

Chapter 3 : Materials and Methods

3.1 Membrane Separation

3.1.1 Chemicals

Nickel (II) sulphate hexahydrate, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ (Fluka Chemika; Analar), Nickel (II) chloride hexahydrate, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (Fluka Chemika; Analar) and Chromium (III) chloride hexahydrate, $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ (Fluka Chemika; Analar) were used to study the effect of operating parameters on the separation characteristics. Sodium chloride (Fluka Chemika; Analar) was used to collect experimental data for modelling purposes.

3.1.2 Preparation of Metal Solutions

Three different feed concentrations were prepared for the experiments which involve Nickel chloride, Nickel sulphate and Chromium chloride. The feed solutions were prepared in concentrations of 50 mg/L, 30 mg/L and 10 mg/L.

The experiments for modelling purposes were carried out by using sodium chloride (NaCl) solutions. The feed solution of NaCl was prepared in

concentrations of 30 mg/L, 50 mg/L and 80 mg/L. The data from these experiments were used for comparison between experimental and calculated values. The solutions were prepared by weighing requisite amount of chemicals (χ g / 500 L), according to the formula below.

$$\chi \text{ g / 500 L} = \frac{C_r}{M_m / M_r} \times 500 \text{ L} \quad \dots\dots\dots (1)$$

where, C_r = feed concentration in mg/L (ppm)

M_m = atomic weight of the metal

M_r = molecular weight of the metal salt

500 L of feed solution was prepared for each run.

3.1.3 Membrane Unit

The membrane unit used in this experiment is B-14A-PT model from Applied Membranes Inc. The components of this unit are given in the Figure 3.1 below.

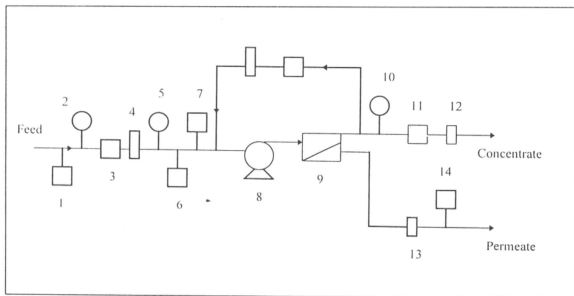


Figure 3.1 : Schematic diagram of reverse osmosis system.

Component identification:

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|--------------------------|--|
| 1. Sample Valve | -For sample of feed water. |
| 2. Temperature Indicator | - Monitors feed water temperature. |
| 3. Solenoid Valve | - Opens during operation for feed water. |
| 4. Cartridge Filter | - 5 micron sediment filter. |
| 5. Inlet Pressure Gauge | - Measures pressure of incoming water. |
| 6. pH Sensor | - Probe to monitor feed pH. |

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|-----------------------------|---|
| 7. Low Pressure Switch | - Shuts the system down if the inlet pressure is lower than 40 psi. |
| 8. High Pressure Pump | - To pressurize the incoming water to the design pressure. |
| 9. Membrane Module | - Membrane elements housed in fiberglass epoxy pressure tubes. |
| 10. System Pressure Gauge | - Measures the pressure at the exit end of the membrane module. |
| 11. Control Valve System | - To adjust concentrate flow. |
| 12. Concentrate Flowmeter | - Measures the amount of concentrate water going to drain. |
| 13. Permeate Flowmeter | - Measures the amount of permeate. |
| 14. Permeate Quality Sensor | - Measures the quality of the permeate. |
| 15. Recycle Valve | - Flow control for recycle of concentrate. |
| 16. Recycle Flowmeter | - Measures the amount of recycle concentrate. |

The capacity of this unit is 5677.5 L/day at 25 °C. System pressure of the unit is 225 psi and the pump feed pressure must be maintained above 40 psi.

3.1.4 Reverse Osmosis Membrane Module

The membrane module used in this membrane unit is spiral wound type. The construction of this module is illustrated in Figure 3.2.

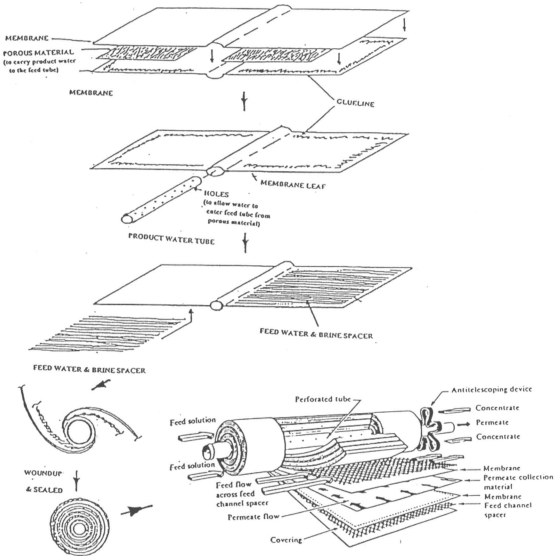
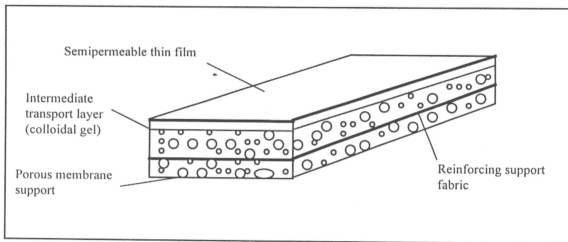


Figure 3.2: The construction of the membrane module.

