Fourier Transformed Infrared (FTIR) spectroscopy for analysis of cholesteryl acrylate liquid crystall-indium tin oxide composites

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Fourier Transformed Infrared (FTIR) spectroscopy for analysis of cholesteryl acrylate liquid crystall-indium tin oxide composites

A Afrizal*, A Rahman, F V Florencia and L Jati
Department of Chemistry Faculty of Mathematics and Natural Science, Universitas Negeri Jakarta, Gedung KH. Hasjim Asj’arie Lantai 1 Jl. Rawamangun Muka Jakarta 13220 Indonesia

* afrizal@unj.ac.id

Abstract. The aim of this research is to synthesis composites and polymerizations of cholesteryl acrylate with dopan of Indium Tin Oxide (ITO). Composite of cholesteryl acrylate and ITO have succeeded in becoming a composite with blending process in variations concentrations of ITO. The photopolymerization process by UV curing method with variation of UV lamp power that is 10, 25, 40 and 55 watt. Analysis of functional group with FTIR showed vibration of In-OH bond with C-O at wave number 1056,99 cm⁻¹. Other peak in wave number 2929,87 cm⁻¹ for vibration of groups in bond C-H in vinyl, wave number at 2860,01 cm⁻¹ for vibration bond of C-H alyfatic. Spectrum FTIR of ITO showed at wave numbers: 437,84; 487,99; 534,28; 619,15 cm⁻¹. Therefore, after photopolymerizations process, peak at 2100 cm⁻¹ has disappeared in spectrum FTIR of Cholesteryl acrylate which showed molecule of cholesteryl acrylate had polymerized.

Conductivity of composite of cholesteryl acrylate and ITO was determined using by LCR meter. In general conductivity value and permittivity of composite of cholesteryl acrylate and ITO increase with the increasing of the concentration of dopan ITO was added to the composites. SEM images of composite of cholesteryl acrylate and ITO show that pores of cholesteryl acrylate had been filled by ITO particles. The SEM-EDX data show that the content of the elements contained in the composite cholesteryl acrylate and ITO are: C 76.91%, O 3.13% and In 5.13%. The value conductivity (σ) of composite of cholesteryl acrylate-ITO is 4.078 x 10⁻¹⁰ S/m, therefore polymer of cholesteryl acrylate-ITO of σ is 11.2 x 10⁻¹⁰ S/m.

1. Introduction
Cholesteryl acrylate is one of the liquid crystal which has that interesting physical and chemical properties. Cholesteryl acrylate has been successfully made and was analyzed. Cholesteryl acrylate can showed optic properties, specially being sensor for electromagnetic radiation [1]. Synthesis of cholesteryl acrylate according to reference from Merlo et. al., 2001 which had been modified [2]. Combination of cholesteryl acrylate with liquid crystal of benzoic ester type, showed the product could be absorp ultraviolet area with wave length in 460 nm-820 nm [3]. Combination of two materials or more using blending technique [4]. Based on result of research previous, therefore this research focused on synthesis and characterization of composite of cholesteryl acrylate-indium tin oxide (ChoA-ITO). Special that’s characterization is conductivity properties.
This research focus on cholesteryl acrylate which combined with indium Tin Oxide (ITO). The material to combine in this research advantage for give effect of electric on polymer liquid crystal cholesteryl acrylate. The material that choice is ITO, because ITO is one of the materials for give conductivity for polymer material. Therefore, some research had been using ITO for coating of cholesteryl acrylate [5]. Research on combine of liquid crystal nematic with dopan of Indium Tin Oxide (ITO) can decrease resistivity from \(7.73 \times 10^{11}\) until \(1.21 \times 10^{11}\) \(\Omega/m\) [6]. ITO have conductivity high in the report of doudi et.al. [7] the conductivity was \(567\) S/m. Therefore, the goal of this research are studies and characterizations of composite Cholesteryl acrylate-ITO (ChoA-ITO).

2. Material and methods
Chemicals for synthesis of cholesteryl acrylate: Cholesterol, \((S-(-)-2\)-methyl-butanoic, hydroquinone, N, N'dicyclohexylcarbodiimidy (DCC), N, N- dimethylpiridine-4-amine (DMAP), dichloromethane, PTSA (p-toluene-sulfonate), KOH, concentrated HCl, ethanol, methanol, benzene, KI, \(K_2CO_3\), DMF, Acrylic Acid and petroleum ether which is obtained from Merck. The first step were synthesis and characterization of cholesteryl acrylate monomer liquid crystal. The second step, blending process of cholesteryl acrylate with Indium Tin Oxide (ITO). The third steps, testing of conductivity properties of blended product. The second step was blending process between cholesteryl acrylate monomers with ITO using dichloromethane solvent and using variations of ITO concentration were: 0, 10, 20, 30, 40, and 50 % w/w and then was printed on the surface of the glass that has been coated with Polyimide. Then heated at a temperature of 55°C for 30 minutes to remove the solvent. Measuring of conductivity on sample of thin film composite of cholesteryl acrylate-ITO using LCR-meters on frequency 1 kHz was conditioned.

