Removal of suspended solids and residual oil using membrane separation technology

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Abstract

The application of membrane separation technology can be an effective means of treating palm oil mill effluent (POME). Palm oil mill effluent was first pretreated to remove the high content of suspended solids and residual oil. The pretreatment process has three stages of separation. The first stage was removal of suspended solids using flocculation while the second and third stages were removal of residual oil using solvent extraction and adsorption respectively. These three stages were important in reducing membrane fouling at the membrane separation process. Membrane separation was subsequently applied to remove any residual suspended solids and oil remaining after the pretreatments. Several operating conditions such as membrane type, pH and pressure were varied to find the optimum conditions for membrane separation process. The treatment efficiency of the process was measured as percentage removal of suspended solids and oil respectively. It was found that, in membrane separation process, GH and CE(GH) membrane gave 63% and 49% reduction in suspended solids and residual oil respectively at pH 9 and pressure of 1000 kPa.

Keywords : Palm oil mill effluent (POME), flocculation, solvent extraction, adsorption, membrane separation.

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1. Introduction

Malaysian palm oil industry has grown to become the most important agriculture based industry. Currently there are about 3.0 million hectare of land under palm oil cultivation and 300 palm oil mills to process the fresh fruit bunches (FFB) [1]. The processing of FFB primarily for palm oil results to concomitant production of wastes in the form of palm oil mill effluent (POME) [2]. Fresh POME is a colloidal suspension containing 95 - 96 % water, 0.6 - 0.7 % oil and 4 - 5 % total solids including 2 - 4 % suspended solids which are mainly debris from palm fruit mesocarp. The characteristics of a typical POME is shown in Table 1 [3].

Table 1
Characteristics of palm oil mill effluent

Parameter	Concentration*
pH	4.7
Oil and grease	4 000
Biochemical oxygen demand	25 000
Chemical oxygen demand	50 000
Total solids	40 500
Suspended solids	18 000
Total volatile solids	34 000
Ammoniacal nitrogen	35
Total nitrogen	750

*All parameters in mg/L except pH.

Over the last two decades, several innovative treatment technologies have been successfully developed and employed by palm oil mills to treat their POME [4-5]. They are based on anaerobic, aerobic and facultative processes that rely on suitable bacteria to break down the organic matters. These conventional biological treatments of anaerobic and aerobic or facultative digestion systems need proper maintenance and monitoring as the processes rely solely on microorganisms to break down the pollutants.

Anaerobic digestion of POME (or organic matters) also generates vast amounts of biogas, about 28 cubic meters per tone of POME treated. Biogas contains about 65 percent of methane and 35 percent of carbon dioxide and trace amount hydrogen sulphide. Only very limited palm oil mills harness biogas for heat and electricity generation [1]. Both methane and carbon dioxide are green house gases and methane is more potent. Biogas is also corrosive and odorous. Thus its emission to environment is undesirable.

Besides the biological treatment, evaporation process has been suggested to treat POME [3]. POME containing 3 – 4% total solids was used as feed for the evaporation process. A concentrate of 20 – 30% solids content was produced and the water is recycled back into the plant. About 85% of the water in the POME can be recovered as distillate. Unfortunately, energy requirement is a major constrain in this process. It is reported that 1 kg of steam is used to evaporate 1 kg of water from POME.

Membrane separation technology is in a state of rapid growth and innovation. Over the last 40 years and particularly in the last two decades, numerous different separation processes have emerged in which synthetic membranes play a prominent role. Nowadays, membrane separation technology is widely used in the medical, semiconductor, beverage, pharmaceutical and chemical industries where water quality is of paramount importance [6]. Membrane technology, which can achieve a reduction in the volume of wastewater by a factor of typically five to ten times, offers potential savings on effluent discharges as well as producing a clean water stream that can often be reused as wash water or as cooling water within the facility [7].

POME has been found to contain many valuable plant nutrients in substantial amount. This positive development has resulted in a paradigm shift in the management of POME. Recently, it has changed the concept of treatment and disposal to beneficial utilization. It is believed that membrane separation technology will be able to treat POME in a more beneficial way. In this present study, membrane separation technology was used to treat POME.

