MEMBRANES SEPARATION PROCESS FOR OILY WASTEWATER TREATMENT

Hasan Ferhood Makki, Ahmed Faiq Al-Alawy, Maha H. Al-Hassani and Zaid Waadulla Rashad
Chemical Engineering Department – College of Engineering – University of Baghdad – Iraq.

ABSTRACT

Pilot-scale dead end microfiltration membranes were carried out to determine the feasibility of the process for treating the oily wastewater which discharge from some Iraqi factories such as power station of south of Baghdad and the general company of petrochemical industries. Polypropylene membranes (cylindrical shape) with different pore diameters (1 and 5 micron) were used to conduct the study on micromembrane process. The variables studied are oil concentration (100 – 1000 ppm), feed flow rate (20 – 40 l/h), operating temperature (31 – 50°C) and time (0 – 3 h). It was found that the flux increases with increasing feed flow rate, temperature and pore size of membrane, and decreases with increasing oil concentration and operating time. It was found also that the effect of feed oil concentration has the greatest effect on the fouling of membrane among other variables. The percent rejection of oil improved significantly with decreasing oil concentration but decreased with increasing feed temperature, pore size of membrane and operating time. Feed flow rate has slightly effect on oil rejection. The type of oil used in this work is 20W-50 gasoline and diesel engine oil.

A general model of dead end filtration mode has been successfully evaluated to explain fundamental mechanisms involved in flux decline during dead end microfiltration of oily water emulsions. Analysis of the fall in flux with time for the polypropylene membrane (5 μm) indicates that intermediate and standard pore models give the best prediction for experimental behavior. Empirical correlations for the prediction of the flux and percent reject of oil were determined in this study. These equations have the correlation coefficient 98.87% and 91.49% respectively.

KEYWORDS: Membrane Separation; Microfiltration; Oil; Wastewater; Polypropylene Membranes.

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INTRODUCTION

Oily water emulsions are one of the main pollutants emitted into water by industry and domestic sewage (1). If oil and greases are not removed from wastewater before discharging, they can cause subsequent difficulties in surface waters. Oils can blind the pores of activated carbon, ion-exchange resins, membranes, reverse osmosis and ultrafiltration units in water and wastewater treatment plants and reduce the effectiveness and life of such plants (2). Other environmental problems caused by oil and grease are toxicity to soil and aquatic organism, damages to humans, taste and odor problems, and high chemical oxygen demand (COD). Typical oil concentrations from various industrial sources are (3): petroleum (10 – 7200 mg/l), Metals (100 – 5000 mg/l), food processing (14 – 10550 mg/l), wool (3000 – 20000 mg/l), textiles (20 – 12260 mg/l) and cooling and heating (7 – 1200 mg/l). Iraqi requirements for the oil in the discharge water are 10 mg/l (4). Various types of technologies exist for treatment of oily waters. These methods are gravity separators, dissolved air flotation, coalescers, biological treatment and activated carbon adsorption (5). Over the past several years, advances have been made in developing an industrial wastewater reclaim system for a separation process for oily industrial wastewater which is extremely effective and economical in recycling of aqueous parts washing solutions. This process is based on a membrane technology that has major technical and commercial advantages over other approaches that have been tried for this application (6).

Membrane processes are those in which a membrane is used to permeate high-quality water while rejecting the passage of dissolved and suspended solids. In the water industry, the membrane processes have been used for demineralization and for removal of both dissolved and suspended particles. Tremendous improvements have been made in recent years, and the utilization of membrane technology has dramatically increased in potable water treatment. It is expected that membrane processes will be used more and more in the future as more stringent drinking water quality standards will likely become enforced. Therefore, Membrane processes such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) are increasingly being applied for treating oily wastewater (7). Membranes have several advantages such as more widely applicable across a wide range of industries, the quality of the treated water (the permeate) is more uniform regardless of influent variations, no extraneous chemicals are needed, making subsequent oil recovery easier, membranes can be used in-process to allow recycling of selected waste streams within a plant, concentrates up to 40±70% oil and solids can be obtained by UF or MF, membrane equipment has a smaller foot print, energy costs are lower compared to thermal treatments and the plant can be highly automated and does not require highly skilled operators (8, 9).

Microfiltration (MF) is the oldest membrane technology but it seems to be remaining in its childhood much longer than its relative's ultrafiltration and reverse osmosis (10). Microfiltration refers to filtration processes that use porous membranes to separate suspended particles with diameters between 0.1 and 10 μm. Thus, microfiltration membranes fall between ultrafiltration membranes and conventional filters. Currently there are three basic types of membranes being offered for microfiltration systems (11, 12): (a) Polymer types such as polysulfones and polypropylene are popular due to their low cost, (b) Ceramic membranes typically have a sintered metal membrane such as zirconium or titanium oxide over the support structure of an aluminum oxide tube and (c) Carbon/graphite fiber tubes use a sintered carbon membrane surface for microfiltration.

Although MF membranes can successfully treat produced waters, they experience a decline in permeate throughput or flux as a result of fouling. This flux decline is due to the adsorption and accumulation of rejected oil, suspended solids, and other components of

produced water on the membrane surface (external fouling) or in the membrane pores (internal fouling). This fouling can be irreversible or resistant to cleaning, making the original flux unrecoverable. Fouling can be reduced through the use of different or surface modified membrane materials, various operating strategies and pretreatments, and hydrodynamic techniques (13). However, specific fouling mechanisms and reduction strategies during microfiltration of produced water are not well understood (14). In the present study, to achieve low content of oil in permeate and high permeate flux, effects of operating parameters (oil concentration, temperature, feed flow rate, pore diameters and time) in a microfiltration unit were studied. Simple models have also been employed to help analyze the polypropylene membrane-fouling process. The experimental results of the oily wastewater treatment in the membrane process can be formulated in a form of empirical correlations.

MEMBRANE FOULING MODELS

Fouling is widely perceived to be the most significant issue affecting the design and operation of membrane filtration facilities (15). The permeation flux of particle-free water across a clean membrane can be described by Darcy's law as:

$$J = \frac{\Delta P}{\mu R_m} \qquad (1)$$

Where J is the permeation flux, ΔP the transmembrane pressure, μ the absolute viscosity of the water and R_m the membrane resistance (16). For any constant pressure dead-end filtration, fouling of the membrane can be analyzed using different pore blocking models namely complete pore blocking model, standard pore blocking model, intermediate pore blocking model and cake filtration model (17).

