



Effect of Doped Indium Tin Oxide on Bonding and Morphology of Monomer Cholesteryl Acrylate

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Monomer cholesteryl acrylate was doped by indium tin oxide (ITO) which succeeded in becoming composites in photopolymerization process using UV curing method having various polymerization time (15, 20, 25, 30 and 35 min) at 60-70 °C. The goal of this study is to know the effect of polymerization time to characterize the conductivity properties of composite cholesteryl acrylate-ITO. All the spectra confirmed the peaks at 600-550 cm⁻¹ which attributed the doping of ITO in the composite cholesteryl acrylate-ITO. The XRD patterns of polymer cholesteryl acrylate-ITO has nematic chiral phase at $2\theta = 15-20^\circ$. Some of the peaks are appeared at 15.2734° and 16.7266°, while other peaks of indium tin oxide appeared at 30°, 50° and 60°. The surface morphology of polymer cholesteryl acrylate-ITO showed the existence of sphere. The value conductivity of polymer cholesteryl acrylate-ITO is found to be 1.8390×10^{-10} S/m.

Keywords: Cholesteryl acrylate, Indium tin oxide, Photopolymerization.

INTRODUCTION

Process of photopolymerization is one of the polymerization techniques that use electromagnetic radiation beam as an initiator in the process of monomer initiation. This technique is called UV curing technique. One of the factors that affect process UV curing is ability of the radiation beam to be used. The source of radiation source is UV lamp. Cholesteryl acrylates have acrylate functional group that can be polymerized. Process of polymerizations monomer cholesteryl acrylate for making polymer cholesteryl acrylate. According to literature [1] polymer cholesteryl acrylate can be use for applications. To have maximal at application, in this study cholesteryl acrylate was doped by material conductor is indium tin oxide (ITO). Therefore, doped ITO could be increasing conductive properties of polymer cholesteryl acrylate.

The cholesteryl acrylate molecules are arranged as thin films and parallel to each other forming a helix continuously and regularly. Each layer in the cholesterol structure has a rigid parallel molecular direction on the top and bottom layers. Layers with the direction or orientation of the molecule will

be repeated and have a high degree of stability. The spaced molecular value of the twisted acrylates of acrylates is expressed by the pitch value (P) by undergoing a full 360°. The pitch value of the resulting crystalline cholesteryl acrylate will be the base when applied. Its value is strongly influenced by the temperature and addition of other substances (dopants) mixed with the cholesteryl acrylate. Substances added in this study are indium tin oxide (ITO) in which conductive oxide materials have transparent properties [1-3]. According to the literature successfully perform the photopolymerization of perfluorinated acrylic compounds using UV radiation with a wavelength range of 350-450 nm and heated using hotplate at a temperature of 160 °C for 1 h [4].

Addition of ITO into monomer cholesteryl acrylate while was polymerized can be a material can be using for many applications. ITO can be changed structures of monomer cholesteryl acrylate, especially that monomer cholesteryl acrylate was polymerized. Therefore, this paper reports the influence of ITO doped on bonding and morphology of monomer cholesteryl acrylate. For polymerizations process did variations of time polymerization on photopolymerizations are: 15, 20, 25, 30

and 35 min. In addition, this work analyzed how the effect of ITO dopant on the value of the conductivity of the resulting cholesteryl acrylate-ITO polymer [4].

EXPERIMENTAL

Materials used in this study include acrylic precursor synthesis, cholesterol, DMAP, DCC and ITO. The materials used for the synthesis of acrylic precursors are EHB, CBA, K_2CO_3 , KI, KOH, HCl, acrylate acid, hydroquinone and PTSA. Instruments for product analysis include Fourier transform infrared (FTIR), LCR-meter, scanning electron microscopy (SEM), energy dispersive X-ray (EDX), X-ray diffraction (XRD).

Synthesis of cholesteryl acrylate: A total of 2.00 g of acrylic precursor, acryloyloxy butyloxy benzoate (ABB), treated with 200 mL of dichloromethane solvent and 1.72 g of DCC, were then incorporated in a three round neck flask connected to a Liebig cooler. Previously, it was first conditioned to start with the stirrer at 0-5 rpm; C, then after pour in a rounded and mixed base flask, then distilled for 30 min at 0-5 rpm; C and flowed with N_2 gas. Then the added mixture of cholesterol as much as 3.00 g and DMAP as much as 0.2 g and then distirrer back for 5 h at room temperature. After that the filtered product and the formed filtrate are evaporated to form a brown precipitate. The adduct is added methanol and heated until dissolved. The resulting mixture is cooled in the refrigerator to form crystals and filtered. The resulting precipitate and filtrate are then purified by column chromatography using hexane:ethyl acetate = 5:1. The product formed is recrystallized with methanol solvent. Photopolymerization of composite cholesteryl acrylate-ITO at variations of time polymerization [5,6]. Sample preparations are made according to the five variations of time polymerizations. Each preparations sample consisted of 20 mg of monomer cholesteryl acrylate-ITO dissolved using a pure grade dichloromethane solvent in a small beaker. Then the solution was added 1 mL initiator of darocure 1173. Added dopant ITO much as 20 % and then the solution is oriented on the plate glass plate. Then the glass plate preparation is inserted into the UV-curing tool. The heating temperature at the hotplate is set at a temperature range of 60-70 °C. Polymerization process with time polymerization are 15, 20, 25, 30 and 35 min. The result of this process is a thin film material which will then be characterized.