In order to apply membrane separation technology, pretreatment processes were carried out to reduce the high content of suspended solids and residual oil in the fresh sample of POME. The pretreatment process has three stages of separation. The first stage was removal of suspended solids using flocculation while the second and third stages were removal of residual oil using solvent extraction and adsorption respectively. These three stages were important in reducing membrane fouling at the membrane separation process. Membrane separation was subsequently applied to remove any residual suspended solids and oil remaining after the pretreatments. A proposed pretreatment and membrane separation processes scheme is shown in Figure 1.
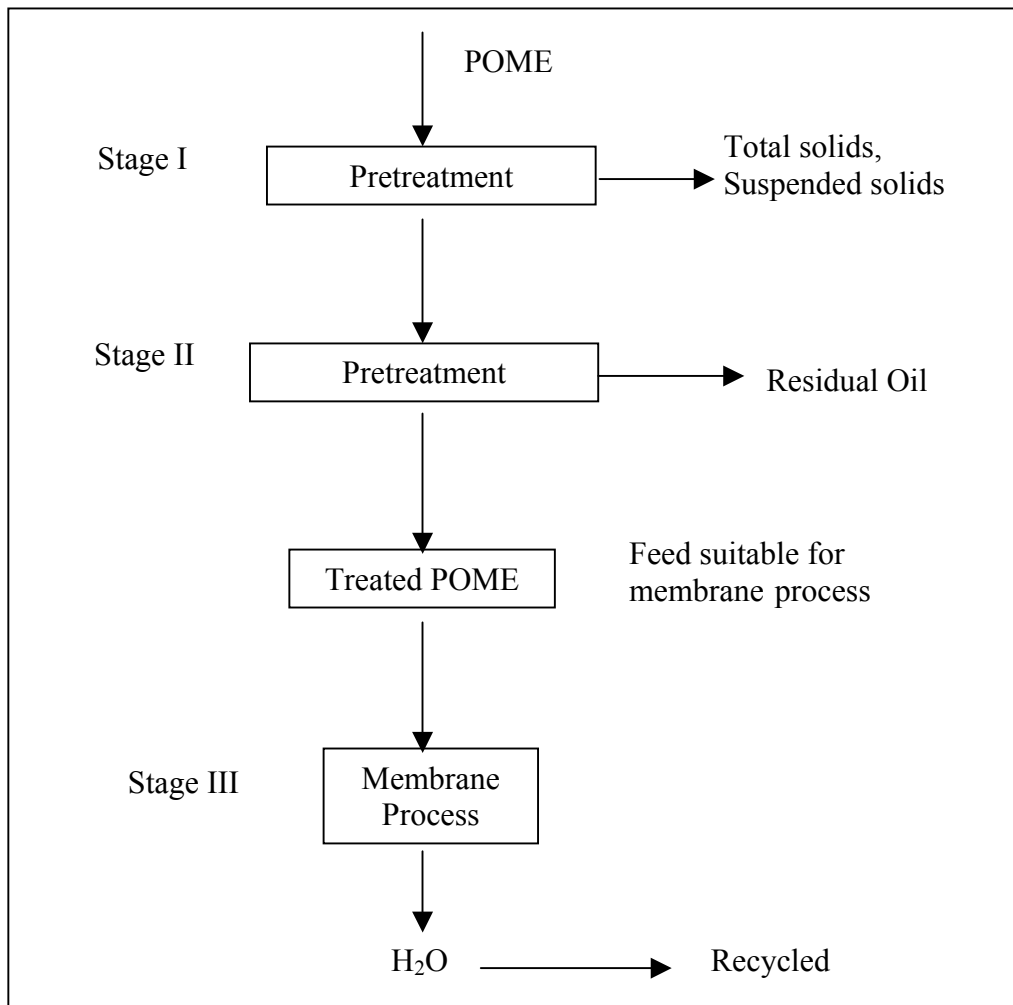

FIGURE 1: Schematic Diagram of Pretreatment and Membrane Separation Processes.