The complete blocking filtration assumed that each particle reaching the membrane participates in the blocking phenomenon by pore sealing, which leads to the assumption that particles are not superimposed one upon the other (18). In standard blocking model, the particle diameter is much less than the pore diameter, thus, the particles can enter most pores, deposit on the pore walls, and thus reduce the pore volume. The decrease of pore volume is also proportional to the permeate volume (1). Intermediate blocking occurs when the size of the solute particles are

similar to the membrane pore size. In this model, it is assumed that amembrane pore is not necessarily blocked by the solute particles and some particles may settle over others. Therefore, the non-blocked membrane surface area diminishes with time and some particles are expected to obstruct the membrane pore entrance without blocking the pore completely (19). In the cake filtration model, it is assumed that the flux decline is due to the accumulation of a cake at the surface of the membrane and that cake thickness increases proportionally with the volume of permeate produced.

For microfiltration at a constant transmembrane pressure, the permeation fluxes under each of theses case may be given as:

a. Complete pore blocking model

$$J = J_0 \exp(-k_b t) \qquad (2)$$

b. Standard pore blocking model

$$J = J_0 (1 + \frac{1}{2} K_s (A J_0)^{0.5} t)^{-2} \qquad (3)$$

c. Intermediate pore blocking model

$$J = J_0 (1 + K_i A J_0 t)^{-1} \qquad (4)$$

d. Cake filtration model

$$J = J_0 (1 + 2K_c (A J_0)^2 t)^{-0.5} \qquad (5)$$

Where J_0 depends on the transmembrane pressure, membrane resistance and viscosity of the filtrate and is expressed as $J_0 = \Delta P / \mu R_m$. The various K terms represent mass transfer coefficients for the associated filtration laws. In the case of constant pressure filtration, the term $(A J_0)$ is constant and the filtration laws can be simplified to:

a. Complete pore blocking model

$$\ln(J) = \ln(J_0) - k_b t \qquad (6)$$

b. Standard pore blocking model

$$(1/J^{0.5}) = (1/J_0^{0.5}) + k_s t \qquad (7)$$

c. Intermediate pore blocking model

$$(1/J) = (1/J_0) + k_i t \qquad (8)$$

d. Cake filtration model

$$(1/J^2) = (1/J_0^2) + k_c t \qquad (9)$$

Where $k_s = (1/2) K_s A^{0.5}$, $k_i = K_i A$, $k_c = 2K_c A^2$.

Consequently plotting the left-hand side flux functions for each model against time are the tests to determine the more appropriate model and the means to obtain the mass transport parameters from the slope. Therefore, a plot of $\ln(J)$ vs. t, $(1/J^{0.5})$ vs. t, $(1/J)$ vs. t and $(1/J^2)$ vs. t shall be a straight line with slope of k_b, k_s, k_i and k_c, with y-intercept of $\ln(J_0)$, $(1/J_0^{0.5})$, $(1/J_0)$ and $(1/J_0^2)$ for complete pore blocking, standard pore blocking, intermediate pore blocking and cake filtration model, respectively. The appropriate fitness and competence of various fouling models can be confirmed by comparing the values of coefficient of correlation (R^2) obtained from the linear regression analysis (20).

MEMBRANE OIL REJECTION

The rejection percentage (R %) is a combination factor between the oil concentration in feed (C_F) and oil concentration in the product (C_P). It is calculated according to the following formula.

$$R \% = \left(1 - \frac{C_P}{C_F}\right) * 100 \qquad (10)$$

The decrease of oil concentration in permeate will increase the rejection percentage and vice versa (21).

EXPERIMENTAL

Materials

The Pentek PS5-10C Smooth Core Filter Cartridge is a spun polypropylene filter cartridge used to reduce dirt, sandy, rust and sediment from water. Also known as part number 255694-43. Ideal applications include water, organic acids, oils, concentrated alkalis, organic solvents and electroplating solutions. The Ametek, Pentek, USA Filter PS510C Specifications:

- Manufactured from pure 100% polypropylene.

- Designed for purity and chemical compatibility.

- Spun fibers from a true gradient density from outer to inner surfaces.

- Micron Rating: 5 and 1.

- Temperature Range: 4.4 ^{o}C to 62.8 ^{o}C

- Dimensions: L = 25 cm & d = 6.5 cm

- Effective area: 0.051 m^2

This filter should be changed every 3 – 6 months based on water quality and usage.

Equipments

Oil – water emulsions were prepared by vigorous mixing of oil and water in the QVF glass vessel (30 l), using a stirrer (JANKE & KUNKEL GmbHu. CokG, England, 1 KA – WERK, RW 14 H, Staufen) at an agitation speed of 0 - 2000 rpm. Classic oil 20W-50 (gasoline and diesel engine oil), was used for the preparation of the oil-water emulsions. Demineralized water, of 15 – 25 µS/cm conductivity, was used for preparing emulsion with oil concentration of 100, 500 and 1000 ppm. Pressure gauge is used in the feed line to indicate the feed pressure (range of 0 – 6 bar). The physical and chemical properties of the oil are given in Table 1.

Experimental Procedure

The experimental setup is shown schematically in Figure 1. The feed was pumped by means of a centrifugal pump (11.4 – 54.6 l/min, 3 – 13.7 m. H, 210 Watt, STUART TURNER LTD. HENLEY ON THAMES ENG, England) to pass through membranes (5 µm and 1 µm) to remove oil from oil – water emulsions. The feed temperature was varied between 31 and 50 °C by means of a submersible electrical coil (220 Volt, 1000 Watt) and thermostat of range from 0 to 80 °C. The water flow rate was regulated by means of globe valve connected at the discharge of the pump (the main feed line), and measure with a calibrated rotameter with range flow (10 – 100 l/hr). Furthermore, a by-pass line with valve was located at the outlet of the pump to direct any excess flow of water back to the feed vessel (Q.V.F. cylindrical vessel of 30 liter capacity).

