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Rotating Cylinder Microfiltration of Oil–in–Water Emulsion Using Novel Slotted Pore Filter

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ABSTRACT

Nowadays, oil-in-water emulsion has become an important topic in petroleum industry, which produces oil-in-water emulsion in the recovery of crude oil. Oil-in-water emulsion produced in crude oil recovery causes problems at different stages of the production in the petroleum industry. Recently, microfiltration has been applied in the separation of oil from water. In filtration of oil-in-water emulsion, there is the possibility of oil drops deforming and squeezing through the slot of membrane so the separation efficiency will decrease. This research has studied cross flow filtration of oil-in-water emulsion in a rotating system and also visualized the interaction of oil drops and slot shaped membrane pores. The drop or bubble–slot experiment used a slot with different width. It has been found that the squeezing of an oil drop in the slot is really determined by the pressure applied and velocity of the surrounding fluid. Cross flow microfiltration experiment was conducted using tubular slotted pore membrane with rotation to generate shear on the surface of membrane. Kerosene and crude oil were tested using 5.3 and 7.5 microns membrane at different rotation speed and permeate velocity. Experimental results indicated that in a no blocking condition, the movement of oil drops was determined by shear force and permeate drag force. While in blocking condition, the rejection of oil drops was determined by the formation and characteristic of the secondary membrane formed on the surface of membrane. Blocking will improve the filtration performance in relation to oil rejection, but it will increase the pressure needed or decrease the flux rate through the membrane.

Keywords : Microfiltration, slotted pore filter, emulsion, drop, crude oil

1.0 INTRODUCTION

Recently, water containing dispersed oil has become of interest to many industries. Oil contaminated water can be found in many industries, including metal finishing, food, textile, and petroleum industries. The presence of dispersed oil in water can be harmful to aquatic life, as it attenuates light and perturbs normal oxygen transfer mechanisms. In the petroleum industry, oil-in-water emulsion is mainly produced in the recovery of crude oil. The oil-in-water emulsion produced in crude oil

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recovery can cause problems at different stages of the production in petroleum industry. Produced water cannot be injected again into the well or discharged into the body of receiving waters, because it contains high concentration of oil, grease and suspended particles. Because of this problem, such industries have tried to separate oil from water before discharge of the water or using it again in the recovery of crude oil by reinjection into the oil bearing strata. Many methods have been carried out to separate oil from water in the petroleum industry. Such methods are chemical demulsification, gravity or centrifugal settling, pH adjustment, filtration, heat treatment, membrane separation and electrostatic demulsification [1]. Each of these methods has its own advantages and disadvantages. For example, demulsification process using a hydrocyclone can reduce the content of free oil but is ineffective for removal of fine oil [2]. Hydrocyclones used for treating oil-in-water emulsion can remove oil drops with diameters in the range from 10 to 300 μm, but remove smaller size drops less efficiently [3]. A potential problem associated with emulsions produced from crude oil production where the dispersed phase consists of very stable micron and submicron droplets, as these cannot be treated by gravity flotation due to the long residence time required for the droplets to rise and chemical addition is usually necessary to encourage coalescence and break the emulsions [4]. Therefore, new improved technologies have been investigated to overcome this problem. One of these methods is membrane technology.

Several studies have been conducted to prove that membrane technology, especially ultrafiltration and microfiltration, are suitable for treating oil-in-water emulsion. In the vegetable oil industry, membranes can be used in many areas, such as degumming [5]. Different materials of membrane have been used to separate oil from water. Polymeric membrane, such as PVDF and PTFE, has been used to separate oil from water in corrugated and flat dimensions [6]. Polyacrylonitrile (PAN) membrane has been also used and flux can be adjusted between 100 up to 2000 l m^{-2} hr^{-1} [7]. Another material, which has been used to separate oil from water is porous glass. This membrane material really acts as a wetting and coalescing medium, and its pore size and trans membrane pressure exerted cause droplets to deform and enter the membrane pore, and to rupture eventually [8]. An investigation using polymeric and ceramic membrane called Ceramesh to separate oil-in-water emulsion using dead-end and cross flow modes was conducted by Koltuniewicz *et al.* [9]. The results showed that the Ceramesh membrane is superior to polymeric membranes because its pore size is tighter than polymeric membranes. A new improvement in separation of oil from water using membrane is the utilization of metal microfilter. Metal microfilters with slotted pore geometry have been used to filter colloidal suspensions using cross flow filtration to provide shear rate at the filter surface. The slot width varied from 10 to 20 μm. The feed was a suspension with particle diameters similar to the pore diameters. Results indicated that under identical conditions, the slotted pore geometry did not foul as badly as the circular pore filter. Flux of up to 9000 l m^{-2} hr^{-1} were possible, however, for 2 μm slot width fouling was identified at fluxes as low as 200 l m^{-2} hr^{-1}. This new microfilter membrane design did not suffer from internal plugging of the filter matrix because it does not have an internal structure [10]. Cumming *et al.* [11] have also investigated microfiltration of oil/ water emulsion using a stirred cell fitted with Nuclepore filters with pore sizes of 2, 5, 8 and 10 μm. The filters were produced by nuclear bombardment followed by chemical etching and can be classified as true filters because they have pores which pass directly through the filter from one side to the other. A simple theory based on interfacial tension, contact angle and pore size is described and the results compared with experimental results, which treated oil emulsion drop size in the range from 1-40 μm. There was a good similarity between model and experiment for the 2 μm filter, but the model reliability decreased with increasing pore size.

