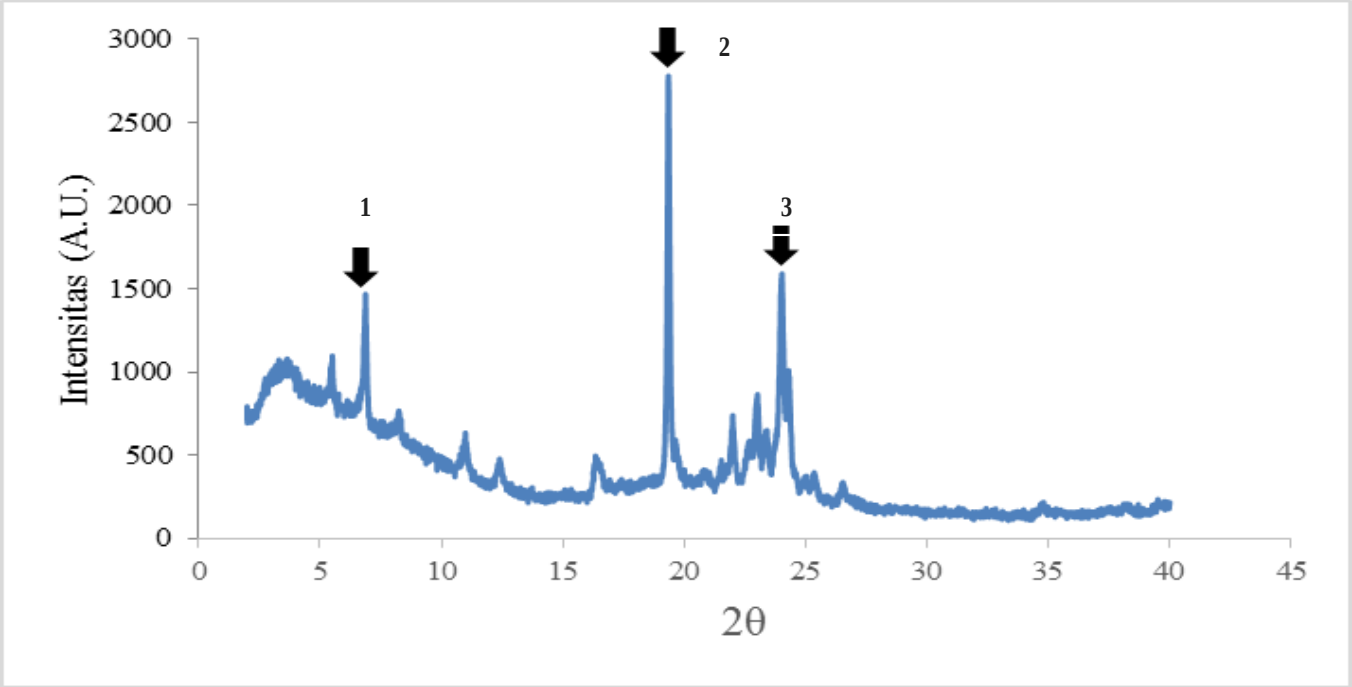

Figure 3.2. Tengkawang Seed Fat X-Ray Diffraction Pattern

The XRD analysis results in Figure 3.2 show some peaks and noise. Sharp peaks indicate the presence of crystals in the sample while noise usually indicates amorphous shape. Peak 1 at an angle of 6.88º with an intensity of 1468 cps, Peak 2 at an angle of 19.35º with an intensity of 2776 cps and a Peak 3 at an angle of 24º with an intensity of 1566 cps. This indicates that at such angles and intensities they have a similar arrangement of atoms that make up a plane of crystals whose sums vary each peak. The number of arrangements of these atoms affects the pattern of intensity. At an angle of 2θ 25-45º tengkawang seed fat does not show peak crystallinity, but is amorphous in shape with small curves and wide patterns.

IV. CONCLUSION

Tengkawang seed fat has polymorphism properties with three crystalline forms predicted to peak at 1, namely α with an angle of 6.88º, the intensity of 1468 cps and occurs at a temperature of 14.08 ºC, the second is β′ with an angle of 19.35 º, intensity amounted to 2776 cps and occurred at a temperature of 23.55 ºC, and the 3rd was β at an angle of 24º, the intensity of 1566 cps, and occurred at a temperature of 37.22ºC.

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