3. Result and discussion
Monomer Cholesteryl Acrylate was synthesized using method of steglich esterification, which reaction between precursor acrylate with cholesterol. Acryloyloxy buthiloxy bezoic acid as precursor acrylate was produced by reaction between acrylate acid with hydroxy buthiloxy benzoic. Characterizations of cholesteryl acrylate using spectrophotometer FTIR in Figure 1, showed a peak at wave number 2929.87 cm\(^{-1}\) for vibration of groups in bond C-H in vinyl, wave number at 2860.01 cm\(^{-1}\) for vibration bond of C-H alyfatic. There were two sharpest peaks that for identification of many groups of C-H (sp\(^2\) and sp\(^3\)). Therefore, for group of C=C at 1405.51-1649.14 cm\(^{-1}\).

![FTIR spectrum of cholesteryl acrylate](image)

**Figure 1.** FTIR spectrum of cholesteryl acrylate.

Composite ChoA-ITO was prepared in photopolymerization process that variation of concentration of ITO were 0, 10, 20, 30, 40, 50 % w/w. UV curing process in this research at temperature 75°C, this
temperature based on reference before by Hikam, Soegiyono, & Riswoko [3]. Product blend of ChoA-ITO placed at thin plat with thickness was 2,392 mm and cross-sectional area was 400 mm$^2$.

In general, the conductivity value increased with increase dopan ITO was added for blending, but at ITO 20%, the value conductivity decrease (Figure 2). This result may be because dopan ITO fill pores of cholesteryl acrylate, which this is conditions explained reach the threshold of doping process. So, it was valued conductivity of sample decreased. But at ITO concentration were 30%, 40%, and 50%, value conductivity of Cho-ITO was going back up. This is showing was blending more polarized in compare only cholesteryl acrylate [9]. Each material was doping has a threshold of conductivity value of dopan concentration. Therefore decrease conductivity value at ChoA-ITO at composition in 20% [10].

![Figure 2. Concentration of samples ChoA-%ITO versus conductivity.](image)

**Table 1.** Concentration of samples ChoA-%ITO versus conductivity.

<table>
<thead>
<tr>
<th>ITO (w/w, %)</th>
<th>$\sigma \times 10^{-10}$ (S/m)</th>
</tr>
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<tbody>
<tr>
<td>0</td>
<td>9.94</td>
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<tr>
<td>10</td>
<td>13.0</td>
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<tr>
<td>20</td>
<td>11.2</td>
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<tr>
<td>30</td>
<td>13.6</td>
</tr>
<tr>
<td>40</td>
<td>17.2</td>
</tr>
<tr>
<td>50</td>
<td>25.1</td>
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</tbody>
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Spectrum FTIR of ChoA-ITO in Figure 4, don’t differ with a spectrum of FTIR cholesteryl acrylate, only shifting some wave number. Peak of spectrum FTIR of Cho-ITO at finger print at 447.49; 513.07; 547.78; 596.00 cm$^{-1}$. That peaks on shifting from spectrum FTIR of ITO (Figure 3). Spectrum FTIR of ITO at wave number 437.84; 487.99; 534.28; 619.15 cm$^{-1}$. Peak at 2100 cm$^{-1}$ has disappeared in spectrum FTIR of Cholesteryl acrylate which showed molecule of cholesteryl acrylate had polymerized. Spectrum of cholesteryl acrylate showed a peak for function group of C-O. Therefore, on spectrum of ChoA-ITO was confirmed bonding between Indium with OH.

4. Conclusion
Liquid crystal of cholesteryl acrylate has been combined with dopan Indium Tin Oxide (ITO) using blending techniques. Thin layer blend ChoA-ITO was characterized are physical and chemical properties. The conductivity value composite of ChoA-ITO increase with increase of ITO concentration with a threshold value of doping concentration ITO in blending at 20% w/w. Characterization of SEM showed was doped of ITO filled pores of cholesteryl acrylate. FTIR spectrum showed any interaction between cholesteryl acrylate with ITO.
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