CHAPTER III
EXPERIMENTAL

3.1 Chemical

Crude glycerol was supplied by Thai Oleochemicals. The composition is 2.10% of salt, 95.60% of glycerol (C3H8O3), 1.33% MONG and less than 1% of water. Demineralized water with conductivity lower than 5 μS was used to prepare the synthetic glycerol solutions simulating the different water concentration.

3.2 Apparatus

Ion-selective membranes operate based on the same principle and materials as ion-exchange resins, and they are used to specifically transport ions away from their counterions. Anion-selective membranes are permeable to anions but not to cations, while cation-selective membranes are permeable to cations but not to anions. The membranes are not water-permeable.

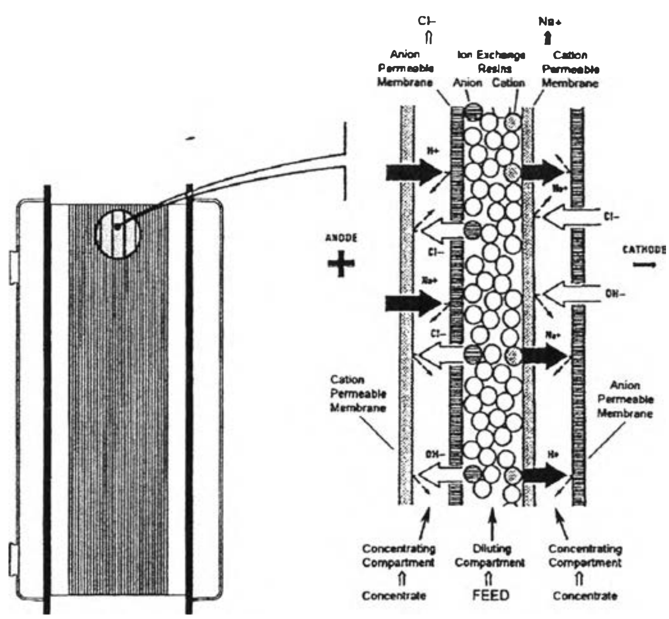

Figure 3.1 Schematic diagram of the EDI process.

The experimental set up consists of an EDI module with a 48VDC power supply (Snowpure XL-060-RL), three flow meters with adjustable valve for each stream, 2 diaphragm pumps, ultrafiltration filters, a conductivity meter and an ammeter (True RMS meter, Fluke).

All instrument were installed on an aluminium framed that was designed for this experiment as showed in figure 3.2.