RESULTS AND DISCUSSION

Monomer cholesteryl acrylate was added by ITO in process of photopolymerization. In generally that process known as the addition polymerization through the following steps: initiation, propagation and termination. The first step initiation is the formation of a cholesteryl acrylate radical of monomer. Propagation step is propagating of chain of polymer. The termination step is stop propagate of chain of polymer. All the step going the radiations process that sample of monomer cholesteryl acrylate and ITO was irradiated with lamp UV.

Process of photopolymerizations was performed at variations time of radiations, different polymerization time are 15, 20, 25, 30 and 35 min that temperature process at 60-70 °C. The photopolymerization process using 10 mg of a monomer of cholesteryl acrylate was dissolved in a pure grade dichloro-

methane solvent and then added an initiator of Darocore 1173 1 μ L and 20 % added dopan ITO the mixture was placed to a 2 mm \times 2 mm glass plate. Used Darocure 1173 as the initiator for working on ultraviolet light absorption areas with wavelengths between 225 to 375 nm this corresponds to the work of photo-initiators on photopolymerization using UV Curing method.

Analysis function group of polymer cholesteryl acrylate-ITO using spectrum FTIR (Fig. 1) showed bonding between cholesteryl with ITO. Based on spectrum product polymer in process time length radiation of 15 min until 35 min the peak shifting to bigger wavenumber. This phenomenon maybe because bonding between atomic carbon with oxygen more strength in molecule polymer cholesteryl acrylate-ITO. The peak in wavenumber of 1707.00-1701.21 cm^{-1} for vibration interaction between In-OH bond with C-O bond. This is because the interaction of the cholesteryl acrylate polymer with ITO surface. Based on the above IR absorption spectra analysis spectra that of the four samples the polymer cholesteryl acrylate-ITO contains two overlapping peaks in the 3398.57 cm^{-1} wavelength bands until 3334.92 cm^{-1} which is thought to be the vibration of the O-H bonds of the alcohol and carboxylates, it is assumed that the O-H peak of the alcohol originates from the initiator of Darocure 1173.

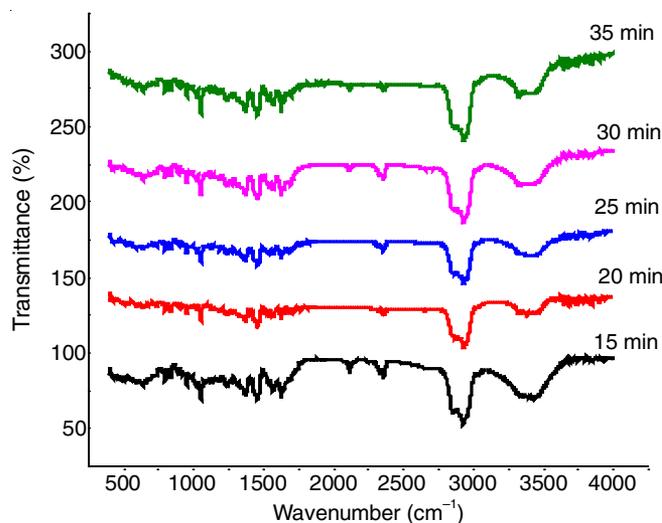


Fig. 1. FTIR spectrum of polymer cholesteryl acrylate-ITO from UV curing that resulted at 15, 20, 25, 30, 35 min time of polymerizations

There are some overlap peaks in wavenumber 1051.34 cm^{-1} , this is showed vibration C-O with peak for vibration In-OH in ChoA-ITO in spectrum polymer was resulted at 15 min. Other overlap in ChoA-ITO in spectrum polymer was resulted at 20 min in wavenumber 1051.94 cm^{-1} . And other in ChoA-ITO in spectrum polymer was resulted at 25 min in wavenumber 1053.51 cm^{-1} ; in ChoA-ITO was resulted at 30 min in wavenumber 1054.71 cm^{-1} ; in ChoA-ITO was resulted at 35 min in wavenumber 1057.11 cm^{-1} . That wavenumber which is assumed to be vibration of C=O carbonyl bond, according to Afrizal (2010) there is peak at 1729 cm^{-1} region which is carbonyl group. Then at the wavenumber 1550 cm^{-1} until 1690 cm^{-1} that predict is the presence of aromatic C=C bonds [7,8].