Among the issues related to separation with membranes that have received extensive attention are concentration polarization and fouling. Concentration polarization is influenced by a number of factors such as flux, trans membrane pressure, cross flow velocity and the flow regime. Filtration in rotating systems has been suggested as being an efficient way to substantially reduce the effects of concentration polarization [12, 13].

From many studies which have been conducted by many researchers to separate oil-in-water emulsion, it can be suggested that a further study of separation of oil from water using membrane is still needed, especially to enhance the flux of permeate and the rejection coefficient. This research will try to use true surface filter microfiltration membranes to separate oil from water because true surface filters have pores which pass directly through the filter from one side to the other side, so it will exclude the possibility of internal fouling of the filter.

2.0 THEORY

2.1 Deformation of Oil Drop in Pore or Slot of Membrane

Another phenomenon which should be considered seriously in the separation of oil from water using membrane is the deformation of oil drops in the pore of the membrane. The successful separation of oil-in-water emulsion using membranes is determined by the rejection of oil drops, especially big drops. Clearly, when filtering deformable material such as oil control of trans membrane, pressure is critical to prevent large drops from being pushed through small pores. Figure 1 is a typical description of deformation of a big drop on the surface of membrane:

It is possible to define a threshold pressure as being the pressure to be overcome in the transmission of a drop through a pore. An analysis based on the Young-Laplace equation developed for circular pore membranes and considering the radius of curvature of the advancing portion of the drop and the radius of curvature of the lagging interface, leads to the following expression for threshold pressure (P_{th}) [14] :

$$P_{th} = 4\gamma_{o/w}\frac{\cos\theta}{d_p}\left(1-\left[\frac{2+3\cos\theta+\cos^3\theta}{4\left(x/d_p\right)^3\cos^3\theta-\left(2-\sin\theta+\sin^3\theta\right)}\right]^{1/3}\right) \tag{1}$$

Where d_p is the pore diameter. Equation (1) is valid for circular pore membranes and it is possible to apply it in a rearranged form to deduce the oil drop diameter, which will pass through a given pore size when a pressure is applied to the membrane. In this instance, the pressure applied to the membrane is the total pressure drop over the membrane. However, in the case of a slotted pore the full pressure

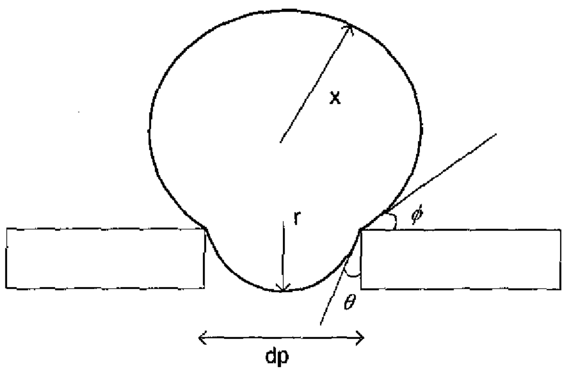

Figure 1 Schematic of an oil drop at pore entrance of membrane

differential over the membrane does not cause the oil drop to deform and pass into the permeate as the drop cannot plug the pore completely off. There will be a region on either side of the drop, sitting on a filtering slot, through which liquid can flow into the permeate by flowing around the drop, unlike in the case of the circular pore. The squeeze of a big drop is influenced by many factors, one such factor is velocity of the feed solution, which can be correlated to shear rate on to the surface of the membrane. The trans membrane pressure relative to the membrane pore size and size distribution of oil drops in the feed has also been reported to be critical operating variables as are membrane composition and solution chemistry [11, 15].

3.0 METHODS

The filtration experiments were done using slotted pore membrane developed by Micropore Technology, UK. Two membranes used in the experiment were tubular slotted pore microfiltration membranes with different slot widths to compare their filtration performance. The diameter of each membrane was 1.5 cm and its length was 15 cm. In order to determine the actual cut off point of each membrane used in these experiments, all the membranes were used to separate and fractionate a suspension of latex particles with concentration of 100 mg l^{-1}. Cut off point here means the minimum size of particle that can be rejected 100% by the membrane during filtration. This experiment was done in dead end mode at linear velocity of permeate 0.17 cm s^{-1}. The cut off point of membranes was analyzed from rejection curve, which was obtained by analyzing the amount of oil drops in permeate and feed samples using a Coulter Multisizer II. The result of this experiment would be compared with oil filtration experiment result at the same linear velocity of permeate.