Permeate (filtered water) was collected every 15 minutes and volume of the permeate during the interval was measured and recorded. Concentration of oil in permeate was measured by UV-ultraviolet/visible spectrophotometer (Shimadzu model UV-160 A). Figure 2 show the calibration curve for the oil in water. The filtration flux was calculated by dividing the permeate volume by the product of effective membrane area and time. After recording the results, the solution

(remaining in feed vessel), was drained by means of a drain valve. The whole system was washed by warm water for 20 min, and then drained away. Also, detergent solution was allowed to circulate through the equipment for further 20 min, and then drained. Finally the equipment was flushed away with distilled water.

RESULTS AND DISCUSSION

Effect of Oil Concentration

Figures 3 to 4 show the effect of oil concentration on membrane flux and oil rejection for polypropylene membrane (5 μm). All experiments have been carried out at a temperature of 31°C and transmembrane pressure of 0.8 bar. It can be seen that the permeation fluxes is a decreasing function of filtration times. The flux and oil rejection decreases with increasing concentration of oil. The results show that permeation fluxes of oil feed concentrations 100, 500 and 1000 ppm decrease steeper at early filtration times (0.25 – 2 h) and by increasing the time from 2 to 3 hour, the flux slowly change and remained approximately constant.

However, when the concentration increases to 1000 ppm, the flux decreases because a layer of oil forms on the membrane surface. At lower concentrations, an oil layer formed on the membrane surface can be removed by hydrodynamic action of flow. But at higher concentrations, the hydrodynamic action cannot remove the oil layer. By increasing the operation time, this layer becomes thicker and the flux decreases as well as increasing of oil concentration in permeate i.e. decreasing the rejection of oil.

Effect of Feed Flow Rate

The effect of flow rate on flux through membrane and oil rejection is shown in Figures 5 and 6 for polypropylene membrane (5 μm). The flux increased (363.3 to 726.6 l/m^2.h) with an increase in feed flow rate (20 to 40 l/h) at time equal to 0.25 hour due to a concomitant increase in the Reynolds number and the transition to turbulent flow (i.e. increasing the feed flow rate prevents the concentration buildup in the solution at the vicinity of the membrane surface, and resulting in decreasing the concentration of

oil in product and increasing flux). Increasing of flow rate will slightly decrease the concentration of oil in permeate (50 t0 34 mg/l) at t = 0.25 h, and cause increase in oil rejection (90 to 93.2%) at t = 0.25 h.

EFFECT OF TEMPERATURE

As the operating temperature of feed increase, the flux will increase. This is shown in Figure 7. The viscosity of feed dramatically decreases with increasing temperature from 31°C to 50°C, and as a result, its permeation through the membrane becomes easier. This enhances permeate flux. Increasing temperature also increases oil content in permeate. Therefore, the rejection percentage of oil decreased with increase in operating temperature. This is shown in Figure 8. It must be mentioned that by increasing temperature, the flux increased, but higher temperatures increase the operational cost of the unit. Thus, temperatures higher than 40°C are not recommended.

EFFECT OF PORE SIZE OF MEMBRANE

Figure 9 presents the variation of permeate flux with time for polypropylene membranes with pore sizes of 5 μm and 1 μm. For two membranes, the order of water flux and ejection of oil are:

Flux (for 5 μm) > Flux (for1 μm) and Rejection Oil (for 5 μm) < Rejection Oil (for 1 μm)

Membrane pore size of 5 μm has a high water flux because it has high pore size which easier the flow through membrane. Figure 10 illustrates the effect of pore size on rejection percentage for oil. The fact that the permeate concentration does not generally increase with membrane pore size provides further support for the filtration being controlled by the fouling layer rather than the membrane. Generally, both membranes showed a similar trend in the relationship of permeate flux and rejection of oil.

THE FILTRATION MODELS

Figures 11 – 15 show model prediction and experimental data for different cases of Hermia's model. In most cases the models exhibit

a reasonable agreement with experimental data giving linear correlations. The model correlations for each case are given in figures. The estimation of the flux at t = 0 (J_0), from the intercept, gives the following values, 375.7, 381.05, 387.9 and 409.57 $l/m^2.h$ for the complete pore blocking, standard pore blocking, intermediate pore blocking and cake filtration models, respectively. These values are different from the initial experimental flux, measure at 391 $l/m^2.h$. The best agreement with experimental data is given by the intermediate pore blocking model and came in second level, the standard model for polypropylene membrane (5 μm).

CORRELATION ANALYSIS

The experimental data obtained from the membrane separation process experiments were correlated as a relationship between the flux (J) with different operating conditions namely; the feed oil concentration (C_F), feed flow rate (Q_F), operating temperature (T) and time (t), for polypropylene membrane (5 μm). The resulting correlation is:

$$J = 21.229 C_F^{-0.0519} Q_F^{1.018} T^{0.195} t^{-0.188} \qquad (10)$$

The correlation coefficient for the above equation is 98.87%.

Also the empirical correlation of the rejection percentage of oil R% as function of C_F, Q_F, T and t lead to the following equations:

$$R\% = 213.323 C_F^{-0.094} Q_F^{0.0819} T^{-0.0164} t^{-0.155} \qquad (11)$$

The correlation coefficient for the R% equation is 91.49%. The curve fitting procedure was done by STATISTICA software to find the constants and powers in Equations 10 and 11. Figures 16 and 17 show the observed values versus predicted values of flux and oil rejection obtained from Equations 10 and 11 respectively.

CONCLUSION

- The polypropylene membrane is inexpensive and available commercially. Through three hours from operating it showed efficiencies higher than 50%; therefore, this membrane can be recommended for oily wastewater treatment.

- The time of the experiment was run for three hours in order to keep the membrane and the possibility of cleaning. Then used again in other experiments.

- The flux of the membrane increases with increasing operating temperature, feed flow rate and pore size of membrane. While, the flux decreases with increasing feed oil concentration and operating time.

- The rejection of oil increases with increasing flow rate and pore size of membrane. While, the rejection decreases with increasing feed oil concentration and feed temperature.

- The flux and oil rejection decreases with increasing operating time because the fouling deposited on the surface of the membrane.

- High flow rates are suitable in order to remove the oil layer from the membrane surface. Also, temperatures higher than 40°C are not recommended because higher temperatures increased operational costs.

- The 5 μm polypropylene membrane is more permeable and exhibits a higher flux than does the 1 μm polypropylene membrane. The polypropylene membrane (1 μm) has high oil rejection.

- Experimental results in this work were in excellent agreement with intermediate pore blockage and standard models.

