3 MATERIALS AND METHODS

3.1 Equipment

AccuPyc II 1340 Micromeritics, Brookfield viscometer (DV-II+ Pro), Mettler Toledo pH meter, FT-IR Spectrometer Nicolet iS10 model, Grant-bio Multifunction Rotators, 360º Vertical. Centrifuge and hot plate and magnetic stirrer was used in this investigation.

3.2 Chemicals

1-methylimidazole (CH₃C₃H₅N₂), methyl diethanolamine (MDEA), diethyl sulphate, sodium acetate, 1-ethyl-3-methylimidazole, acetone, toluene and Fe₂O₃.

3.3 Methods

1. Ionic liquids screening
   i. Fedors' group contribution method
   ii. Small group contribution method

2. Ionic liquids synthesis
   i. Metathesis process
   ii. Alkylation process
   iii. Neutralization process

3. Ionic liquids characterization
   i. pH test
   ii. Density
   iii. Viscosity
   iv. Fourier Transform Infra-Red (FTIR) analysis

4. Solubility study

Figure 3.1: Flow chart for the solubility test
3.3-1 Ionic liquids screening

The solubility parameter of ILs was identified by using Fedors’ and Small group contribution method. Fedors’ and Small have compiled a table of molecular parts and their contribution to cohesive energy and molecular volume to allow the estimation of the solubility parameter as in the Appendix A. The estimation solubility parameter for Fedors group contribution method can be calculated by using Eq.7 while Small group contribution method by using Eq.8. The step of calculation as in Appendix B.

3.3-2 Ionic liquids synthesis

All the selected ILs were synthesized by using different methods. There are three methods to synthesis for different ILs:

i. Metathesis

Metathesis, for instance, involves the exchange of ions in a solution containing two ion pairs to produce the thermodynamically most stable ion pairs (Ajam, 2005). This method is to produce 1-ethyl-3-methylimidazolium acetate, [EMIM] [Ac].

Figure 3.2 (a): Metathesis steps
Equimolar amounts (0.5 M) of sodium acetate (NaOAc) and 1-ethyl-3-methylimidazolium chloride, [EMIM] Cl were mixed by using 100mL of acetone as solvent. The solution was stirred for 24 hours. The mixture will appeared into two layer where the bottom is sodium chloride while the top are [EMIM] Ac and acetone. Then, the mixture was separated by using centrifuge for 5 minutes with 5000rpm speed. The acetone was separated by using rotary evaporator to obtain the [EMIM] [Ac].

ii. Alkylation
Alkylation is the transfer of an alkyl group from one molecule to another. The alkyl group may be transferred as an alkyl carbocation, a free radical, a carbanion or a carbene (or their equivalents) (Smith & March, 2001).