2. Materials and methods

Fresh sample of POME was collected from Felcra Nasaruddin Palm Oil Mill, Ipoh, Perak. Technical grade aluminium sulphate hydrate [$Al_2(SO_4)_3 \cdot xH_2O$ (x=13-16 H_2O)] was used for the preparation of the alum solution in the flocculation process. Six technical grade solvents which are hexane, benzene, pentane, petroleum benzene, petroleum ether and heptane were used in the solvent extraction process. For the adsorption process, a sample of synthetic rubber latex with 15.98 m^2g^{-1} surface area was used as an adsorbent. Four different types of ultrafiltration membranes (G-series membrane; GN, GM, GK and GH) and one type of reverse osmosis membrane (CE membrane) were tested to extract the residual oil from POME. The molecular weight cut off (MWCO) of GN, GM, GK and GH is 10000, 8000, 3500 and 2500 respectively. The effective membrane surface area was 21.24 cm^2.

Figure 2 shows the schematic diagram of membrane separation process rig that is used in this project. This unit contains a membrane cell, flow in and flow out pressure gauges and high-pressure pump.
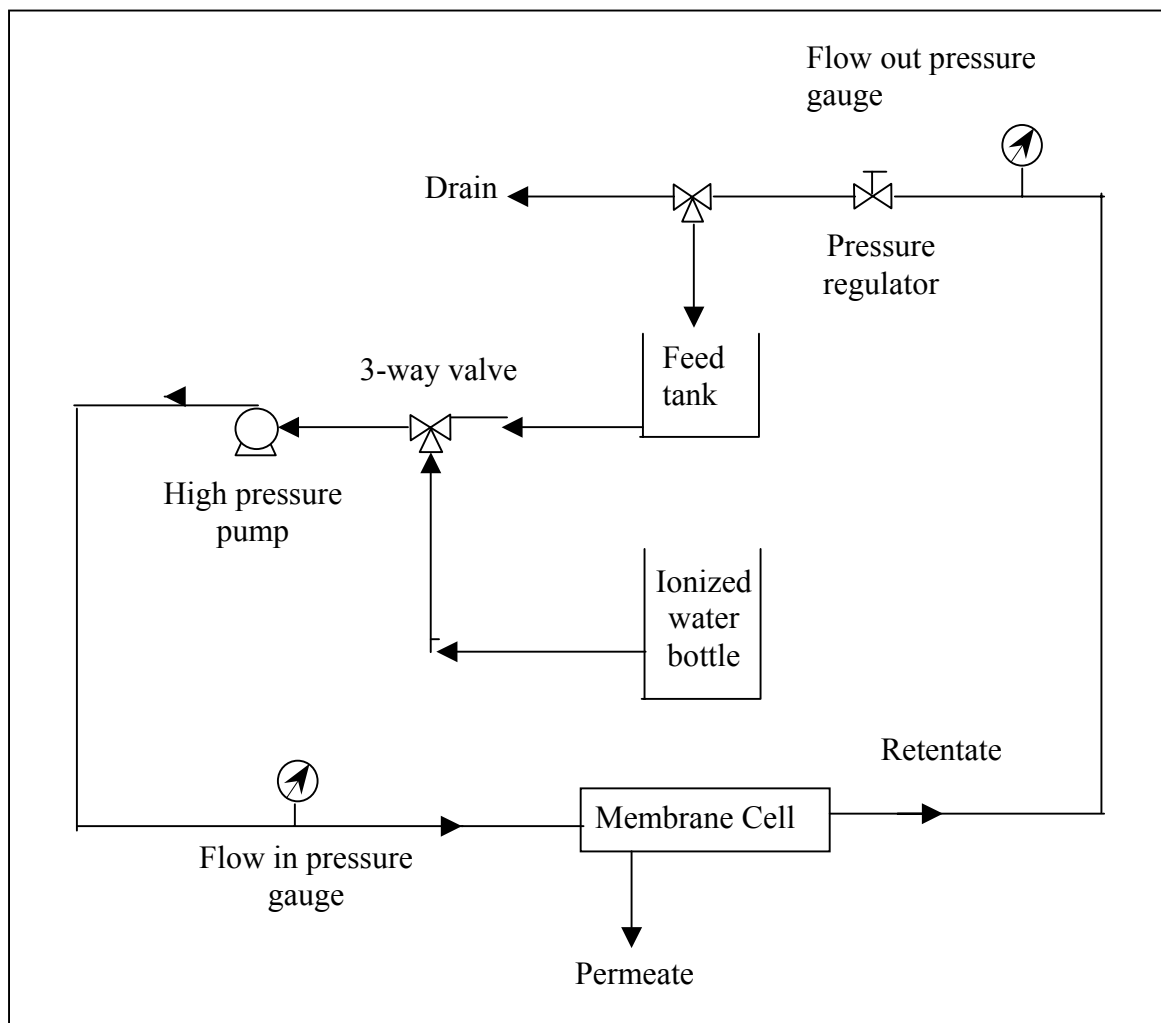
FIGURE 2: Schematic Diagram of the Membrane Separation Process Rig.