The oil filtration experiments were done using two different challenge emulsions, i.e. kerosene-in-water emulsion and crude oil-in-water emulsion. Physical properties of these oils, such as density, viscosity and interfacial tension have been measured using picnometer, viscometer and pendant drop method, respectively and can be seen in Table 1.

Table 1 Physical properties of kerosene and crude oil at 20°C

Substance	Density (kg m^{-3})	Viscosity (cP)	Interfacial tension (mN m^{-1})
Kerosene	800	2.1	19.1
Crude oil	860	75	21.7

The feed emulsions were prepared by dispersing kerosene or crude oil in distilled water. As kerosene or crude oil will not mix with water, it was homogenized for 1 minute using a homogenizer at 5000 rpm speed of rotation. The amount of kerosene used in the experiments was 1000 mg dispersed in 1 liter distilled water, while the concentration of crude oil was fixed at 200 mg l^{-1}. During experiment, the emulsions were placed in the beaker and stirred by magnetic stirrer to prevent coalescence and floating of oil drops to the surface of the emulsion. The filtration experiments were conducted in two different modes, i.e. dead end filtration and cross flow filtration using the rotation of the membrane to generate shear on the surface of the membrane. Typical surface of the membrane used in the experiments can be seen in Figure 2.

Two membranes have been tested using latex particles and from the tests, the cut off points of each membrane were 7.5 micron and 5.3 micron, respectively. Open area of 7.5 micron membrane was 5%, while for 5.3 micron membrane was 2%. Before and after each experiment, membranes were cleaned using hot 'fairy liquid' for 10 minutes in a sonification bath then they were also treated

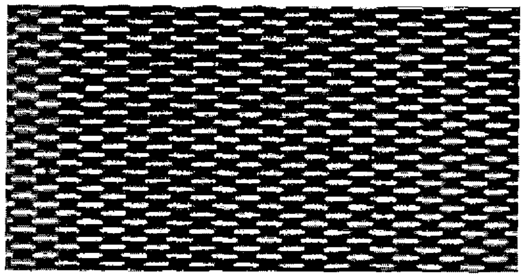

Figure 2 Array of slots of membrane

by contacting it with 'Silwet' 3%-w solution for 10 minutes in a sonification bath to make the surface of membrane more hydrophilic. Membranes were contacted with the feed emulsion by placing them in the feed tank for 1 hour. A peristaltic pump was used to provide pressure difference through the membrane and induce the permeate flow. During each experiment, pressure difference was recorded in a personal computer (PC) using Pico Technology software, while the flux of permeate was recorded using a balance connected to the PC. Samples of feed and permeate were taken after each experiment or after 60 minutes. The ability of membrane to reject oil drops from water was measured and analyzed using oil rejection curve. This curve was obtained by analyzing the amount of oil drops in permeate and feed samples using a Coulter Multisizer II. The schematic diagram of equipment used in filtration experiment can be seen in Figure 3.

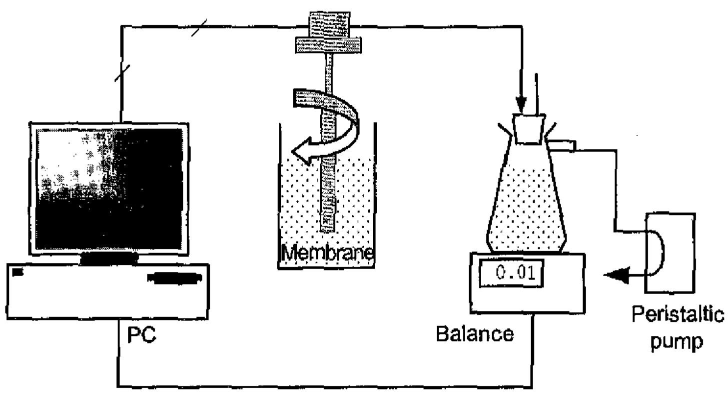

Figure 3 Equipment setup for oil filtration

Another experiment, which was drop – slot interaction experiment, was also conducted to visualize the movement of a drop of liquid or air bubble with different diameters and a slot with different widths and also to record the pressure needed to make the drop or air bubble squeeze through the slot. Two liquids were used, i.e. paraffin oil and surfactant Tween 20. Table 2 below describes physical properties of bubble and drop used during experiment.

Table 2 Physical properties of bubble and drop

Type of drop/bubble	Temperature (°C)	Density of liquid (kg m^{-3})	Interfacial tension (N m^{-1})
Bubble of paraffin oil	20	804	0.0145
Drop of paraffin oil in Tween 20	20	1100	0.001

The experiments were run using two kinds of cell, i.e. cell with 0.098 mm slot width and cell with 0.06 mm slot width, which put in horizontal position. In each cell, the movement of air bubble and drop of liquid was observed and analyzed. Typical schematic diagram of cell or module geometry can be seen in Figure 4.