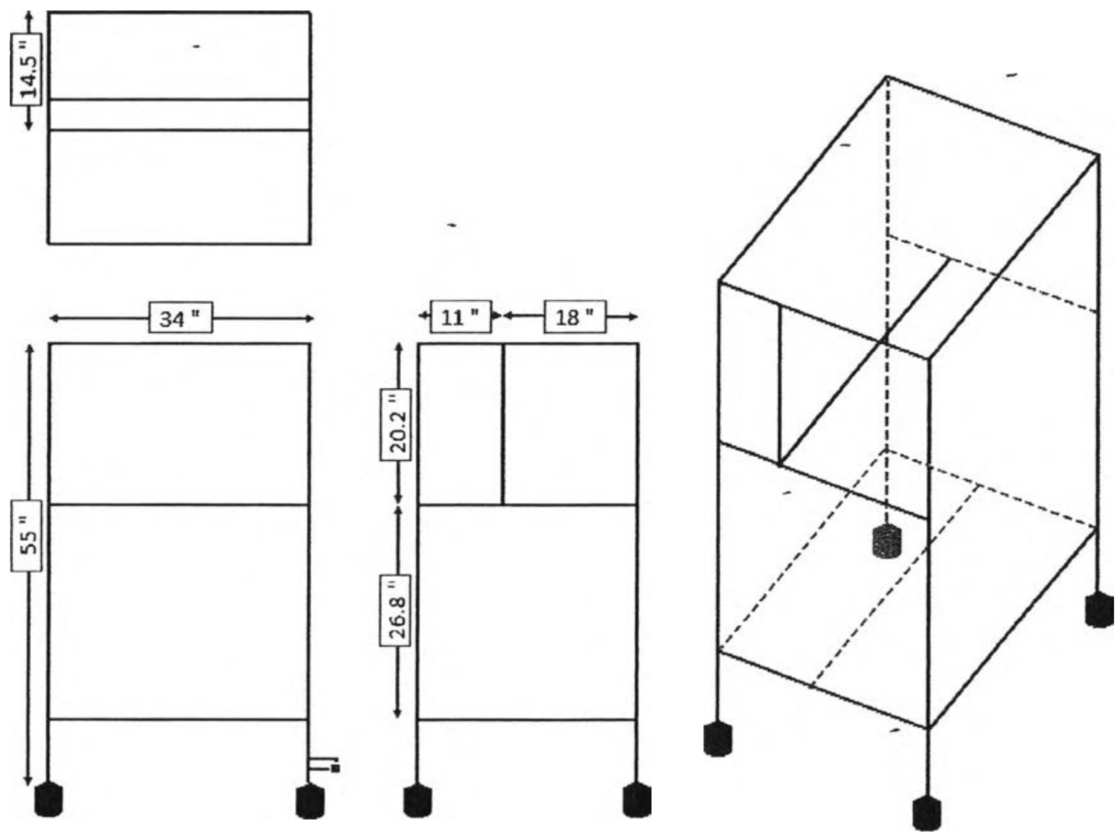

Figure 3.2 The drawing of EDI support aluminium frame.

3.3 Methodology

The EDI can be designed with repeating element, called a “cell-pair” as illustrated in Figure 3.1. The “stack” of cell-pairs is positioned between the two electrodes, supplying the DC potential to the module. Under the influence of the applied DC potential, ions are transported across the membranes from the purifying chambers into the concentrating chambers.

As showed in figure 3.3, glycerol was pumped to ultrafiltration filter and then split into 3 separated streams prior to feed to the EDI module. After the EDI module, these three streams are product, concentrated and electrode stream. The product was re-fed again to the EDI module for 5 cycles. The water concentration was varied from 30% to 70% by vol. The solution was sampled for testing periodically.
The following experimental conditions were used :

- Product stream : product from the purifying compartments, 60 l/h.
- Concentrate stream : flow in the ion-collecting concentrate compartments. Typically 10% of product stream, 6 l/h.
- Electrode stream : flow to the anode and cathode compartments. Typically constant at 10 l/h, independent of the product stream.

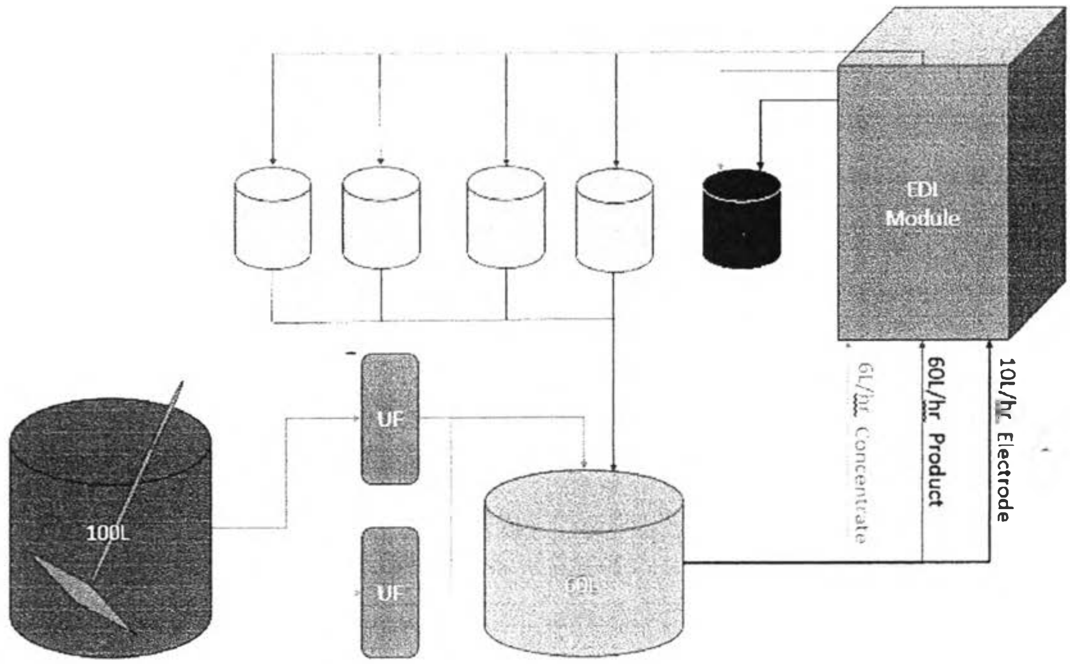

Figure 3.3 Procedure flow diagram.

