Analysis of X ray diffraction spectra of cholesteryl acrylate-Indium Tin Oxide nanoparticle composites

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Abstract. Composites of cholesteryl acrylate with Indiun Tin Oxide (ITO) nanoparticle was made by UV curing technique at various the concentration of ITO nanoparticles. In this research using variation concentration of ITO nanoparticles. Polymer Cholesterol acrylate-Nanoparticle (Cho-ITO) at ITO 10% w/w showed peak for Cholesterol acrylate in 20 = 2,6782°; 5,2275°; dan 15,8050° with value of d spacing (001) was 32,96 Å. For ITO in 20 at 30,6244°; 35,5105°; 45,6814°; 51,0400°; dan 60,6473°, therefore pattern of XRD of Cho-ITO at ITO 20% w/w showed peak for cholesteryl acrylate in 20 at 2,6372°; 5,2022°; dan 15,7867° that value of d spacing lattice (001) is 40,09 Å. For ITO in 20 at 30,5685°; 35,4428°; 41,8550°; 50,9846°; 60,6252°, increasing value of d spacing for (001), that d spacing for cholesteryl acrylate 32,60 Å when added was by ITO 10% w/w dspacing increase untuil 32,96 Å. When added ITO 20% w/w value of d spacing until 40,09 Å.

1. Introduction

Composites of monomer cholesteryl acrylate with Indium Tin Oxide (ITO) by using UV curing photopolymerization is form of Liquid crystal polymer cholesterol acrylate-ITO. Polymer cholesterol acrylate has form Side-chain liquid crystalline polymers (SCLCPs). Many applications of polymer cholesterol acrylate special in display technology, specially in sensor technology. That because that properties of liquid crystalline can be response light polarizations, low molecule weight, thin, and fast response of electricity, and the energy needed low. Therefore liquid crystalline polymers of SCLCPs are an important research topic recently due to their unique optical properties [1].

ITO is one of the conductor material that can be used as a dopant that has give effect conduct for polymer of cholesterol acrylate. ITO as the conductive oxide material is has transparent properties that are expected to be polymer composites. That matter because the manufacture of composites can increase its conductivity. ITO is used as a dopant that has conductivity values. Polymer as nematic liquid crystals (NLCs) are well known for application in liquid crystal display (LCD) devices. Owing to their large optical anisotropy i.e. birefringence and dielectric anisotropy. Now, one of the most interesting properties in nano-science in recent years has been to find liquid crystal/nanocomposite [2].
It has been successful in photopolymerization research using the perfluorinic acrylate compound UV radiation with a wavelength range of 350-450 nm and heated using a hotplate at 160°C for one hour. Process photo-curable liquid crystal goal to the oriented liquid crystalline structures can be fixed by photopolymerization [3].

Cholesteryl acrylate showed liquid crystal phase at a temperature of 93.32°C based on measured using Melting Point. In the previous research using UV lamp on photopolymerization process that called is UV curing method. This research using a variety of UV light power including 10, 25, 40 and 55 watts. Therefore, goal to this research is the effect of lamp power on the characteristics and properties of composite conductivity polymer cholesteryl acrylate-ITO. Thin layer film polymer using polymides because of their good uniformity and stability. However, the polyimide layer formation process is complicated, time consuming, and lowers the transmittance of the resulting LC device because of thick layer thickness on ITO electrodes [4]. Whereas in the application of the latest modern technology, a conductive colesteric liquid crystal polymer is needed. One way to improve the conductivity properties of colesteric liquid crystalline polymers is to combine liquid crystals with a conductive dopant.

2. Material and methods
Material for this research using some chemicals are: Cholesterol, (S-(-)-2-methyl butanoic, hydroquinone, N,N'dicyclohexylcarbodiimid (DCC), N,N- dimethylpiridine-4-amine (DMAP), dichloromethane, PTSA (p-toluene-sulfonate), KOH, concentrated HCl, ethanol, methanol, benzene, KI, K₂CO₃, DMF, Acrylic Acid and petroleum ether which is obtained from Merck. After synthesis of cholesteryl acrylate, which characterization of cholesteryl acrylate monomer liquid crystal by using some instruments: FTIR, Electrothermal analysis, XRD, and DSC. Product monomer cholesteryl acrylate that resulted was blended with Indium Tin Oxide (ITO) nano particle and photopolymerizations. Product polymer cholesteryl acrylate-ITO nano particle was characterizations specially with XRD.

Photopolymerization using UV curing at temperature at 75°C. Time of radiations is 30 minutes and this research with variations power of lamp are 10, 20, 25, 40, and 55 watts. This method using previous research and has been modifications [5].

3. Result and discussion
Figure 1 shows an X Ray Diffraction pattern for polymer cholesteryl acrylate wherein from the figure it is known that a sharp peak of polymer cholesteryl acrylate appears at 2θ 2.7079°; 5,2682°; and 15,6200°. It can also be seen that the sample has the crystalline and amorphous phases observed from the intensity and nature of the peak produced. This is in accordance with the type of sample which is a group of liquid crystals that have a regular arrangement of molecules such as crystals but also exhibit properties such as liquids. The emergence of broad peaks with weak intensity at 2θ is 15-20°[6].

