CHAPTER 3

MATERIAL AND METHODS

3.1 Plant material

The aqueous extracts of *A. ebracteatus* Vahl. were kindly provided by Ms. Pongpun Siripong, Natural Poducts Research Section, Research Division, National Cancer Institute, Bangkok, Thailand.

3.2 Membrane

The flat sheet NF membrane used was NTR 7540, a synthetic polymer composite membrane made of sulfonated polythersulfone by Nitro Denko, which has filtration area of 0.00353 m^2 and the membrane pore radius of 0.70 nm (Wang *et al.* 1995b). The picture of NF plant is shown in Fig. 3.1.

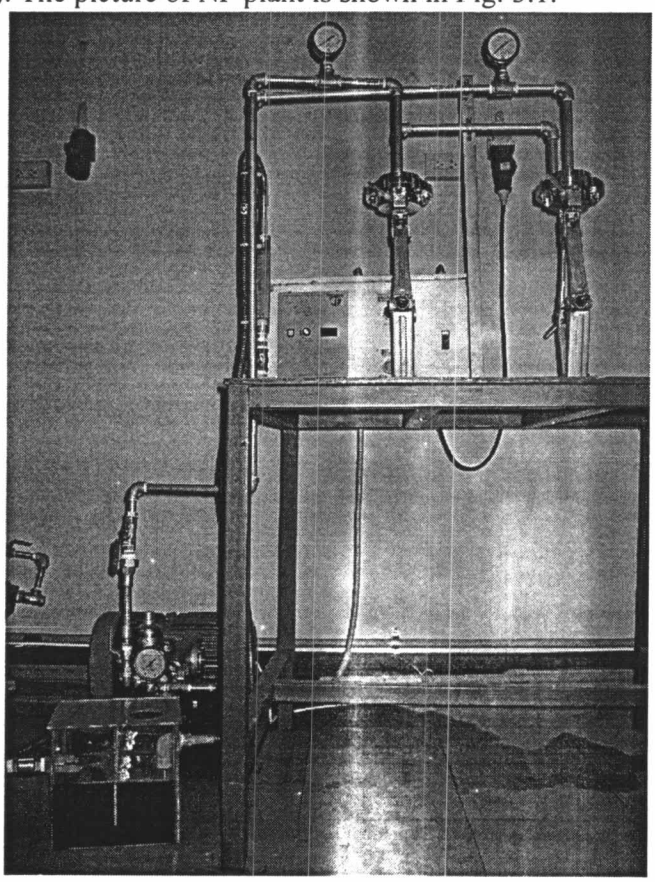

Figure 3.1: Picture of Nanofiltration unit

3.3 Experimental procedure

3.3.1 Continuous filtration for determination of optimal conditions

The experimental procedure for desalinization of aqueous extract of *A. ebracteatus* Vahl. can be divided into 2 parts. Firstly, it was necessary to determine the optimal conditions for the desalinization. In this step, both retentate and permeate were recycled to a feed tank in order to keep constant feed concentration as shown in Figure 3.2. The temperature was controlled at 30°C; this temperature was also applied for all nanofiltration experiments. The study for the effects of transmembrane pressure (TMP) and feed flow rate was performed in the range of 10kg/cm^2 to 30kg/cm^2 and 2 to 4 l/min (or equivalent to flow velocity from 0.5 to 1 m/min), respectively. Before and after each experiment, the pure water permeate flux was measured to check for the membrane performance. The permeate flux for each operating condition was measured after the system was at steady state. After getting the optimal conditions for the desalinization of aqueous extract of *A. ebracteatus* Vahl., these conditions would be used for dialysis process.