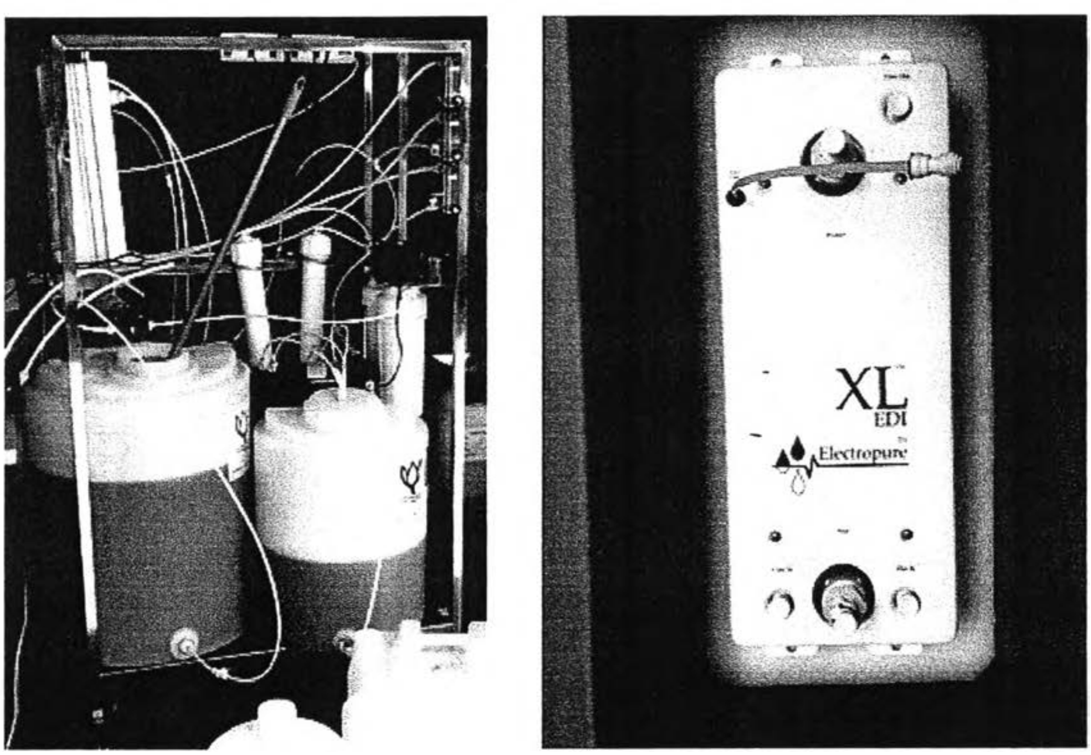

Figure 3.4 Apparatus set-up and EDI module.

3.4 Measurement

The perfomance of ultrafiltration was determined by turbidity meter. The salt concentration before and after treatment was determined by ASTM D 482-00a E1 method. The change of salt concentration in each cycle and each stream was evaluated by conductivity using a conductivity meter. Power consumption was investigate by an ammeter (True RMS meter, Fluke)

3.5 Simulation

HYSYS V7.1 was used to find the dynamic viscosity of the solution in different water concentration, the energy consumption in distillation for water removal after EDI treatment and glycerol purification. Then the energy cost were evaluated.

The basis for simulation were as follows:

- Palmitic acid were use to represented salt and MONG
- Fluid package : NRTL - Ideal
- Feed composition by volume : 66.932% Glycerol, 28.693% Water and 4.375% of Palmitic acid ($C_{16}H_{32}O_2$)
- Feed rate : 3.4 m^3/h
- Feed condition : 30 degree celcius and 101.3 kPa
- Component splitters were used to represent EDI and UF in simulation
- MONG was completely removed by UF
- 20 Stages for glycerol purification column and 10 stages for water removal column with at least 99.5% glycerol content for product
- Column pressure : 101.3 kPa for water removal unit and 1 kPa for glycerol purification unit
- The feed stage was varied to minimize energy requirement
- Reflux ratio of each distillation column : 0.25

The basis for economic evaluation were as follows:

- Fuel oil with the heating value of 9,500 kcal/L was use as fuel of reboiler, the price was 19.79 THB/L (Fuel oil type 5 from The Excise Department's announcement which affected on 24 March 2014)
- Electricity cost was 3.56 THB/kWh (From the announcement of Provincial Electricity Authority for big industrial customers which had effect in January – April, 2014)
- The EDI energy cost were calculated on the basis that the salt removal capability is the linear function to the power consumption
- The distillation calculation was made from reboiler energy flow only with 70% thermal efficiency
- The pretreatment cost by ultrafiltration membrane was neglected