The membrane used for the present work is a thin-film composite reverse osmosis module of the spiral- wound type. The schematic cross-section of thin-film composite membrane is given in Figure 3.3.



Applied Membrane Inc., 1996.

Figure 3.3 : Schematic cross-section of a thin film composite membrane.

3.2 Metal Analysis

3.2.1 Chemicals

Standard solutions were needed for the purpose of heavy metal analysis. The following standard solutions were used:

- a) Nickel standard solution ,1000 mg/L (Fluka Chemika)
- b) Chromium standard solution ,1000 mg/L (Fluka Chemika)
- c) Internal standard, 1.5 mmol/L Cesium (Instrumentation Laboratory)
- d) Nitric acid, 69.0 - 70.0 % (J. T. Baker)

3.2.2 Inductively Coupled Plasma

Nickel and chromium in the sample were analyzed by an Inductively Coupled Plasma - Atomic Emission Spectrometer model 2000 (BAIRD).

The operational procedures involved are centering of the polychromator, running of blanks, measuring of standards, performing the calibration routine and running of the samples. The instrument is equipped with a 40.68 MHz RF generator. The RF power was set at 1100 watts. The coolant gas flow rate was set at 10.0 L min⁻¹ and the auxiliary gas flow rate was 1.0 Lmin⁻¹. The carrier gas flow rate was 0.6 Lmin⁻¹. The vacuum pressure was set at 5 millitorr. The wave lengths and detection limits for Ni and Cr are summarized in Table 3.1.

Table 3.1 : Detection limits for the Baird ICP Optical Emission Spectrometer.

Element	Wavelength (nm)	Detection Limit
Cr	267.72	5
Ni	231.60	7

Ultra pure water prepared from Elgastat (UHQPS) was aspirated into the chamber to check the formation of mist in the spray chamber and to clear the plasma torch. Centering of the polychromator was performed at the initial stage of the run. This is important to optimise the optical alignment of the spectrometer so that the spectral lines are exactly centered on their exit slits.

Nitric acid (0.2 M) was used as blank. For baseline correction , the intensity of the metals in the blank was subtracted from the intensity of the metals in the standards and the samples. Integration time chosen for the measurement was 5 seconds and the number of integration was 3. In the collection of calibration data, intensity of the prepared standards was measured and the average value was calculated. Curve set files were prepared for the elements of interest, using the polynomial calculation routine. Curve coefficients were calculated automatically for each element using the standard data. Standard calibration plots were

obtained for each element.

3.2.3 Flame Photometer

The concentration of sodium in the sample was analysed by the Flame Photometer, model IL 943 (Instrumentation Laboratory). This instrument operates safely within an ambient temperature range of 10 - 40 °C and requires air supply at minimum pressure of 26 psi (1.8 atm, 170 kPa) and a volumetric flow rate of 15 L min⁻¹. Bottled air was used to ensure that the air is free from moisture, dust and oil. Propane fuel was used for this instrument and it was 99 % pure with a dew point of less than -23 °C at 1 atm and the sulphur content was less than 50 ppm. The functioning of the IL 943 is based upon automatic cycles (or routines). The most important cycles are given below:

(a) Ignition cycle : It is initiated by depressing the Flame key (with instrument power on). When the air valve is opened, the pressure switch senses the correct air pressure and the solenoid gas valve is activated. A second cycle begins with the spark electrode, which is activated for 10 seconds. During this time the flame ignites and the flame sensor detects its presence and sends the appropriate signal to the microprocessor. When the electrode stops sparking, the flame indicator becomes permanently lit. Flame ignition is immediately followed by a 3 minute

temperature stabilization period. During this time the instrument carries out three automatic priming cycles.

(b) Dilution cycle : Dilution of sample and standard is carried out automatically by the instrument. The cycle begins with activation of the dilutor which aspirates 20 μL of sample/standard into the sample probe and 2 ml of Cesium diluent into the diluent chamber. The sample and the diluent are dispensed in the spinning cup which agitates at low speed for 9 seconds, thus mixing the dilute sample.

(c) Analysis cycle : In this cycle the detection of the emission wavelength by the photodiodes and the electronic amplification of the signal take place followed by the data check and computation.

(d) Autocal cycle : Both dilution and analysis are included in this cycle. The autocal cycle checks that the Cesium internal standard level and the detection metal signals are within a preset tolerance limit. It then computes new calibration constants to recalibrate the proper channel, and centers the Cesium internal standard level if necessary.

(e) Autozero cycle : The function of this cycle is identical to the autocal cycle, except that a new zero constant for the zero calibrant is computed. Then the final result of the analysis is displayed. The concentration of sodium in the

sample collected is given directly in mmol / L. These result is then converted to mg / L using the formula below :

$$\text{mg / L} = \frac{[\text{mol} \times 10^{-3} \times \text{A g / mol}] \times 10^{-3}}{\text{L}} \dots\dots\dots (2)$$

where ,

A g / mol is the atomic weight of sodium.