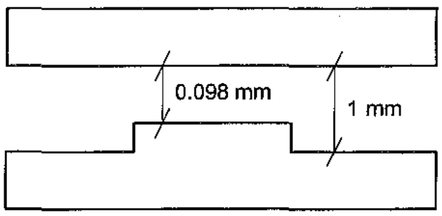

Figure 4 Cell with 0.098 mm slot width

A drop of liquid or air bubble was prepared and placed on one side of the cell/module as can be seen in Figure 4. The diameter of air bubble or oil drop was measured by analyzing its image using paint software. An experiment was conducted by flowing air from the main air valve into the module and air pressure was changed until the drop/bubble squeezed into the other side of the cell. During the experiment, the pressure needed to make the drop or bubble squeezed through the slot was recorded using a pressure transducer and Pico Technology Software, while the movement of the drop or bubble when squeezed through the slot was recorded and videoed using a camera, VCR, and PC. The movement of an air bubble in paraffin oil in the 0.098 mm slot, a drop of paraffin oil in the 0.098 mm slot, and an air bubble in paraffin oil in the 0.06 mm slot have been investigated in this experiment.

4.0 RESULTS AND DISCUSSIONS

4.1 Cut off Point of Membrane

Filtration of solid particle or latex particle gave information about the exact cut off point of membranes used during experiment. From oil rejection curves in Figure 5, it can be seen that membranes have cut off point of 5.3 and 7.5 microns, respectively.

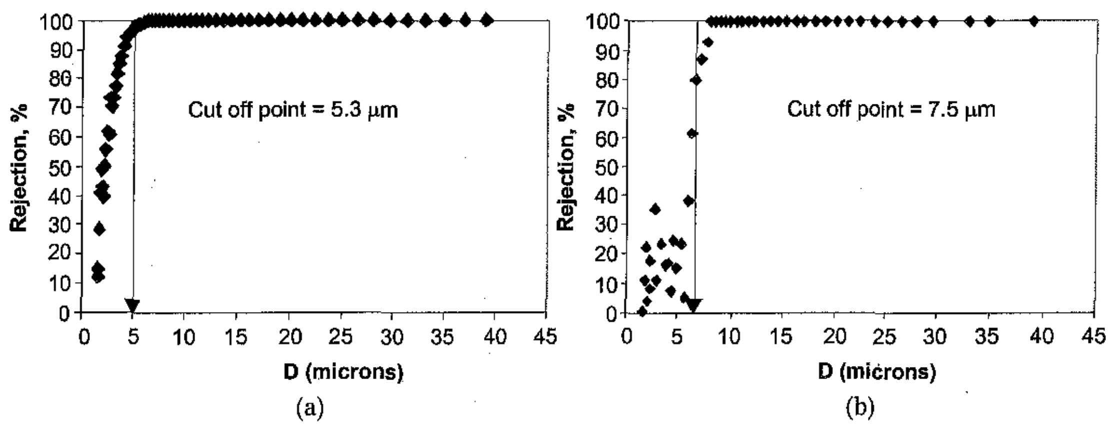

Figure 5 Solid particle rejection for (a) 5.3 μm and (b) 7.5 μm membrane

This information can give a rough prediction for rejection behavior for filtration of kerosene-in-water emulsion. Initially, it can be expected that cut off points obtained from solid particles filtration will be similar with cut off points from kerosene-in-water filtration. For 5.3 microns membrane, oil drops with diameter greater than 5.3 microns will be completely rejected by the membrane, while 7.5 microns membrane oil drops with diameter greater than 7.5 microns will be completely rejected by the membrane. From experiment at similar linear velocity of permeate, cut off points for each membrane can be seen in Figure 6.

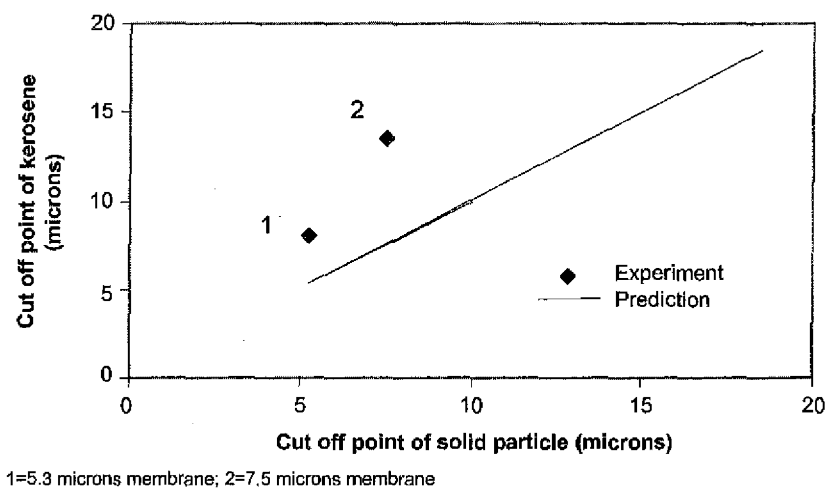

Figure 6 Cut off point of solid particle and kerosene at similar linear velocity (v = 0.17 cm s^{-1}) and similar feed concentration of 100 mg l^{-1}