- The flux (J) and rejection percentage of oil R% from membrane unit are correlated to include the effect of different variables. These equations have the correlation coefficient 98.87% and 91.49% respectively.

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NOMENCLATURE

Symbol	Definition	Units
ΔP	Transmembrane Pressure	bar
A	surface area of the membrane	m^2
C_f	Feed Concentration	ppm
C_P	Product Concentration	ppm
J	Permeation Flux	$l/m^2.h$
J_0	Initial filtrate Flux through the Membrane	$l/m^2.h$
k_b	constant in complete blocking model	h^{-1}
k_c	constant in cake filtration model	h^{-1}
k_i	constant in intermediate pore blocking model	h^{-1}
k_s	constant in standard pore blocking model	h^{-1}
Q_F	Feed Flow Rate	l/h
R	rejection	
R_m	Membrane Resistance	m^{-1}
t	Time	h
T	Temperature	°C

Greek Symbols	**Definition**	**Units**
Symbol		
μ	Absolute Viscosity of the Water	Pa s

Table 1 The Physical and Chemical Properties of Oil

Viscosity grade	20W-50
Colour	Amber
Physical State	Liquid at ambient temperature
Odour	Characteristic mineral oil
Vapour Pressure	Expected to be less than 0.5 Pa at 20 °C
Initial Boiling Point	Expected to be above 280 °C
Solubility in Water	Negligible
Density	888 kg/m^3 at 15 ºC.
Flash Point	215 ºC
Flammable Limits - Upper	1% (V/V)
Flammable Limits - Lower	10% (V/V)
Auto-Ignition Temperature	Expected to be above 320ºC
Kinematic Viscosity	157 mm^2/s at 40 ºC
Pour Point	-27 ºC

Hasan Ferhood Makki
Ahmed Faiq Al-Alawy
Maha H. Al-Hassani
Zaid Waadulla Rasha*d*

Membranes Separation Process For
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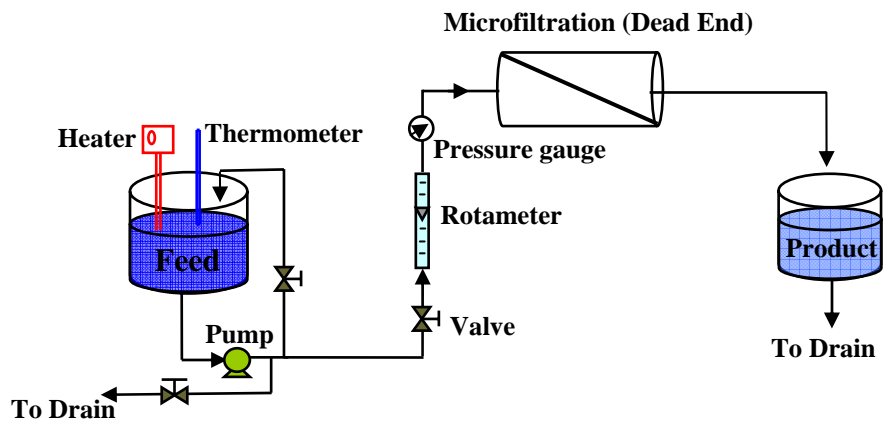
Fig. 1 Schematic Diagram of Microfiltration Process
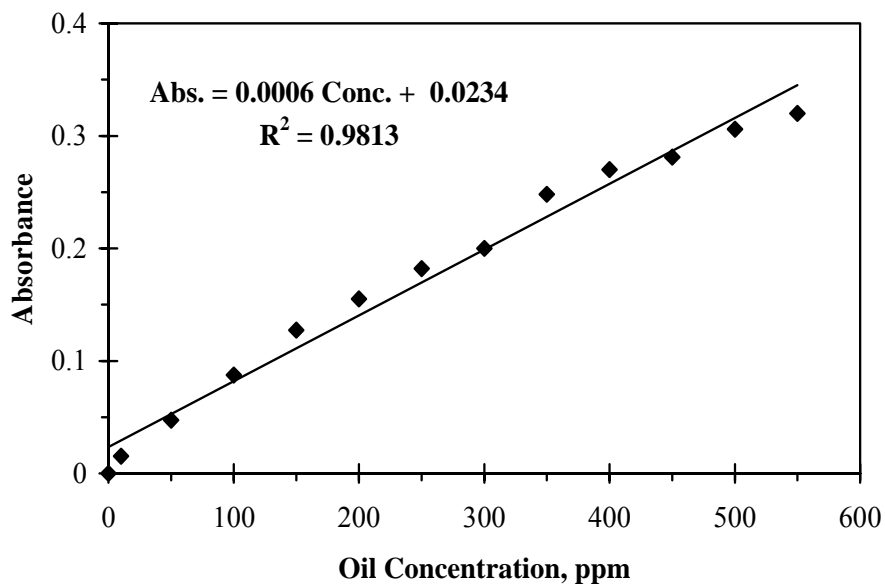
Fig. 2 Oil Concentrations vs. Absorbance (Calibration Curve)
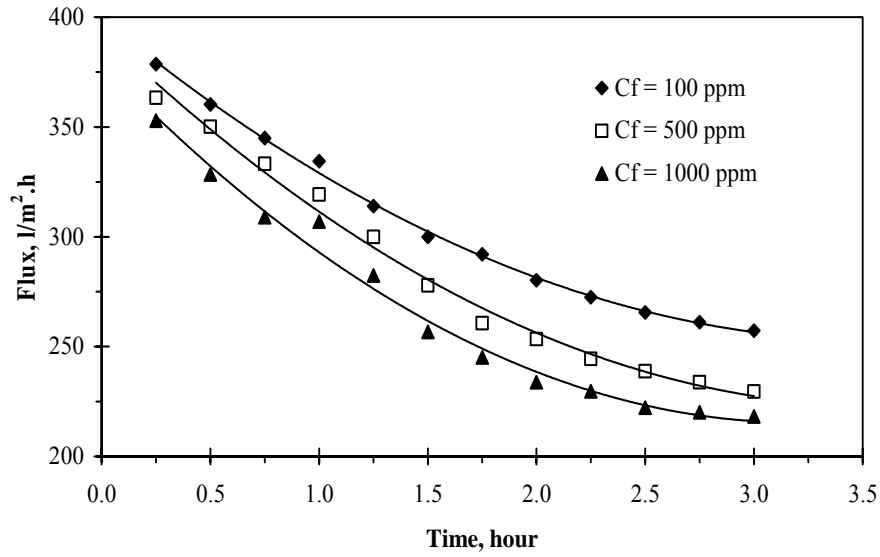
Fig. 3 Flux vs. Time at Different Oil Concentrations (Q$_F$ = 20 l/h and T = 31°C)
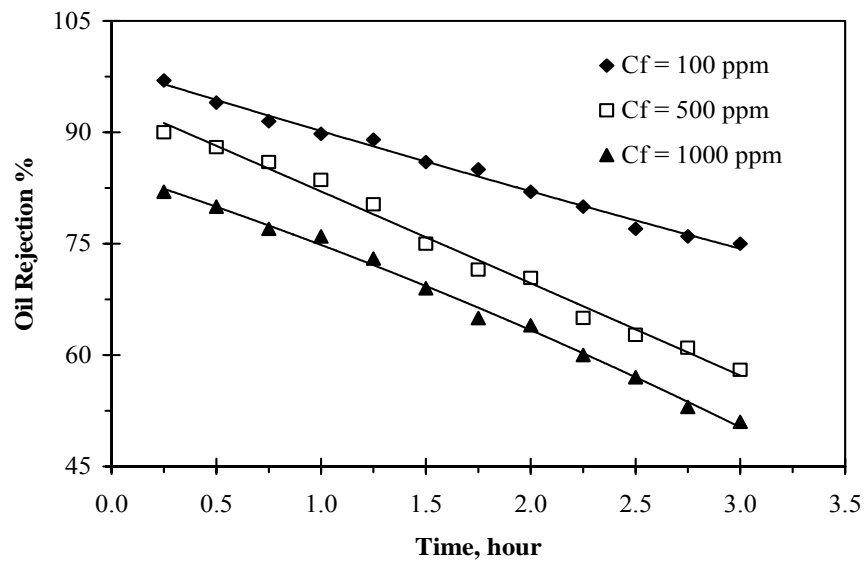
Fig. 4 Oil Rejection vs. Time at Different Oil Concentrations (Q$_F$ = 20 l/h and T = 31°C)

