The Effect of Annealing Temperature to The X-Ray Diffraction Patterns of The Thin Film of Cardanol Compound from Alor Regency NTT Province

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Abstract

The effect of annealing temperature to the X-ray diffraction (XRD) patterns of the cardanol compound thin film from Alor regency, NTT Province has been conducted. The aim of this research is to investigate the effect of annealing temperature on XRD patterns of the cardanol compound thin film. The physics properties that can be determined based on its XRD patterns are crystal structure and crystallite size of cardanol compound thin film. The cardanol compound was obtained from cashew nut shield liquid, CNSL, that was extracted from cashew kernel. The cashew kernel was procured from Alor regency, NTT. This cardanol compound was spin-coated onto ITO – coated quartz substrate at 2000 rpm for 30 s. Cardanol compound was then, annealed on temperature of 250 °C, 300 °C, and 350 °C, respectively. Furthermore, it was analysed with XRD method to obtain its XRD patterns. Based on its XRD patterns analysis, some results can be obtained. They are: a) The effect of annealing temperature can change XRD patterns and crystal structure of cardanol compound thin film, b) the crystal structure of cardanol compound thin film that was annealed on temperature of 250 °C, 300 °C and 350 °C is monoclinic, monoclinic and orthorhombic, respectively. c) the average crystallite size of cardanol compound thin film that was annealed on temperature of 250 °C, 300 °C and 350 °C is 41.9 nm, 69 nm, and 53.2 nm.

Keywords: annealing temperature; cardanol compound thin film; XRD

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Introduction

At present, the research on physics properties of organic materials as alternative active material on electronics devices is currently growing rapidly. It is in consequence of the cost of organic material is cheaper than inorganic material and the fabrication process of electronics devices based on organic material is using spin coating and evaporator (Tryana, 2004; Ngara, 2006). One of the organic materials as local potential from Alor, NTT Province that has been investigated in this research is cashew nuts (Anacardium occidentale L). One of the compounds contained in the skin of cashew kernel is cardanol compound (Muljohardjo, 1999; Budiana, 2005). On June 2009, Ngara, et al., in competitive grant, have determined energy gap of cardanol compound and cardanol complex compound from province NTT. Based on their research result, energy gap of cardanol compound and cardanol complex compound from province NTT is 2.58 eV and 2.48 eV, respectively.

In 2010, Patty, has determined crystal structure, crystallinity and crystallite size of Cashew Nut Shield Liquid (CNSL). In this research, it will be investigated the effect of annealing temperature to XRD patterns of cardanol compound thin film that was annealed on temperature of 250 °C, 300 °C and 350 °C. Based on its XRD patterns, one can find crystal structure, crystallinity and crystallite size of cardanol compound thin film.

Cardanol compound

There are some districts in East Nusa Tenggarat that was producing of cashew plant, i.e. Kupang, Belu, Alor, East Flores, Sikka, West Sumba, and East Sumba (Ngara & Budiana, 2008). In classification systematic, cashew plant has Divisio: Spermatofita, Subdivisio: Agiospermae, Ordo: Sapindales, Familia: Anacardiaceae, Genus: Anacardium, Spesis: Anacardium occidentale L (Muljohardjo, 1999). The cardanol compound was obtained from CNSL that was extracted from cashew kernel (Budiana, 2005). Molecule structure of cardanol compound shown in Figure 1. There are some physics properties of cardanol compound and CNSL from NTT that have
been investigated in Laboratory of Physics of Faculty of Science and Engineering, Nusa Cendana University.

Figure 1. Molecular structure of Cardanol compound

They are:

a. In 2008, Ngara & Budiana have determined energy gap of CNSL.
b. In 2009, Lalus, al have investigated mechanics and property of CNSL from Alor.
c. In 2010, Ngara, al have determined efficiency of solar cell prototype based cardanol complex compound.
d. In 2012, Ngara, et al. have investigated the study on physics mechanism to increase efficiency of organic solar cell based cardanol complex compound.

Crystal structure and crystallite size

The patterns of XRD from a material can be used for determination crystal structure, crystal plane, crystallite size, etc. XRD method is based on Bragg's law, i.e. (Cullity, 1987)

\[ 2d \sin \theta = n \lambda \]  

(1)

Where \( d, \theta, n, \lambda \) is distance of crystal planes, diffraction angular, diffraction order, and wave length, respectively.

In order to find the crystal structure of a material, it is used Hanawalt card method. In this method, one compare between crystal plane distance from three strongest peaks of sample and standard material that have same value. The diffraction peaks of sample and standard material were obtained from its XRD patterns and PCPDFWIN program, respectively. If the values of crystal plane distance of sample material are the same of material standard, the crystal structure of sample is the same crystal structure of standard material.

In order to find crystallite size of a material, it is used Debye-Scherer equation, i.e. (Ngara, 2014)

\[ L = \frac{0.9 \lambda}{\sqrt{\left[FWHM_{ni}\right]^2 - \left[FWHM_{ini}\right]^2} \times \frac{\pi}{180^\circ} \times \cos \theta} \]  

(2)

where \( FWHM_{ni} \) is instrument FWHM.

Methodology

Preparation of cardanol compound

The preparation of sample includes extraction, distillation, evaporation and isolation process of CNSL.