![Figure 1. Pattern of X Ray diffractions of cholesteryl acrylate.](image-url)
Figure 2 shows X-ray diffraction patterns for Cholesteryl acrylate-ITO nano particle composites (ITO 10% w/w) measured at 2θ (a) 0-20° and (b) 0-80°. From the figure, it is known that the sharp peak of cholesteryl acrylate appears at 2θ are 2.6782°; 5,2275°; and 15.8050° with the value of d spacing of field (001) obtained at 32.96 Å. While the typical ITO peak is observed at 2θ are at 30.6244°; 35.5105°; 45.6814°; 51.0400°; and 60,6473°. The diffraction pattern for the Cholesteryl acrylate-ITO nano particle composite (ITO 20% w/w). The X-ray diffraction pattern shows a sharp peak of polyesteryl acrylate that appears at 2θ are 2.6372°; 5,2022°; and 15.7867° with the value of d spacing in the field (001) of 40.09 Å. The typical ITO peak was observed at 2θ are at 30,5685°; 35,4428°; 41,8550°; 50,9846°; 60,6252°.

Figure 2. Pattern of X Ray Diffractions of for Cholesteryl acrylate-ITO nano particle composites (ITO 10% w/w) measured at 2θ (a) 2-20° and (b) 5-80°.

From the diffraction pattern, it can be seen that the sample has a crystalline and amorphous phase in its structure, but the amorphous phase of the Cholesteryl acrylate-ITO nano particle composite (ITO 20% w/w) appears to be greater when compared with the Cholesteryl acrylate-ITO nano particle composite (ITO 10% w/w). The reduction in crystallinity in the sample was also observed from the decrease in X-ray diffraction intensity in the sample so it can be concluded that the Cholesteryl acrylate-ITO nano particle composite (ITO 10% w/w) had higher crystallinity compared to the Cholesteryl acrylate-ITO nano particle composite (ITO 20% w/w). This was confirmed by the increase in the value of spacing for the field (001), where the value of d spacing for the polymeresteryl acrylate polymer was 32.60 Å and when added to ITO 10% w/w d spacing increased by 32.96 Å. But the d spacing value is greater when the addition of ITO is 20% w / w which is up to 40.09 Å. This proves that the ITO nanoparticles have filled the space between the crystalline fields, causing an increase in the d spacing value of the polymeresteryl acrylate polymer. The value of d spacing which is too large when added by ITO 20% w / w causes the composite structure to be more amorphous due to changes in the arrangement of molecules in the sample [7]. Therefore, in Figure 3 showed pattern of X Ray Diffractions of Cholesteryl acrylate-ITO nano particle for variation power lamp (0 watt, 25 watt, and 40 watt). All of patterns no different significant, only at 2 thetas in about 5 any small peak for polymer was resulted at 40 watt and 25 watt.
Figure 3. Pattern of X-ray diffractions of cholesteryl acrylate-ITO nano particle for variation power lamp (0 watt, 25 watt, and 40 watt).

Based on the diffraction pattern above, the three samples showed peaks at 2θ starting at 30° the stated peak is the peak ITO crystallinity. Based on the peak reference the characteristics of ITO are 2θ = 30° who have a crystal field (222) Body-centered cubic oriented (bcc) (Khuram, 2014). Pattern of XRD Pattern of Cholesteryl acrylate-ITO nano particle for 20 and 40 watt samples a new peak appears at 2θ is 7° with high intensity which is assumed to be a cholesteryl acrylate interaction with ITO. ITO growth change the intensity of the new structure formed where at the top of the ITO It looks small, that is the crystallinity of ITO and the amount below the crystallinity of the polymer formed. The ITO can change the orientation of the crystal field from Polymer cholesteryl acrylate-ITO, so that the smaller the percentage of ITO given, the more crystalline peaks formed on the polymer. Crystallographic fields for mathematical phases namely (100), (200), and (300) [8]. Chiral nematic phase will give a peak in area 15-20°. The characteristic peaks of the ITO obtained were 2θ = 30° which had a crystal field (222) Body centered cubic oriented (bcc). The best peak produced is from the 40 Watt UV lamp power.

4. Conclusion
This research has been successfully synthesis composites of cholesteryl acrylate liquid crystal-Indium Tin Oxide (ITO) nano particle. From the diffraction pattern, it can be seen that the sample has a crystalline and amorphous phase in its structure, but the amorphous phase of the Cholesteryl acrylate-ITO nano particle composite (ITO 20% w/w) appears to be greater when compared with the Cholesteryl acrylate-ITO nano particle composite (ITO 10% w/w). The reduction in crystallinity in the sample was also observed from the decrease in X-ray diffraction intensity in the sample so it can be concluded that the Cholesteryl acrylate-ITO nano particle composite (ITO 10% w/w) had higher crystallinity compared to the Cholesteryl acrylate-ITO nano particle composite (ITO 20% w/w).

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