The cut off points of kerosene drops are larger than cut off points of solid particle for all membranes tested. This phenomenon happened because oil drops, which have diameter larger than slots of membrane can deform and squeeze through the slots of membrane. Many factors affect the squeezing of these big drops, such as trans membrane pressure, velocity of permeate, and physical properties of the drop and filter media. When an oil drop rests on a circular pore, the entire trans membrane pressure field distorts the drop encouraging passage, but when resting on a slotted pore the pressure field causing distortion is only due to the permeate fluid drag force on the oil drop, as liquid can still pass around the drop and through the region of the slot left un-plugged by the oil drop. From an experiment using single drop or bubble and small model slot, a correlation of drop size and slot width with critical pressure (pressure needed to make drops or bubbles squeeze through small model slot with different width) is illustrated in Figure 7. The drop size and slot width have been normalized by dividing the drop diameter by slot width. A limited variety of interfacial tensions and geometry approaching the slot were tested and the data appears to correlate to a single line in most cases.

There is some deviation from the correlation for large values of drop diameter with respect to slot width. Under these circumstances, a much lower pressure is required in order to pass the drop into the permeate. Many factors can affect the critical pressure needed such as physical properties of fluid, drops and bubble, and slot dimension used in the experiment. Further investigation is still needed to get a clear understanding on this phenomenon. In this experiment, big drops were investigated further and in all cases, it was possible to observe severely deforming drops, where the drop adopted a dumb-bell shape in order to pass through the slot. In some cases, this led to drop breakup within the slot.

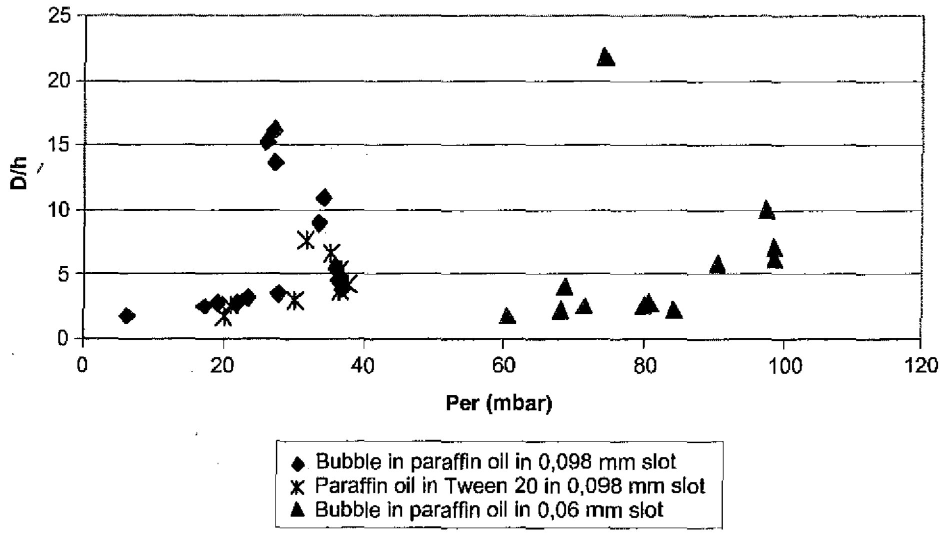

Figure 7 D/h vs critical pressure for different drop/bubble in different slot

A dumb-bell shape drop entering the slot is illustrated in Figure 8, where the convective flow is from left to right. At Stage 2 (Figure 8), the drop has adopted the dumb-bell shape with the bell on the right side within the slot, the bell on the left remaining in the much larger channel approach to the slot. At Stage 3, the drop has entirely passed into the slot and has reformed into a circular shape during its passage through the slot. At Stage 4, the drop has left the slot and has returned to its original shape within a flow channel much deeper than the slot. The correlation in Figure 7 was found to apply for all the drops tested.

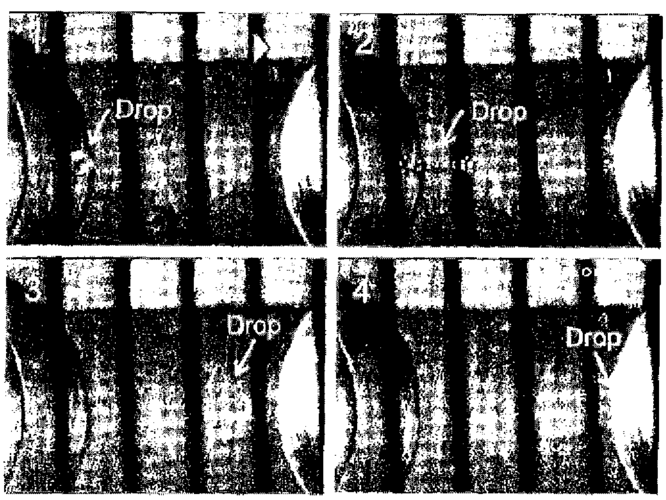

Figure 8 The movement of a bubble in paraffin oil

4.2 Kerosene Filtration

Oil filtration experiment using kerosene-in-water emulsion as challenge emulsion was done using 5.3 and 7.5 microns membranes. Experiment using 7.5 microns membrane at low and high flow rate of pump experienced no blocking phenomenon. The blocking phenomenon was observed by recording the pressure during the experiment. The experiments were conducted in constant flux filtration, so any deviations occurred such as the formation of secondary membrane and blocking phenomenon will be accommodated by the increase in pressure difference across the membrane.