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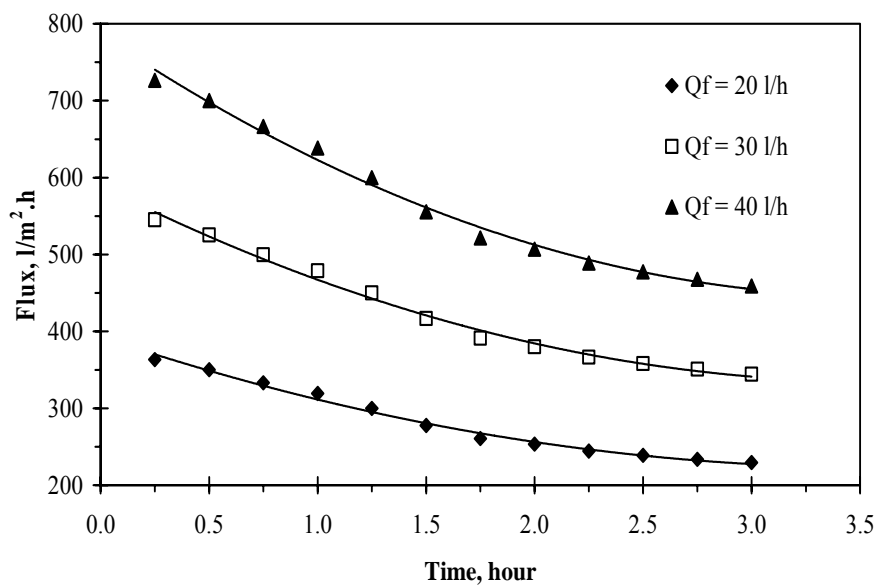
Fig. 5 Flux vs. Time at Different Feed Flow Rate (C_F = 500 ppm and T = 31°C)
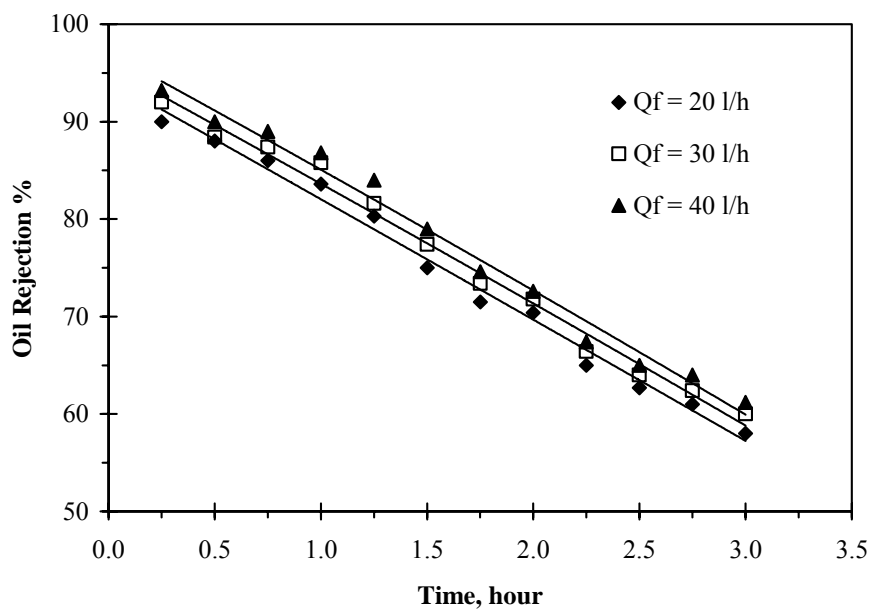
Fig. 6 Oil Rejection vs. Time at Different Feed Flow Rate (C_F = 500 ppm and T = 31°C)
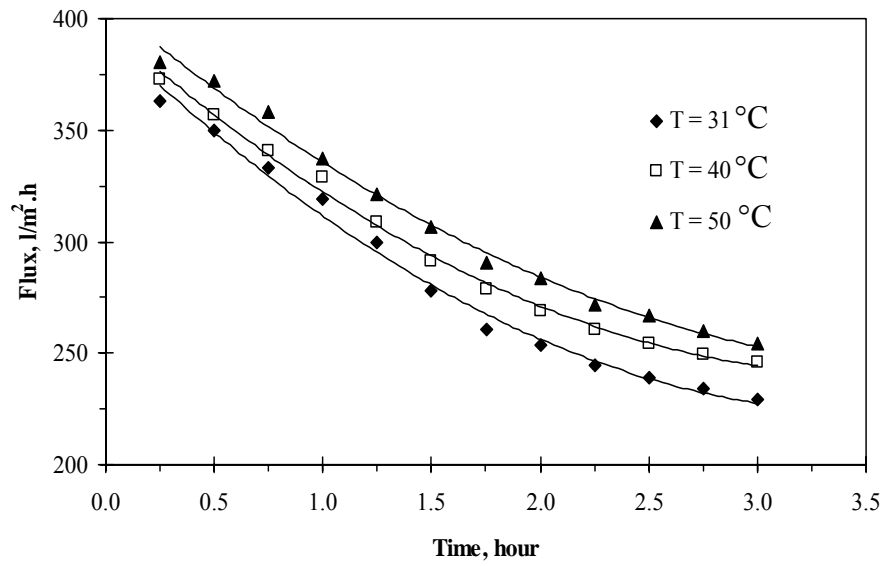
Fig. 7 Flux vs. Time at Different Feed Temperature (C_F = 500 ppm and Q_F = 20 l/h)
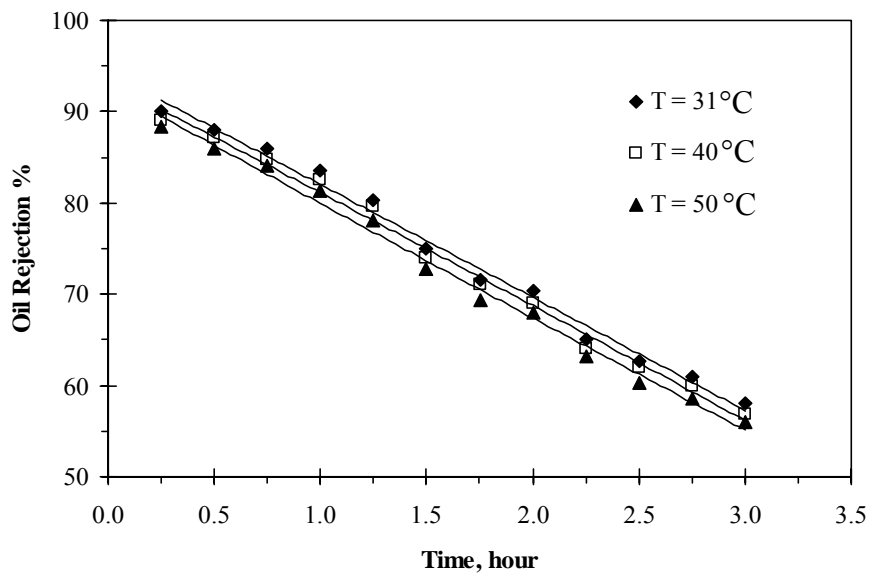
