3 MATERIALS AND METHODS

3.0 Overview

This experiment was conducted to characterize extraction kinetics of FAEE from oil phase by using aqueous ethanol and to determine the distribution coefficient of FAEE between oil phase and aqueous ethanol. This experiment was conducted with three different oil:ethanol molar ratio which is 1:1, 1:3 and 3:1 where oil was firstly diluted with FAEE. The mixture was heated and stirred with different stirred time range between 1-20min. A settling time for the mixture was recorded and a sample was taken for FAEE concentration determination using gas chromatography (GC) analysis. Graph of FAEE concentration against stirred time will be plotted and analysed.

3.1 Introduction

The kinetics of solvent extraction is a function of both the various chemical reactions occurring in the system and the rates of diffusion of the various species that control the chemistry of the extraction process. For solvent extraction systems, there are two additional complications. First, the chemical reactions can take place in two bulk phases, since it dealing with two immiscible liquid layers. Second, the chemical reactions can occur in the two dimensional region called the liquid-liquid interface that separates the two immiscible liquids.

3.2 Chemicals

Table 1: List of chemicals used

<table>
<thead>
<tr>
<th>No</th>
<th>Chemicals Name</th>
<th>Type</th>
<th>Purify</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Absolute ethanol</td>
<td>Liquid</td>
<td>99%, 0.154wt% water</td>
</tr>
<tr>
<td>2</td>
<td>Cooking oils</td>
<td>Liquid</td>
<td>100%</td>
</tr>
<tr>
<td>3</td>
<td>High performance liquid chromatography (HPLC)-grade n-heptane</td>
<td>Liquid</td>
<td>99%</td>
</tr>
<tr>
<td>4</td>
<td>Potassium hydroxide pellets</td>
<td>Solid</td>
<td>100%</td>
</tr>
<tr>
<td>5</td>
<td>Ethyl oleate</td>
<td>liquid</td>
<td>99%</td>
</tr>
</tbody>
</table>
3.3 Preparation of ethyl oleate calibration curve

3.3.1 Samples preparations

A sample of ethyl oleate was diluted first using n-heptane to achieve 3000 ppm concentration in 3 ml vial. The calculation as below:

\[ V_{EO} + V_{nH} = 3 \text{ml} \quad \text{(1)} \]

\[ V_{EO} = \frac{nEO(MWEO)}{\rho_{EO}} \quad V_{nH} = \frac{nnH(MWnH)}{pnH} \]

\[ \frac{nEO}{nEO + nnH} = 0.003 \quad \text{............... (2)} \]

\[ V_{EO} = \frac{0.003}{0.997} \frac{nnH(MWEO)}{\rho_{EO}} \\
\frac{0.003}{0.997} \frac{nnH(310.51g/mol)}{0.87g/ml} + \frac{nnH(100.21g/mol)}{0.684g/ml} = 3\text{ml} \]

Therefore, \( n_{nH} = 0.02 \text{mol} \)

\[ V_{nH} = 2.93\text{ml} \]
\[ V_{EO} = 0.02\text{ml} \]

Another samples with concentration 1500 ppm was prepared in 1 ml vial by dilute the first sample of 3000 ppm with n-heptane by using calculation as below:

\[ M1V1 = M2V2 \]

Where \( M1 = 3000 \text{ppm} \) \( V1 = x \)
\( M2 = 1500 \text{ppm} \) \( V2 = 1 \text{ml} \)

\( x = 0.5 \text{ml} \)

Hence, 0.5 ml sample of 3000 ppm concentration was diluted with 0.5 ml n-heptane to achieve 1500 ppm concentration. Another sample with different concentration which is
1000ppm, 500ppm and 200ppm was prepared using same way. The concentration of each sample will be measured using GC analysis. A graph of area under gas chromatography curve against concentration of ethyl oleate solution was prepared.

3.3.2 Flame ionization detection (FID) analysis

FID device was used to obtain concentration measurements of study samples. Twenty millilitre (20um) aliquots will be diluted in 1ml of n-heptane before GC analysis. External standard calibration will be used for quantification. The GC device will be fitted with a Restek MXT Biodiesel TG column (15m x 0.32mm ID x 10um) and employed both a temperature program (50-380°C) and pressure program (20-65kPa). Solvent used in this analysis were n-heptane.

Figure 12 Gas chromatography device

3.4 FAEE preparation

A mixture of oil:ethanol with the molar ratio of 1:6 was mixed in a conical flask. The calculation as below:

\[ V_{Et} + V_{oil} = 1000\text{ml} \quad \text{.......... (1)} \]

\[ R_{oil:Et} = \frac{1}{6} \quad \text{.......... (2)} \]

\[ R_{oil:Et} = \frac{V_{oil}}{V_{Et}} = \frac{n_{oil}(po)(MW_{Et})}{n_{Et}(pEt)(VEt)(MWo)} \]

\[ \frac{Vo}{VEt} = \frac{(R_{oil:Et})(pEt)(MWo)}{(po)(MW_{Et})} \]

\[ \frac{Vo}{VEt} = \frac{1}{6} \cdot \frac{(0.78g/ml)(890g/mol)}{(0.89g/ml)(46.06g/mol)} = 2.822 \]