From Figure 9, it can be seen that for a short time after starting an experiment, the pressures in all experiments increased to certain values and then stayed relatively constant. The pressure increased because the flow rate of pump was not constant in the initial stage of experiment. From Figure 8, the pressure needed to make the permeate flow is very low compared with a conventional membrane. For a 7.5 micron membrane, the highest flux obtained in the experiment was 1820 liter $m^2 h^{-1}$ (LMH) or equivalent to linear velocity of 1.531 cm s^{-1}, and the pressure needed to obtain that flux was only 95 mbar. For this membrane, the movement of drops into the slots of membranes was not affected by the secondary membranes and the deformation of drops with diameter larger than slot width through the slot was possible.

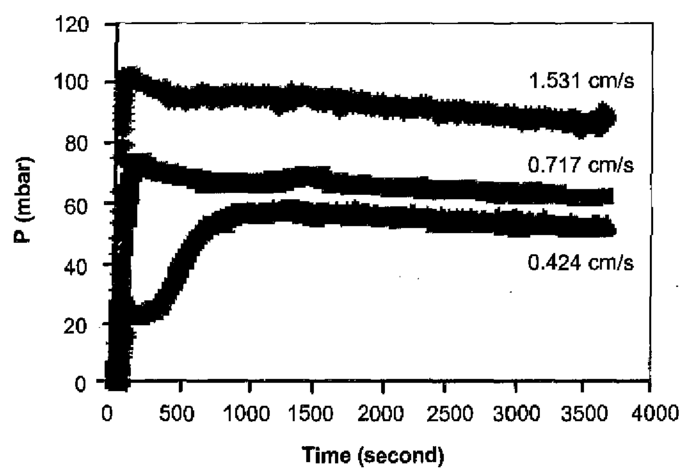

Figure 9 Pressure curve during kerosene filtration using 7.5 microns membrane

Kerosene filtration experiment using 5.3 microns membrane was done at low linear velocity of permeate, i.e. 0.3031 cm s^{-1} and 0.174 cm s^{-1}. In the experiment with this membrane, the pressures during experiment increased significantly, which indicated that there were a significant amount of oil drops settled on the surface of membrane. This phenomenon can be seen from Figure 10.

The blocking phenomenon observed during the experiment with this membrane will influence the overall performance of membrane, including oil rejection. This is because the blocking of membrane by oil drops actually will form another separating layer, which can reject oil drops and prevent them from reaching the membrane to enter the permeate side. This can enhance the ability of membrane to reject oil drops. From the consideration of oil rejection this will be a beneficial effect, but it will also make the membrane life and the permeate flux lower than if there is no blocking during the operation.

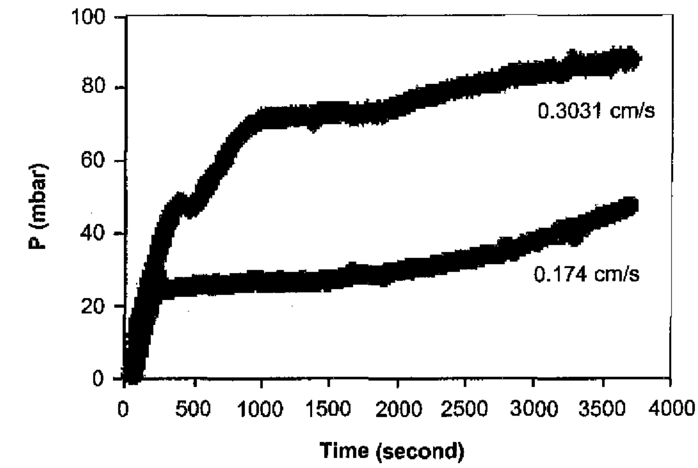

Figure 10 Pressure curve during kerosene filtration using 5.3 microns membrane

4.3 The Influence of Rotation on Kerosene Rejection

As 7.5 microns membrane did not have a tendency to be fouled by kerosene–in–water emulsion, it would be very interesting to investigate the oil rejection efficiency of this membrane.

Figure 11 shows oil rejection curve for oil filtration experiment using 7.5 microns membrane at linear velocity of permeates 0.144 cm s^{-1}. It clearly shows that filtration with rotation can help the rejection ability of the membrane, especially for small drops. It also indicates that at this velocity of permeate, the drops move away from the surface of the membrane because of shear force generated by rotation. As can be seen from Figure 11, surprisingly the cut off point for filtration with rotation is similar with the cut off point obtained by solid particles (~ 7.5 microns). The movement of oil drops into the surface of the membrane in no rotation experiment is only influenced by the liquid drag force and force caused by the density difference between oil and water. While in a rotating system, the movement of oil drops is influenced not only by these forces, but there are also shear forces generated by the rotation of membrane involved.