Fig. 8 Oil Rejection vs. Time at Different Feed Temperature (C_F = 500 ppm and Q_F = 20 l/h)
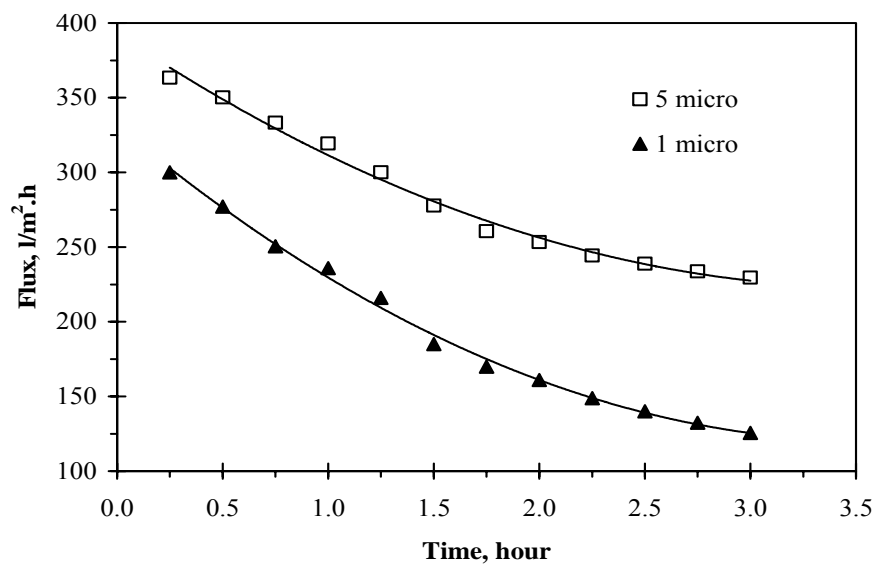
Fig. 9 Flux vs. Time at Different Pore Size (C_F = 500 ppm, T = 31°C and Q_F = 20 l/h)
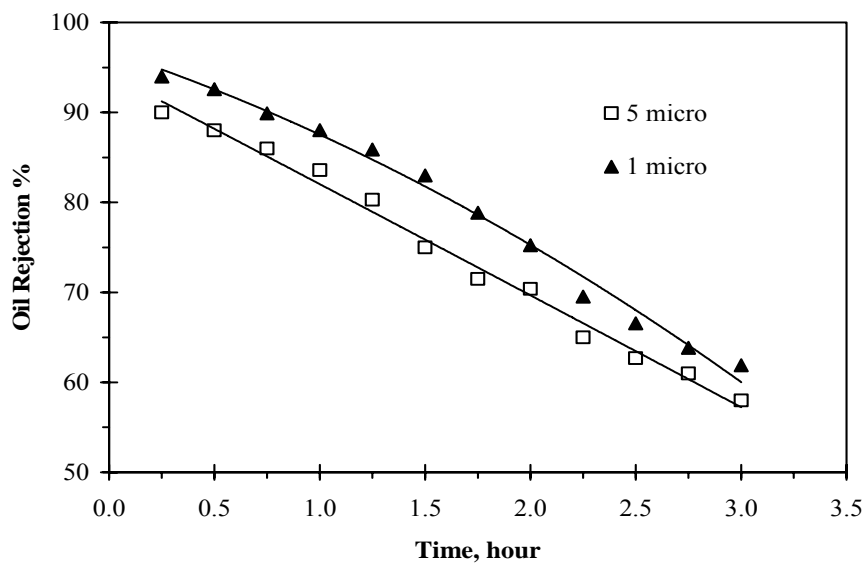
Fig. 10 Oil Rejection vs. Time at Different Pore Size (C_F = 500 ppm, T = 31°C and Q_F = 20 l/h)
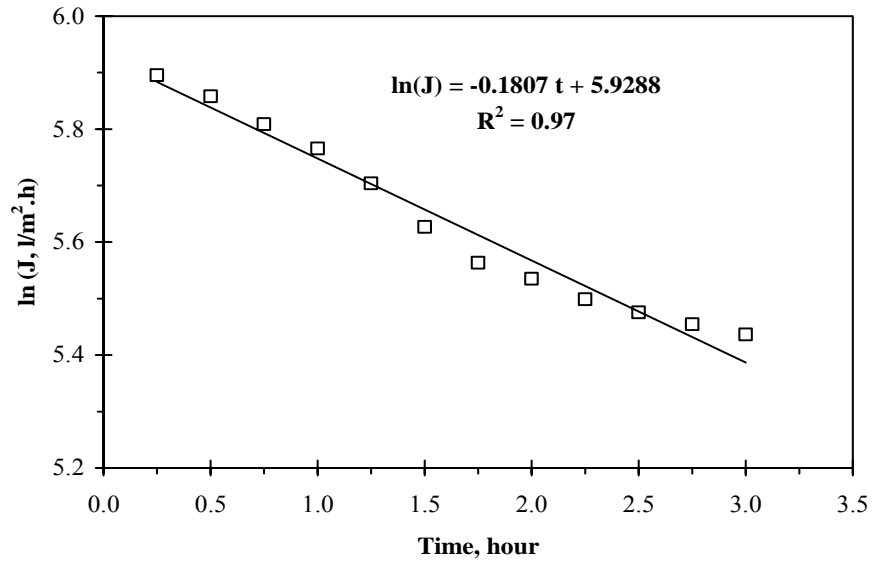
ln(J) = -0.1807 t + 5.9288
R² = 0.97