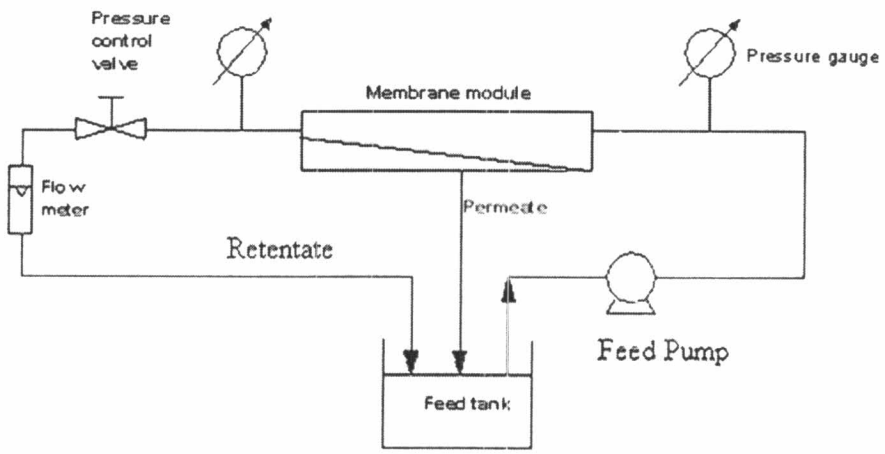

Figure 3.2 Schematic procedure for optimal condition determination.

3.3.2 Diafiltration process

The diafiltration process comprises two steps: concentration and diafiltration. The diagram of diafiltration process is described in Figure 5.3. Differently from part 1, in this part, only retentate was recycled to the feed tank but not permeate. The

permeate was collected separately. For diafiltration step, two membranes connected in series were used to get higher filtration area. The schematic and the picture of NF plant using in the diafiltration mode are shown in Figure 3.4 and 3.5, respectively. Pure water was continuously added to the feed tank controlling by a level control. For more convenient, the retentate after diafiltration process is now called concentrate.

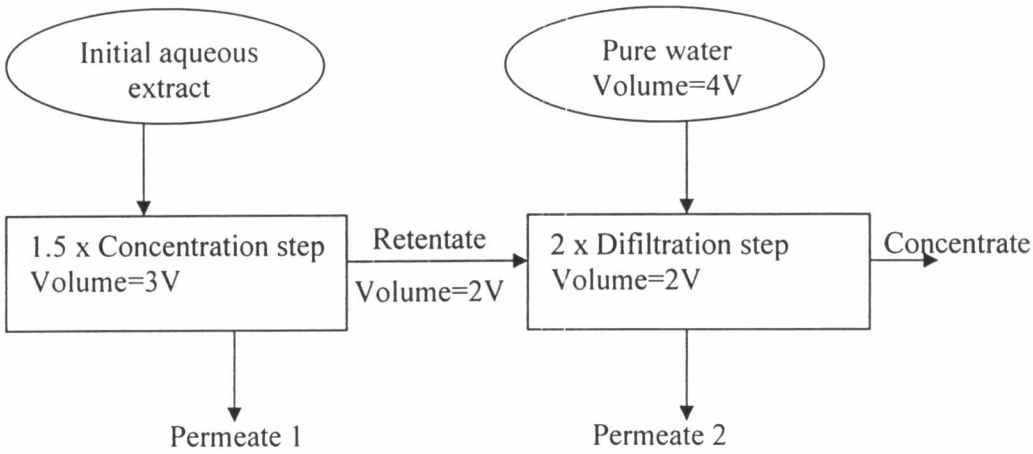

Figure 3.3 Diagram of diafiltration process. The feed of concentration step was the initial aqueous extract of *A. ebracteatus* Vahl. and the retentate after this step the was used as the initial feed of diafiltration step. During the diafiltration step, pure water was continuously added to feed tank to keep constant volume. The experiments in the diafiltration process were performed at the optimal conditions.