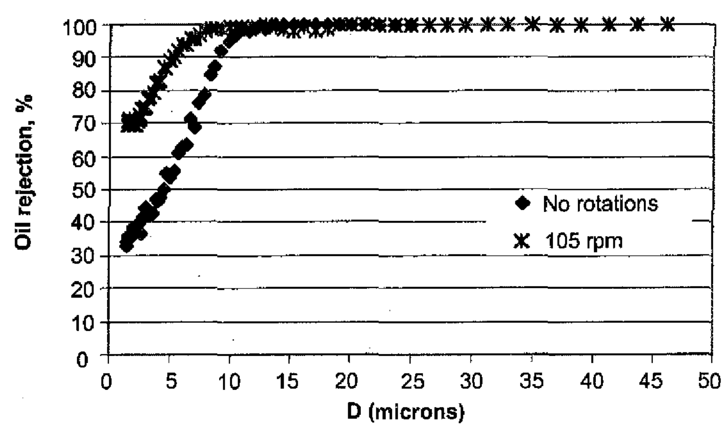

Figure 11 Rejection curve for kerosene filtration using 7.5 microns membrane at v = 0.144 cm s^{-1}

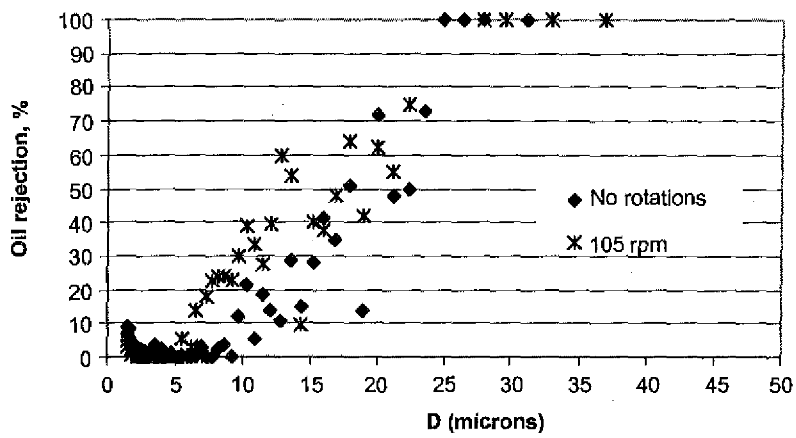

Figure 12 Rejection curve for kerosene filtration using 7.5 microns membrane at v = 1.531 cm s^{-1}

From Figure 12, it can be seen that at higher permeate velocity (v = 1.531 cm s^{-1}), the rejection curves for no rotation experiment is slightly better for small drops. It indicated that at high velocity, the magnitude of shear force and permeate drag force will determine whether the drops rest and squeeze through the slot of the membrane or move away through the slot of membrane. At higher permeate velocity, the rejection of small drops is worse than at lower permeate velocity. As an example, 70% rejection at v = 1.531 cm s^{-1} was for oil drop with diameter of 20 microns, while at v = 0.144 cm s^{-1} the 70% rejection were obtained for 2.5 microns. In this experiment, the ability of a membrane to reject oil drops is really determined by the velocity of permeate and also by the magnitude of permeate drag force and shear force caused by rotation.

Theoretically, in almost all filtration processes, there is the formation of a secondary membrane often called dynamic membrane on top of the primary membrane. This secondary membrane is always formed, whether automatically or by design from constituents in the feed. The formation of the secondary membrane will alter the performance of the primary membrane, in terms of rejection. The characteristics of secondary membrane or dynamic membrane formed during experiment in this research, such as thickness, structure, and its resistance, have not yet measured clearly. Experiments with 5.3 micron membranes resulted in a blocking phenomenon during filtration of kerosene-in-water emulsion. This phenomenon was observed by recording the pressure during experiment as has been shown in Figure 10. Many methods have been tried to overcome this problems. One such method was to induce shear at the surface of membrane. In this research, shear at the surface of the membrane was developed by rotating the membrane. Filtration experiments using 5.3 microns membrane were done to compare the performance of rotating system and dead end filtration. In cross flow microfiltration, the formation of secondary membrane brings about some advantages, such as the rejection of particles becomes better than that of 'naked' membrane and there is a certain degree of retention of species, which are intended to pass through the membrane. Due to that, it will be very useful to also analyze the rejection curve for this experiment.

As the secondary membrane hindered the interaction between oil drops and the surface of membrane, we can expect that the rejection curve obtained from experiment without rotation will be slightly better than the rejection curve obtained from experiments with rotation. The rejection of small oil drops from experiment without rotation was better than from experiment with rotation. For example for 5 microns oil drop at experiment with linear velocity of 0.3031 cm s^{-1}, the rejection was 77% for experiment with rotation and for experiment without rotation its rejection was 92%. It is clear that the formation of secondary membrane will give an advantage in relation to oil rejection, but it will give disadvantage in relation to flux rate or pressure during operation.