Fig. 11 Complete Pore Blocking Model (C$_F$ = 500 ppm, T = 31°C and Q$_F$ = 20 l/h)

(1/J$^{0.5}$) = 0.5358 t + 5.1228
R² = 0.9756

Fig. 12 Standard Pore Blocking Model (C$_F$ = 500 ppm, T = 31°C and Q$_F$ = 20 l/h)

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Membranes Separation Process For
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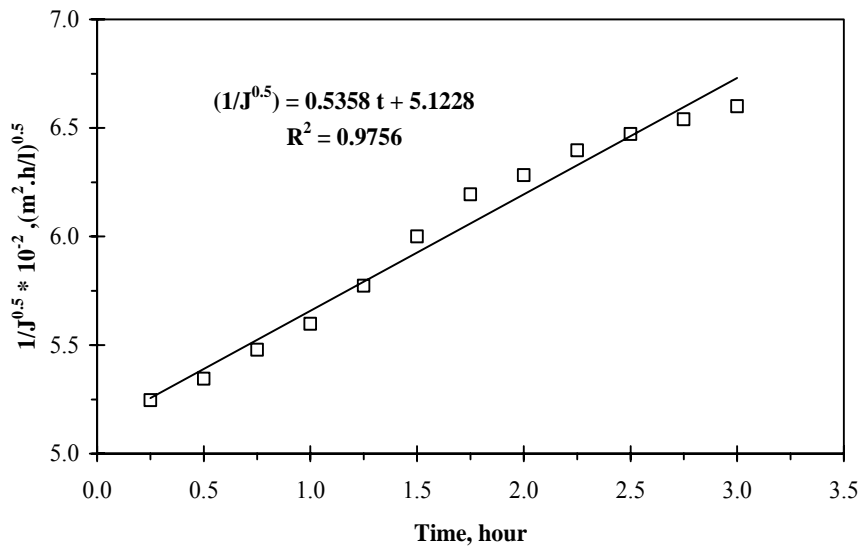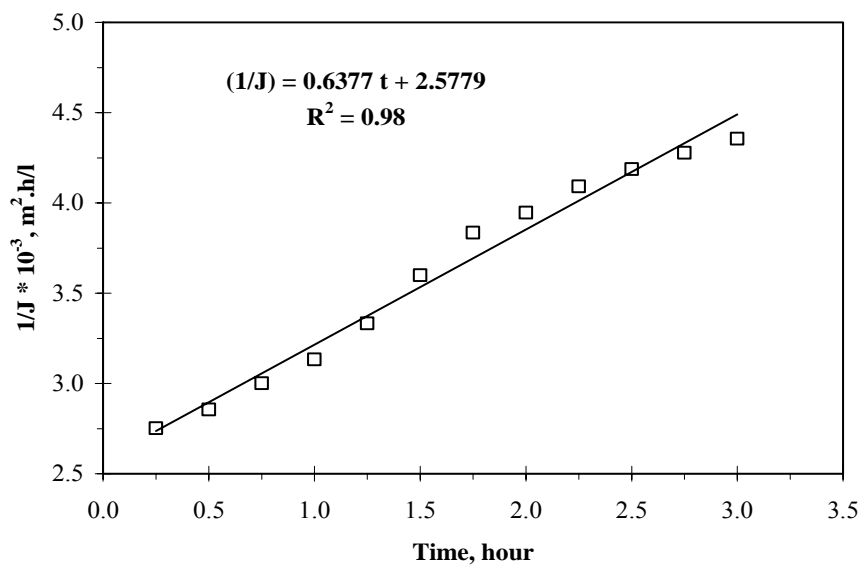