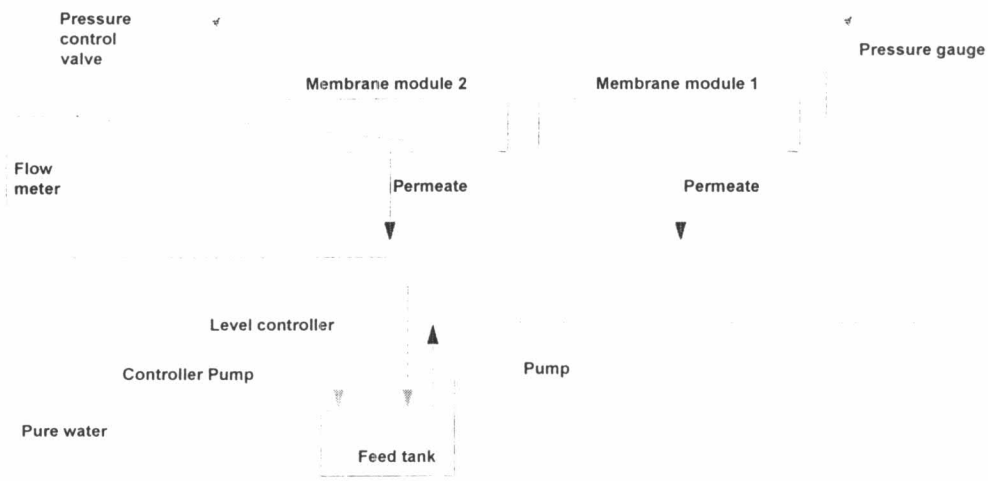

Figure 3.4 Schematic procedure for diafiltration step

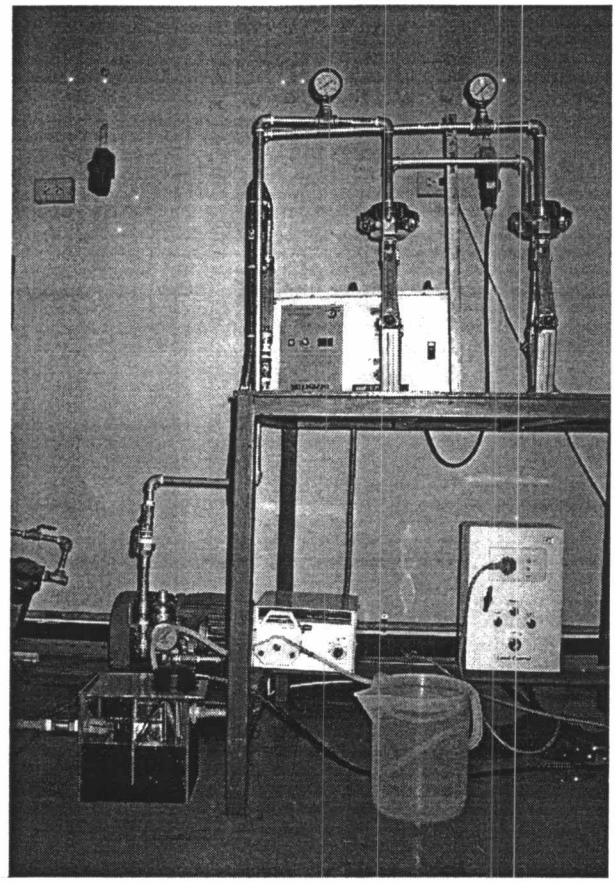

Figure 3.5 Nanofiltration plant for diafiltration step

3.4 Analytical method

The volume of permeate for each experiment was measured by 20 ml or 200 ml (for diafiltration step) cylinder. Salt concentrations, based on the concentration of sodium chloride, were rapidly measured by electrical conductivity and were confirmed by atomic absorption spectrometry (AAS) with air-acetylene flame. The concentration of other ions such as K^{+}, Ca^{2+}, Mg^{2+}, Al^{3+} were also determined by AAS method in order to gain more information about the availability of these ions to pass through the membrane.

To quantify the percentages of carbon, hydrogen and nitrogen in feed; permeate and concentrate, the CHNS/O analyzer (Perkin Elmer PE2400 Series II) was used.

The HPLC analysis was performed by using Hypersil C_{18} 250 x 4.6mm column. The compounds were separated by using the methanol gradient from 10 to 60% in water. The UV detection wave length was 223 nm.

The dry solid was determined by drying in oven at 60°C overnight until weight constantly.

3.5. Calculation

3.5.1 Volumetric permeate flux (or permeate flux)

The volumetric permeate flux (J_v) can be calculated based on volume of permeate measured (V) and time (t) required to reach it as follow

$$J_v = \frac{V}{t.A}$$

Whereas A is filtration area, A=0.00353 m^2, in case of diafiltration calculation A=0.00706 m^2.

The unit of volumetric permeate flux can be expressed in SI unit ($m^3m^{-2}s^{-1}$).

3.5.2 Observed rejection of sodium chloride

The observed rejection (R_{obs}) was calculated as follow

$$R_{obs}=1-\frac{c_p}{c_f}$$

Whereas c_p and c_f are the salt concentration in permeate and feed, respectively.

The rejection coefficient is a measure of the ability of the membrane to separate salt from the feed solution.

For a perfect separation, the permeate salt concentration is the same as the feed salt concentration $c_p=c_f$ and $R_{obs}=0$, it means that the salt can be totally separated from solution. For a completely unselective membrane, the $c_p=0$ and $R_{obs}=1$, in this case, the salt can not be separated from solution by the unselective membrane.