Characterizations of polymer cholesteryl acrylate-ITO by X-ray diffraction pattern can be seen in Fig. 2. In this paper

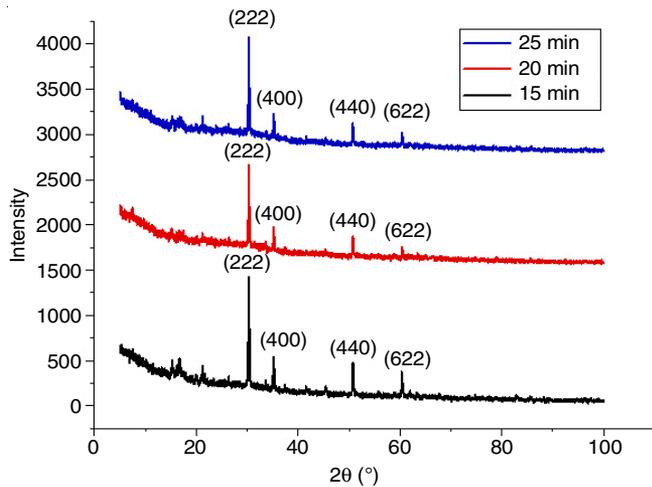


Fig. 2. Diffractogram of polymer cholesteryl acrylate-ITO from UV curing using lamp 25 watt and 40 watt

reported XRD that polymer was resulted at time polymerizations 15, 20 and 25 min. Based on diffractogram that polymer cholesteryl acrylate has phase nematic chiral at peak $2\theta = 15\text{--}20^\circ$. Some peaks appeared are 15.2734° and 16.7266° . Polymer cholesteryl acrylate-ITO was resulted at time polymerizations 20 min shows peaks appeared are 15.2267° and 16.7352° . Polymer that was resulted in time polymerizations 25 min shows peaks appeared are 15.2771° and 16.7256° .

Other peaks for identifications of ITO 2θ at 30° , 50° and 60° . Peaks at 2θ starting at 30° which is stated to be the peak of ITO crystallinity. Based on the top reference the characteristics of ITO are $2\theta = 30^\circ$ which has a crystal (222) oriented area of Bodycentered cubic (bcc) [9]. High intensity at peak XRD

assumed any interaction between cholesteryl acrylate with ITO it is proven on IR data appear peak at wavenumber region is about at 1700 cm^{-1} which is a carbonyl group is thought to emerge a new peak because the C=O bond interacts with In_2O_3 . The growth of ITO changes the intensity of the new structures formed at which the ITO peaks appear small and if the amount is less, then the crystallinity of polymer is also formed. The existence of ITO can change the crystal field orientation of the polymer cholesteryl acrylate-ITO, so the smaller ITO percentage is given the more crystalline peaks formed in the polymer, this is reinforced by Xu *et al.* [10] that the liquid crystal can be doped by a conductive oxide with a smaller percentage so that the crystalline peak of the liquid crystal is formed.

Fig. 3 show that the photo-SEM of polymer cholesteryl acrylate-ITO that existence of spheres on the surface of morphology, then the picture A shows the surface morphology in the sample which looks not yet fully mixed between the polymer and ITO seen the significant micrograph difference the form of bars and spheres in which ITO has a distinctive sphere-shaped morphology. Based on the reference [11-13] that the polymer cholesteryl acrylate has a bar shape with fibers that branch off and morphological structures form a crosslinked network or crosslinks after polymerization. SEM image of B and C the agglomerate has begun to appear on the surface between composite of polymer cholesteryl acrylate-ITO with the marked cross-linked tissue. It has been succeeded that the ITO material can be used as filler or filler or dopant on a polymer matrix *i.e.* cholesteryl acrylate polymer. Furthermore, in the photo D the surface morphology looks to be stacked together, presumably the pile is between the polymer cholesteryl acrylate-ITO and byproducts. EDAX results shows composition of