3.5.1 EDI Simulation

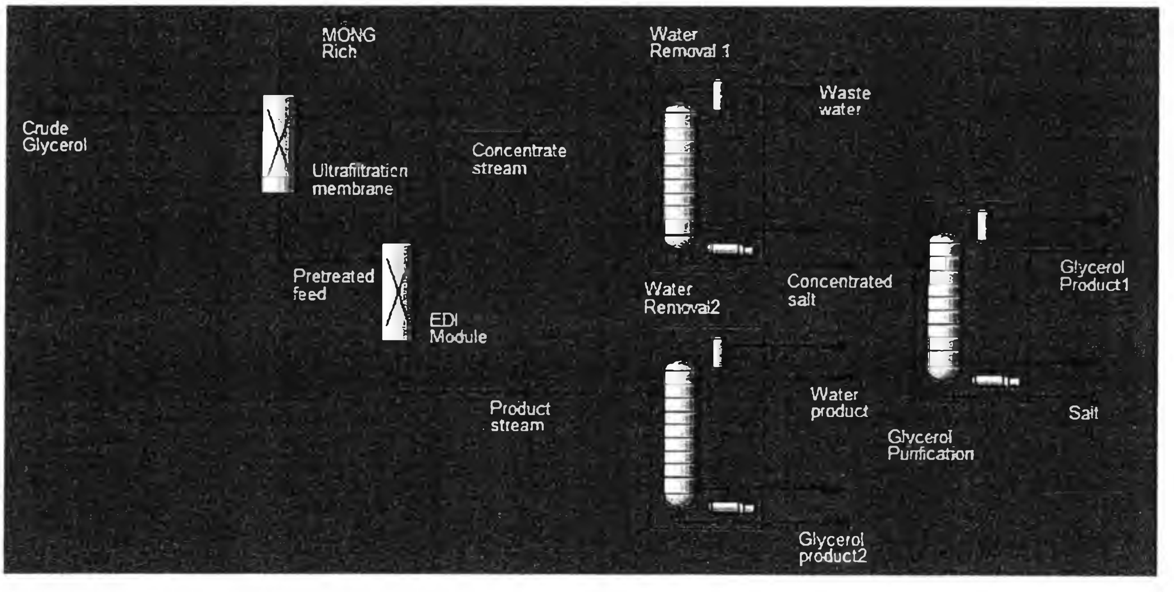

Figure 3.5 Process flow diagram of glycerol purification by EDI.

In the simulated EDI process, As showed in figure 3.5, Crude glycerol was fed in to the ultrafiltration membrane to remove MONG. Then it was fed to EDI module and splitted into 2 streams which was 9% as concentrate stream and 91% as product stream. The electrode stream was neglected due to the very low amount. Product stream was fed to a distillation column to remove the remaining water. Concentrate stream was also fed to adistillation column to remove water, then fed to an another distillation column to purifiy the glycerol. The reboiler energy consumption data was collected for economic evaluation. The energy consumption for pump and condenser was neglected.

3.5.2 Distillation Simulation

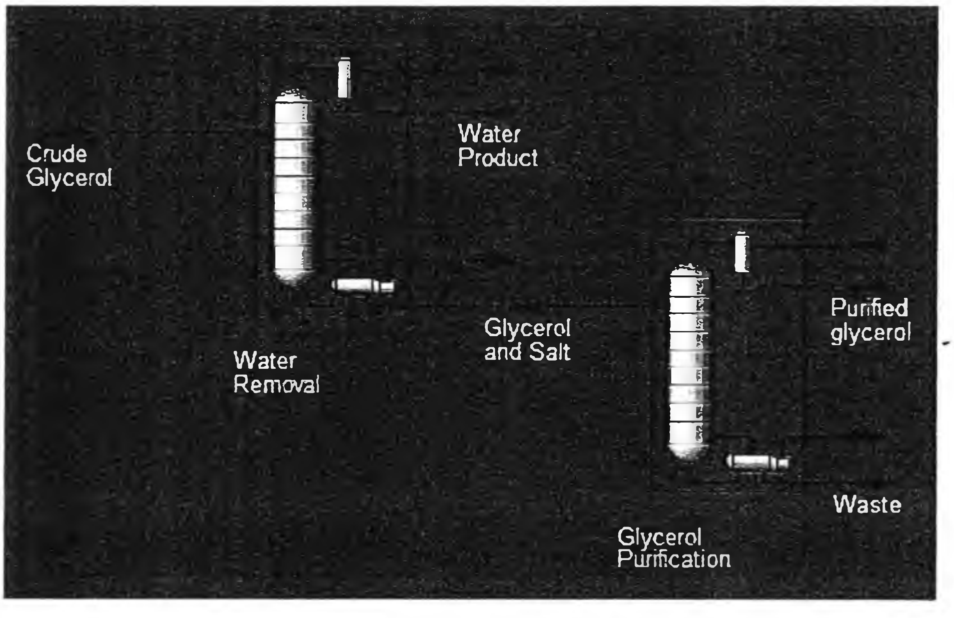

Figure 3.6 Process flow diagram of glycerol purification by conventional distillation.

In the simulated conventional distillation process, the process was easier. As showed in figure 3.6, crude glycerol was fed to first distillation column to remove the water. Then the product was fed to vacuum distillation column to separate purified glycerol from the MONG and salt.