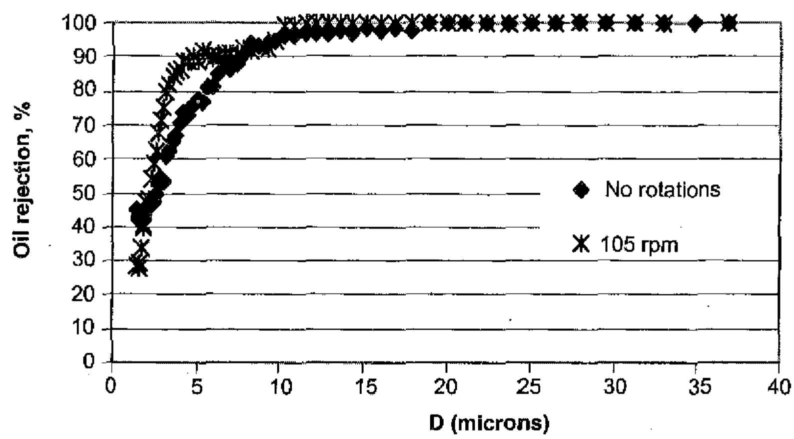

Figure 13 Rejection curve for kerosene filtration with 5.3 microns membrane at v = 0.3031 cm s^{-1}

4.4 Crude Oil Filtration

Crude oil can vary greatly in composition, viscosity, density, and flammability. The chemical composition of crude oil varies between regions and even within the same geological formation. Based on this information, it can be said that the properties of crude oil can be quite different to kerosene because there will be a possibility of the presence of solid particles in the challenge dispersion. So it will be interesting to observe rejection curve for crude oil filtration with 7.5 microns membrane with and without rotation.

Crude oil filtration using the 7.5 microns membrane experienced blocking of membrane by crude oil. This was indicated by the increase of pressure during the experiment. Shear rate generated by the rotation slightly improve the performance of membrane in relation to pressure difference across the membrane, but rotation used in this experiment was low enough to prevent the formation of secondary membrane on the surface of membrane. Therefore, it was not surprising that there was no significant different in the rejection curve for experiment with and without rotation except for small

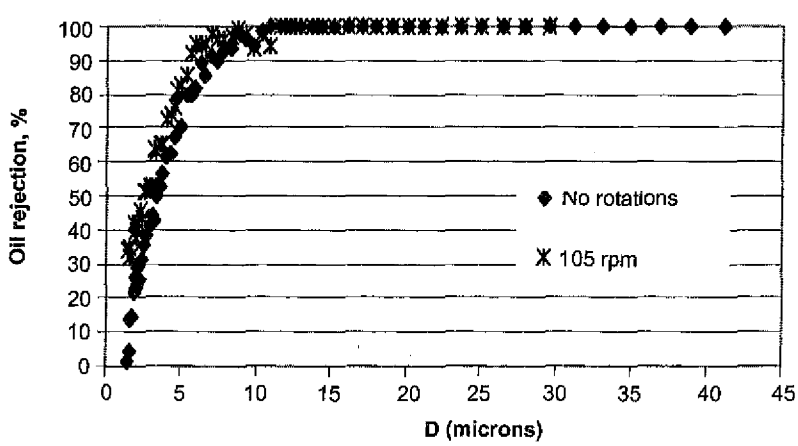

Figure 14 Rejection curve for crude oil filtration using 7.5 microns membrane at v = 0.144 cm s^{-1}

drops where the rejection in experiment without rotation was better than with rotation experiment. An interesting phenomenon can be observed here that the cut off point observed for this membrane is about 8 microns or almost the same with the cut off point obtained using solid particles.

5.0 CONCLUSIONS

Oil-in-water emulsions are important to many industries, including the petroleum industry, because of its impact on process production in such industries and its impact to the environment. In the petroleum industry, oil-in-water emulsions are produced during the production of crude oil. Many attempts have been tried to overcome this problem. This research has investigated the separation of oil from water using slotted pore microfiltration membrane. Experimental results from oil filtration indicated that there are two different phenomena observed during experiments, i.e. blocking and no blocking conditions. In the no blocking condition, the magnitude of shear force generated by rotation and permeate drag force will determine the movement of oil drops. While in blocking condition, the formation of secondary membrane will determine the behavior of membrane during filtration of oil drops. Blocking will improve the efficiency of separation but on the other hand, will improve the pressure or decrease the flux rate.

Direct observations on an analogue system (air bubbles in liquids) showed that passage of drops through a slot can still readily occur if the drop deforms into a dumbell type shape. Under these circumstances, drop breakage is also facilitated resulting in oil passage into the permeate. These effects become less significant with smaller drop size.

NOMENCLATURE

x diameter of drop (microns)
d_p pore diameter (microns)
P_{th} threshold pressure (Pa)
θ, ϕ contact angle
$\gamma_{o/w}$ oil/water interfacial tension (N/m)

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