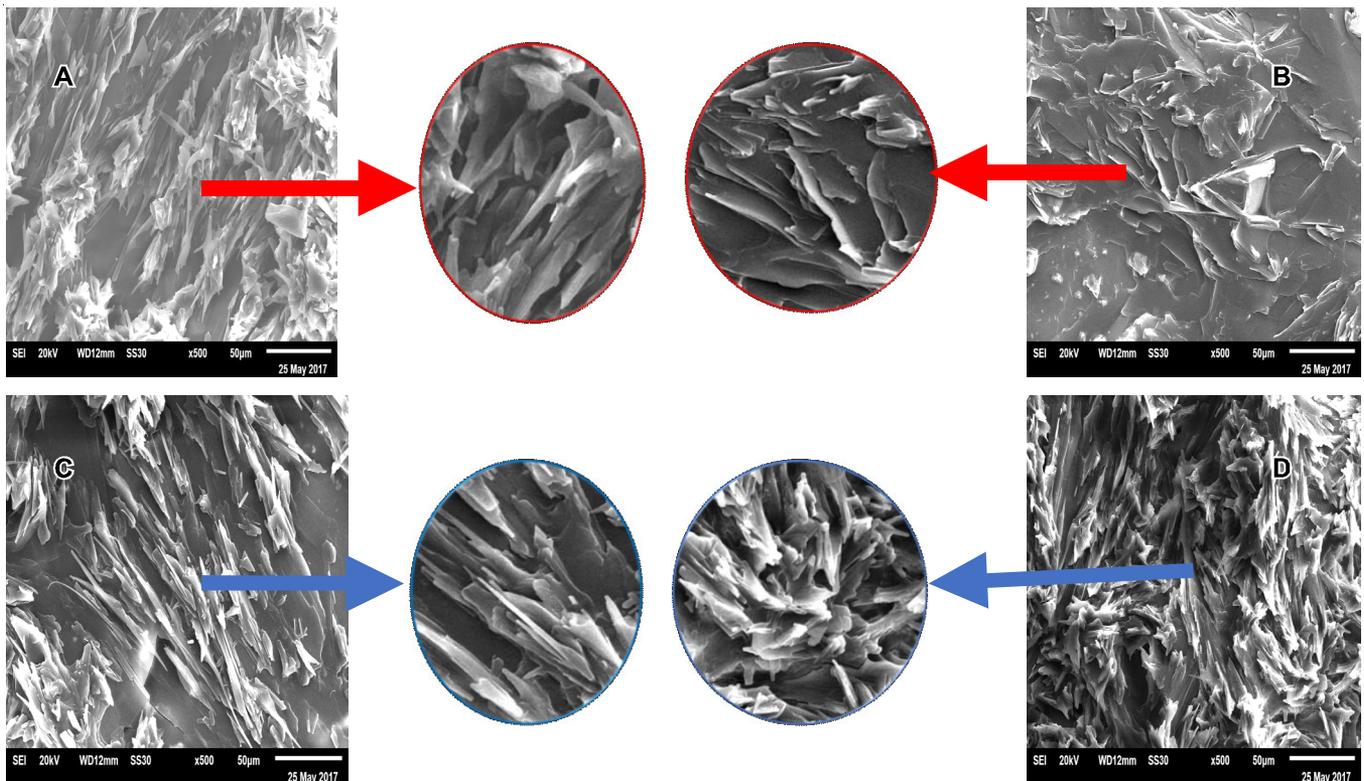


Fig. 3. SEM of polymer cholesteryl acrylate-ITO

polymer cholesteryl acrylate are carbon with mass amount 95.84 %, oxygen equal to 2.90 % while indium equal to 0.55 % [14,15].

Test of conductivity polymer cholesteryl acrylate-ITO showed that polymer was resulted in time polymerizations 20 min highest conductivity value is 1.8390×10^{-10} S/m. This phenomenon showed that 20 min time polymerizations is greater the energy absorbed by monomer cholesteryl acrylate in initiation process. Time photopolymerizations that longer result more than radical of monomer cholesteryl acrylate formation. Therefore, many process propagations happened between monomer with other molecule monomer cholesteryl acrylate. But in time polymerizations for 30 and 35 min showed that conductivity value a significant decrease. This is happen because the radical is formed faster so that the polymerization process more quickly occurs, the short-formed polymer chain [16,17].

Conclusion

Indium tin oxide (ITO) had influenced monomer cholesteryl acrylate by process UV curing founded polymer cholesteryl acrylate-ITO. UV curing method with variations of time polymerizations showed characterization different polymer of cholesteryl acrylate-ITO. Polymer cholesteryl acrylate-ITO was resulted by time polymerizations at 20 min that characterization by SEM have been agglomerated on morphological surface and there are elements of PKA-ITO *i.e.* carbon 95.84 %, oxygen 2.90 % and indium 0.55 % and it's conductivity value 1.8390×10^{-10} S/m.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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