An ultrafiltration membrane was placed inside the membrane cell. Ionized water was first fed to the pressure membrane cell. After stabilization of about 15 minutes, permeate and control valves were closed until the desired pressure was achieved (10 bars). Then, the permeate valve was opened. Control valve was used to control the pressure of the system if necessary. This procedure was continued for another 15 minutes to stabilize the membrane. The feed was then changed to the sample of POME. The system was left for stabilization for 15 minutes. After 30 minutes of running the system, permeate flux was recorded. The turbidity (NTU) value was later analyzed. After 5 hours, the feed was changed back to ionized tank in order to clean and remove any particles off the system. The parameters varied were types of membranes used, pH of the sample (4 – 9) and pressure of the system (10 – 15 bars).

3. Chemical analysis

For suspended solids analysis, about 5 cm^3 of the supernatant sample was pipetted and diluted to 25 cm^3 with distilled water. This analysis was based on the nephelometric turbidity units (NTU), which represented the colloidal and residual suspended matter. For that purpose, 2100P Turbidimeter (Hach. Company, Colo, U.S.A.) was used.

In order to determine the oil concentration in the treated sample, the extracted POME was transferred to a separating funnel. The POME container was rinsed with 30 ml of petroleum ether and solvent washings was added to separating funnel. The mixture was shake vigorously for 2 min and then left for 5 min. The aqueous layer was drain into sample container. Next, solvent layer was drained through a funnel containing a filter paper and 2 g anhydrous sodium sulphate, both of which have been solvent-rinsed, into a clean, tared conical flask. The extraction was repeated with another two portions of 30 ml petroleum ether. The entire sample container was rinsed each time with the petroleum ether before adding it to the separating funnel. The solvent was then distilled off using the rotary evaporator. The drying was completed in the oven at 103 ^{o}C for 5 to 10 min. The flask was cooled in a desiccator for about 30 min and weighed. The drying and cooling steps were repeated until the weight becomes constant.

4. Results and discussion

An alum concentration of 4000 mg dm^{-3} and above was found to produce the best reduction in suspended solids. Meanwhile, the best supernatant quality was obtained at pH 4 with a turbidity value of 1751 NTU. At 150 rpm, the flocs performed was bigger and stronger which readily settled to the bottom of the beaker, thus give the best rapid mixing rate. The lowest turbidity value was obtained at mixing and sedimentation time of 90 min and 270 min respectively.

For solvent extraction process, the best ratio of solvent to POME was obtained at 0.6 with mixing rate of 200 rpm and mixing time of 20 min. This result was achieved at pH 4. Among the six solvents tested in this study, n-hexane was found to be the best solvent to extract almost 56.4% residual oil from the sample of POME.

Adsorption of residual oil increased as the dosage of synthetic rubber was increased until at 300 g dm^{-3} where 67.2% of residual oil was adsorbed. The optimum mixing rate was

showed at 100 rpm at mixing time of 3 hours. The best removal of residual oil was obtained at pH 9.