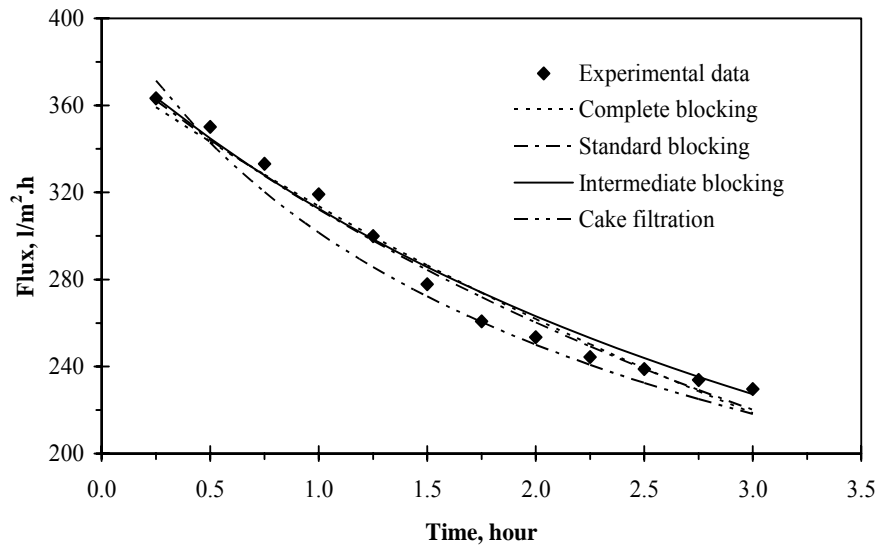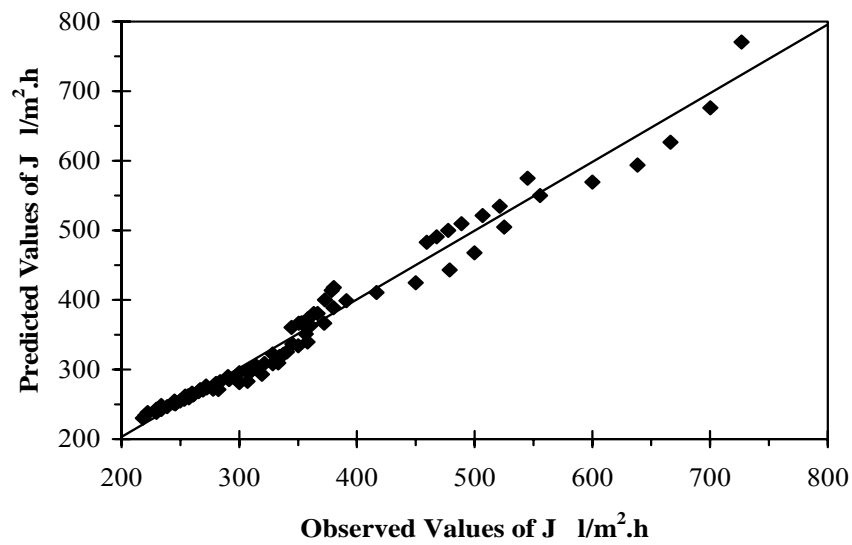**Fig. 13 Intermediate Pore Blocking Model (C_F = 500 ppm, T = 31°C and Q_F = 20 l/h)**
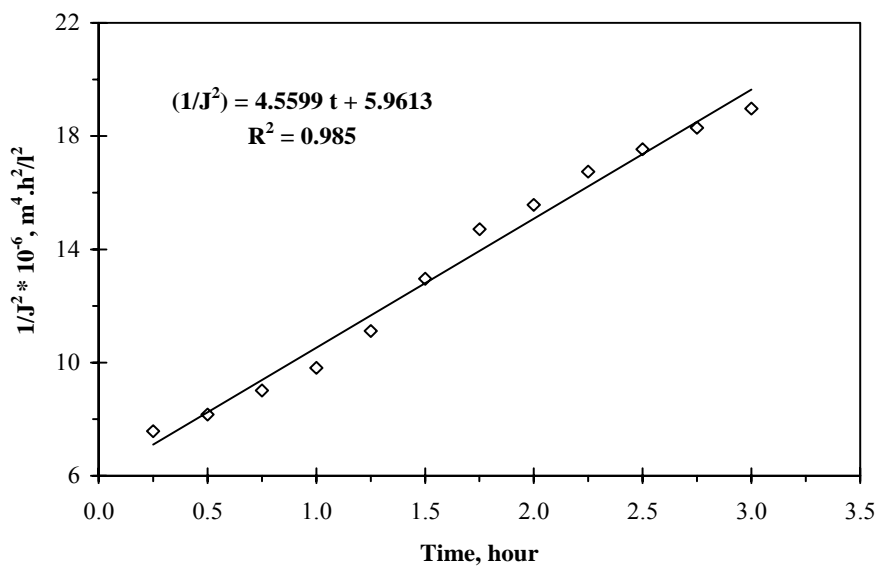
Fig. 14 Cake Filtration Model (C_F = 500 ppm, T = 31°C and Q_F = 20 l/h)

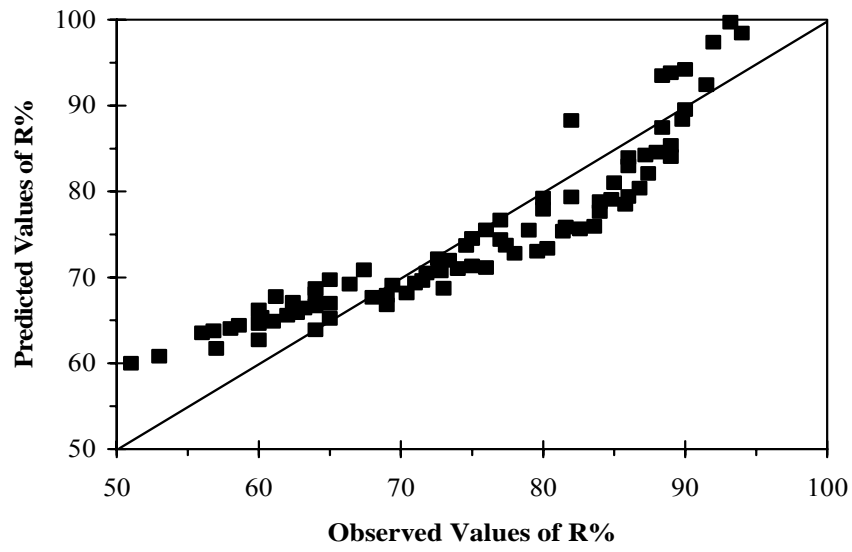**Fig. 15 Comparison of Filtration Model Prediction with Experimental Data for Polypropylene Membrane (5 μm)**

Fig. 16 Observed Versus Predicted Values of Flux

Fig. 17 Observed Versus Predicted Values of Oil Rejection