3 MATERIALS AND METHOD

3.1 Overview

This chapter describes about the materials and methods used to conduct the present research study. The three parts in this chapter consisting the description of materials experimental procedures and product analysis.

3.2 Materials

3.2.1 Materials of esterification

Acrylic Acid (99%), 2-Ethyl Hexanol (99.6%), and Polymerization Inhibitor, Phenothiazine were purchased. Acrylic Acid and 2-Ethyl Hexanol as the reactant were mixed in the presence of water. Phenothiazine was used to inhibit the acrylic acid polymerization. Amberlyst 15(dry) as the strongly ion exchange resin that have 4.7 meq H+/g by dry weight was used as catalyst. All the materials used in the present study are summarized in Table.

<table>
<thead>
<tr>
<th>NO</th>
<th>NAME OF CHEMICALS</th>
<th>TYPES</th>
<th>BRANDS</th>
<th>PURITY</th>
<th>PURPOSE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Amberlyst 15</td>
<td>Solid</td>
<td>SIGMA ALDRICH</td>
<td>99%</td>
<td>Catalyst</td>
</tr>
<tr>
<td>2</td>
<td>Acrylic Acid</td>
<td>Liquid</td>
<td>SIGMA ALDRICH</td>
<td>99%</td>
<td>Reactant (Carboxylic acid)</td>
</tr>
<tr>
<td>4</td>
<td>2-Ethyl Hexanol</td>
<td>Liquid</td>
<td>SIGMA ALDRICH</td>
<td>99.6%</td>
<td>Reactant (Alcohol)</td>
</tr>
<tr>
<td>5</td>
<td>Phenothiazine</td>
<td>Solid</td>
<td>SIGMA ALDRICH</td>
<td>99%</td>
<td>Polymerization inhibitor</td>
</tr>
</tbody>
</table>
3.2.2 Materials of product analysis

Hexane GC grade (≥98.5%) will be purchased and used as a solvent for the product analysis using gas chromatography to identify the chemical composition of the sample.

3.3 Experimental Procedures for Esterification Reaction

The preparation of reaction mixture from dilute acrylic acid and 2-ethyl hexanol were carried out following the method described in Ahmad et al., (2014). 6.4 ml sample of acrylic acid (AA) and 43.6 ml of 2-ethyl hexanol(2-EH) with the molar ratio of AA: 2 EH of 1:3 were prepared first. The amount of water with respect to acrylic acid were varied to form the dilute acrylic acid with the range of 10-90 wt% of water. The esterification was conducted in a three neck flask equipped with condenser, temperature controller and temperature probe. 2-EH charged into the flask and heated to desired temperature separately. The acrylic acid and the water were added until the temperature maintained and start the reaction. The catalyst was loaded after the temperature maintained. The reaction mixture stirred using magnetic stirrer. 5wt% of MEHQ which respect to acrylic acid as polymerization inhibitor and 10 wt% of Amberlyst 15 were added to the mixture. The mixture stirred at 500rpm and heated. The reaction temperature was controlled at 80°C. The condenser was attached to the vessel to reflux water. The experiment was run for 6 hours and the sample was collected for every 1 hour. The experiment was repeated without using polymerization inhibitor. All the other parameters remained the same.
3.4 **Product analysis**

3.4.1 **2-Ethyl hexylacrylate sample analysis**

The sample was collected every 1 hour and analyzed using gas chromatography (GC) equipped with a flame ionization detector (FID) and DB-200 column in order to detect the composition of acrylic acid, 2-ethyl hexanol and 2-ethyl hexylacrylate in the sample. The calibration curve was generated by GC and after getting the result, the yield and conversion of product will be calculated using equation below:

\[
yield(\%) = \frac{C_{2EHA}}{C_{AA0}} \times 100\%
\]

\[
conversion(\%) = \frac{C_{AA0} - C_{AA}}{C_{AA0}} \times 100\%
\]

Where:

- \( C_{2EHA} \) = concentration of 2-ethylhexyl acrylate
- \( C_{AA0} \) = initial concentration of acrylic acid
- \( C_{AA} \) = final concentration of acrylic acid

3.4.2 **Amberlyst 15 sample analysis**

In order to examine the morphology of Amberlyst 15, scanning electron microscopy (SEM) was used for fresh and used catalyst after the reaction. Another testing were fourier transform infrared spectroscopy (FTIR) had been used to check the functional group of catalyst and nitrogen physisorption analyzer also had been used to catalyst surface area (Ahmad et al., 2014). The working principle of each instrument for characterization of the catalyst can be shown as below:

3.4.2.1 **Scanning Electron Microscopy (SEM)**

In analysing the morphology of the catalyst, scanning electron microscope method was used (Carl Zeiss EVO50). The catalyst was dried at 100 °C for 6 hours times and stored in desiccators in order to eliminate the moisture. Then by using a carbon conductive pad, the samples were mounted on a metal stub and the silver metals acts as conductor were placed at both sides the sample. Lastly, under vacuum state and in an argon atmosphere, the samples were coated with gold and were observed.