As shown in Figure 3, the GH membrane gave the lowest turbidity of 360 NTU at pH 4 and pressure of 10 bars. The GK, GM and GN gave turbidity value of 365, 465 and 471 respectively. Since pore size was comparable to the MWCO, the higher the MWCO, the greater the passage of water and suspended solids through the membrane and hence the higher observed values of turbidity.
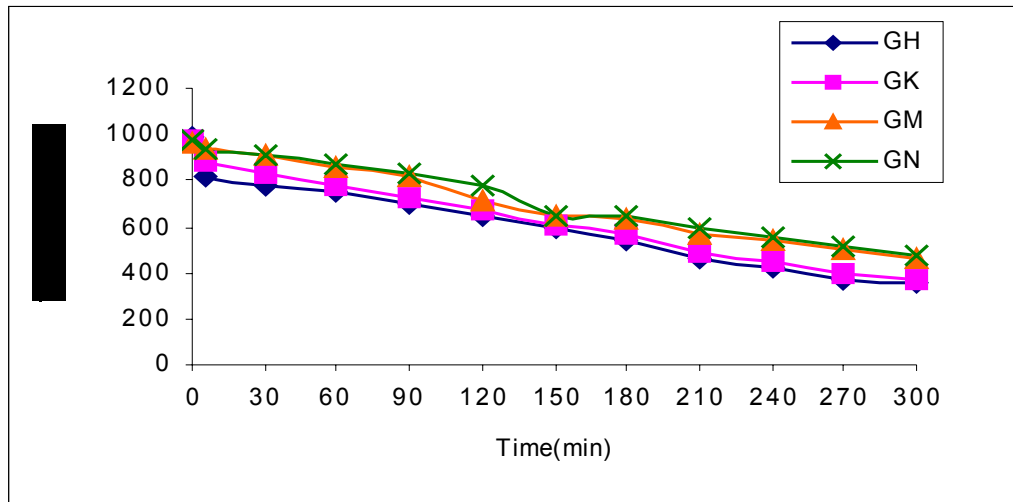
**FIGURE 3**: Turbidity Analysis Using Four Different Types of Membrane

The effect of pressure on the permeate flux for GH membrane are shown in Figure 4. Relationship between flux and pressure can be represented as below [8]:

$$J = \frac{\Delta P - \Delta \Pi}{R\mu}$$

where

J	=	flux, m/s
ΔP	=	pressure, Pa
$\Delta \Pi$	=	osmotic pressure, Pa
R	=	resistance, m^{-1}
μ	=	viscocity, Pa.s

Therefore, increasing the pressure would force more water to pass through the membrane which would result in the higher permeate flux recorded at 15 bars compared with 10 bars.
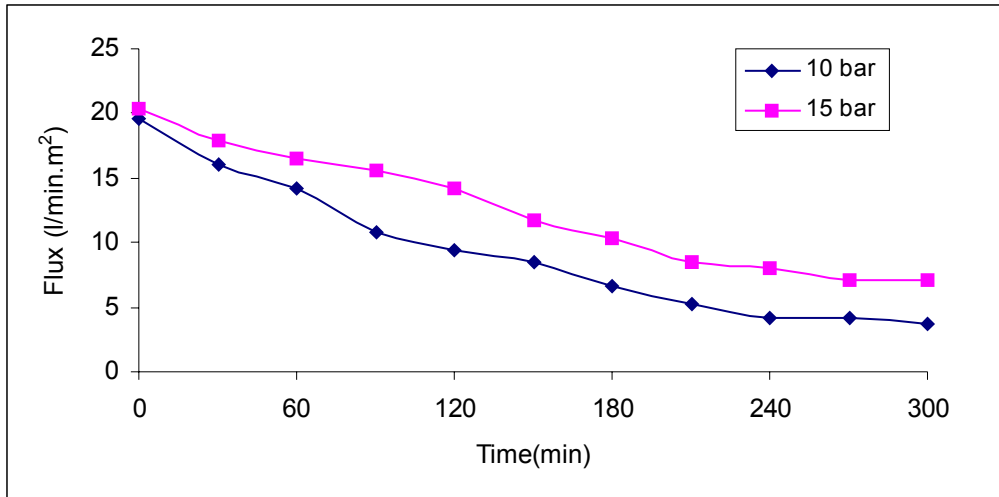
FIGURE 4: The Permeate Flux for GH Membrane at Different Pressure and pH 7.