Sample analysis by using XRD method

Cardanol compound was deposited on glass substrate with spin coating on 2000 rpm for 30 s. There are three samples cardanol compound thin film. Then, these samples were annealed on temperature of 250°C, 300°C, and 350°C, respectively. Furthermore, they were analysed by using XRD method in Chemistry Department, UGM, Yogyakarta. Based on their XRD patterns, crystal structure of cardanol compound can be found by using Hanawalt card method. Meanwhile crystallite size can be found by using Eq. (2).

Results and Discussion

XRD patterns of cardanol compound thin film that was annealed on temperature of 250°C, 300°C, and 350°C shown in Figure 2, Figure 3 and Figure 4, respectively. Based on Figure 2, The number of diffraction peaks of this sample is eight peaks. Three diffraction peaks that have strongest intensity are occurred on diffraction angular of 26.43°, 19.18°, and 18.76°. The distance of their crystal planesis 3.36 Å, 4.62 Å and 2.72 Å. Based on these crystal plane distance values and using Hanawalt card method with helping PCPDFWIN program, crystal structure of this cardanol compound thin film is the same crystal structure of 4,6-dinitroresorcinol (C₆H₄NO₃) with ID number is 05-0504. The crystal structure of 4,6-dinitroresorcinol (C₆H₄NO₃) is monoclinic. Therefore, the crystal structure of cardanol compound thin film that was annealed on temperature of 250°C is monoclinic. The strongest diffraction peak is occurred on diffraction angular of 26.43°. It is meant, on diffraction angular of 26.42°, cardanol compound has periodic layer structure or it has many crystal plane that have same Miller index.

The crystallite size of cardanol compound on diffraction angular of 26.43°, 19.18°, and 18.76° is 45.83 nm, 36.67 nm, and 43.14 nm, respectively. The average crystallite size of this sample is 41.88 nm.

Based on Figure 3, the number of diffraction peaks of this sample is five peaks. The strongest diffraction peak is occurred on diffraction angular of 18.94°. Three diffraction peaks that have strongest intensity are occurred on diffraction angular of 18.94°, 24.29°, and 25.08°. The distance of their crystal planes is 4.68 Å, 3.36 Å, and 3.55 Å, respectively. Based on these values and using Hanawalt card method with helping PCPDFWIN program, crystal structure of this cardanol compound thin film is the same crystal structure of Sodium bis (L-malato) borate with ID number is 46-1951. The crystal structure of Sodium bis (L-malato)
borate is monoclinic. Therefore, the crystal structure of cardanol compound thin film that was annealed on temperature of 300 °C is monoclinic.

Figure 2. XRD patterns of cardanol compound thin film was annealed on temperature 250 °C

Figure 3. XRD patterns of cardanol compound thin film was annealed on temperature 300 °C

Figure 4. XRD patterns of cardanol compound thin film was annealed on temperature 350 °C

Based on Figure 3, The crystallite size of this sample on diffraction angular of 18.94°, 24.29°, and 25.08° is 58.2 nm, 61.7 nm, and 87.1 nm, respectively. The average crystallite size of this sample is 69 nm.

Based on Figure 4, the number of diffraction peaks of this sample is fourpeaks. The strongest diffraction peak was occurred on diffraction angular of 34.57. Three diffraction peaks that have strongest intensity are occurred on diffraction angular of 34.57, 24.90°, and 26.59 with their crystal plane distances of 2.59Å, 3.57Å, and 3.35Å, respectively. Based on these values and using Hanawalt card method with helping PCPDFWIN program, crystal structure of this cardanol compound is the same crystal structure of Poly(chloro-1,4-phenylene-terephtalate) \((\text{C}_{14} \text{H}_{10} \text{ClO}_4)_n\) with ID number is 42-1680. The crystal structure of Poly(chloro-1,4-phenylene-terephtalate) \((\text{C}_{14} \text{H}_{10} \text{ClO}_4)_n\) is orthorhombic. Therefore, the crystal structure of cardanol compound that was annealed on temperature of 350 °C is orthorhombic.

Based on Figure 4, The crystallite size of this sample on diffraction angular of 34.57°, 24.90°, and 26.59 is 42.1 nm, 57.3 nm, and 60.3 nm, respectively. The average crystallite size of this sample is 53.2 nm.

**Conclusion**

The effect of annealing temperature can change the XRD patterns of cardanol compound thin film. Some diffraction peaks shift their positions and again some other peaks did not appear on annealing temperature of 300° and 350°. The crystal structure of cardanol compound thin film that was annealed on temperature of 250 °C, 300 °C, and 350 °C is monoclinic, monoclinic and orthorhombic. The average crystallite size of cardanol compound thin film that was annealed on temperature of 250 °C, 300 °C and 350 °C is 41.9 nm, 69 nm, and 53.2 nm, respectively.

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Presentation Discussion

Questions from:

- Umar: Why there is a peak in the graph (1) are flat?
- Indriana: What compounds in the sample? If a Cardanol is a Crystal or not?

Answers to:

- Umar: There is a flat peak in the graph (1) because no crystals are found, while in the other compound are found as crystals.
- Indriana: XRD cannot identify these compounds but can be aware of its existence. Cardanol is crystal when identify by GCPDS method but not always. It depending on the method that used.
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