Figure 5 shows the effect of pH on the permeate flux for GH membrane. The permeate flux after 5 hours of filtration at 15 bars and pH 9 is 6.59 l/min.m^2. At pH 7, the value increased to 7.06 l/min.m^2 and further increased to 7.53 l/min.m^2 at pH 4. Therefore, higher flux is noticeable within the acidic pH. This is because, at lower pH, effect of concentration polarization and membrane fouling can be minimized [9]. Besides, zeta potential of the feed samples will also reduce at low pH [10].
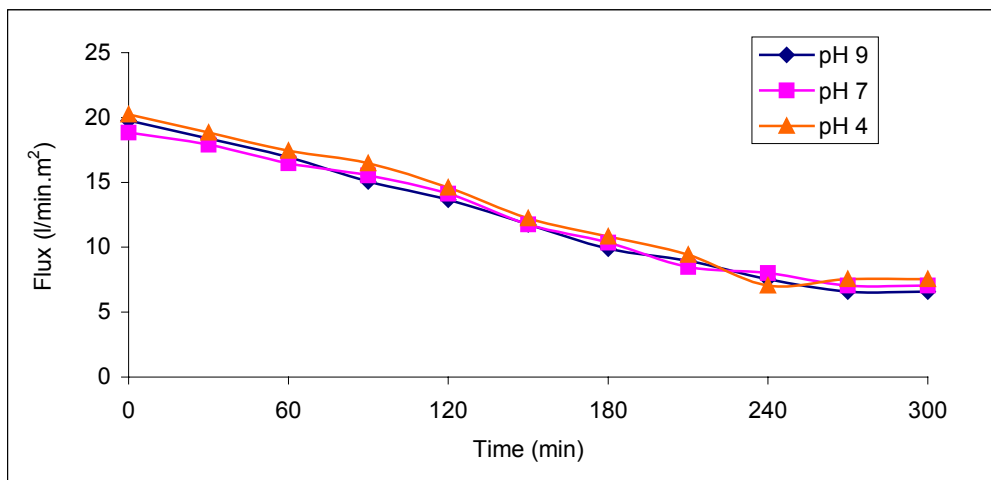
FIGURE 5: The Effect of pH on the Permeate Flux for GH Membrane at Pressure of 15 bars.

Table 2 simplifies the percentage removal of residual oil under the effect of pH and pressure from treated POME. Since the movement of oil across the membrane was determined by the flow of water, the higher the permeate flux, the greater the passage of oil and hence the higher observed values of flux. Higher permeate flux means that more residual oil exists in the sample. Therefore, percentage removal at 15 bars was low compared with pressure of 10 bars. As discussed earlier, acidic pH will decrease the concentration polarization and membrane fouling. Besides, at this pH, oil molecules tend to stay near the

membrane surface and hence increase their possibility to be pushed out through the membrane [11].

Table 2
Percentage removal of residual oil under the effect of pH and pressure.

Membrane Type	Percentage Removal (%)		
	pH / Pressure	10 bars	15 bars
CE (GH)	4	40.3	35.7
	7	41.9	36.4
	9	49.2	40.1

5. Conclusions

The membrane separation process was found to be effective for the removal of residual suspended solids and oil from pretreated POME samples. The optimum removal of suspended solids and residual oil was achieved with GH and CE (GH) membranes at a system pressure of 10 bars and pH 9. At these conditions, the turbidity value was reduced from 984 NTU to 360 NTU after 5 hours of filtration. Meanwhile, the oil concentration was decreased from 100 mg dm^{-3} to 51 mg dm^{-3} after 30 min of filtration.

The research findings show that membrane separation technology is a better treatment technology as compared with the standard methods for treatment of POME in terms of water recovery and its recycling in the mill.

Acknowledgements

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