

Reclamation from palm oil mill effluent using an integrated zero discharge membrane-based process

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This research emphasizes eloquently on membrane technology for treatment of palm oil mill effluent (POME) as it is the Malaysia's largest and most important agro based industry. Findings established significant quality improvement with an efficient recovery of water from palm oil mill via innovative membrane application. Conventional bio-methods, whilst adhering to the Department of Environment's (DOE) discharge regulations, produces brownish liquid which pales in comparison to the crystal clear water obtained through membrane treatment. The pre-treatment process consists of coagulation-flocculation using green environmental coagulant bases such as *Moringa oleifera* (MO) seeds. The ultrafiltration polyvinylidene difluoride (PVDF) and thin film composite (TFC) reverse osmosis were vital for the membrane processes. The system gave 99% suspended solids reduction in suspended solid and 78% of water present was successfully recovered. This technology guarantees water recovery with drinking water quality; meeting the US Environmental Protection Agency (USEPA) standard or could be recycled into the plant with sludge utilization for palm oil estates, thus enabling the concept of zero discharge to be executed in the industries. In addition, green and healthy antioxidants such as oil and beta-carotene can be recovered from POME further demonstrate. Silica gel showed better performance in separation of carotenes from oil at temperature 40°C using adsorption chromatography with 1154.55 ppm. The attractiveness of this technology, enabling the utilization of reuse of agricultural waste into potentially value added products.

Keywords: palm oil mill effluent (POME), membrane, wastewater, water recycling, carotenes.

INTRODUCTION

Palm oil has been produced highly year by year based on world demand. The ever exponential increasing in oil palm has made Malaysia second largest producer and exporter of palm oil and its products. However the escalate in production and processing of oil palm and its derivatives also comes with its inherent disadvantages, namely environmental pollution and its adverse effects on human and ecological system. Palm oil industry has been declared as one of industries that contribute greatly to wastewater problems in Malaysia. According to Ahmad et al., (2003) each tones of crude palm oil (CPO) produced more than 50% of water namely as palm oil mill effluent (POME)¹. This implies that increasing the production of palm oil will increase the volume of liquid POME waste.

POME consists of colloidal suspension of 95% of water, 0.6–0.7% oil and grease and 4–5% total solids including 2–4% suspended solids, originally a mixture from sterilizer, condensate, separator sludge and hydrocyclone wastewater^{2,3}. The discharge of fresh POME is hot at around 80–90°C, acidic (pH 4.5) and non-toxic. The organic content of POME, was measured by biochemical oxygen demand (BOD, 3 days, 30°C) averages about 25.000 mg/L with chemical oxygen demand (COD) of 50.000 mg/L and oil and grease content exceeded 4000 mg/L which is highly polluting³. According to the above characteristics, the discharge of untreated POME into the water streams definitely gave an impact to the environment especially aquatic life by oxygen depletion and suffocation.

Consequently, POME must be treated properly to meet the standard discharged limit stipulated by Environmental Quality Act (EQA) of 1974 and specific regulations for POME in 1977 (EQA 1974, 2005). It is mandatory for all

palm mills to treat wastewater on site to an acceptable level before they are permitted to be discharged into water sources. Conventional treatment such as ponding system is not enough to support the treatment because huge amount of POME is produced during processing. In addition, these treatments have several difficulties in complying with the discharged limit and retrieving oil through these treatments is not viable. Thus, palm oil industry needs to shift towards becoming a more environmental friendly industry as it has to be sustainable and competitive to increase its long-term profitability and sustainability.

To overcome waste discharge problem, an effective, reliable and economic separation process that has the potential of overcoming waste discharge problem is membrane separation technology. The process has received great attention in the last two decades. It has the prospect of replacing traditional separation process such as distillation, adsorption, extraction and, crystallization⁴. Integrated membrane based process was introduced in treating POME is a combination between pre-treatment and several types of membranes such as microfiltration (MF), Ultrafiltration (UF) and Reverse Osmosis (RO)⁵. It is employed for treating POME and able to reduce the process to less than two days compared to conventional treatment system (aerobic and anaerobic process) which requires the treatment period of 80 to 120 days.

Integrated membrane based process promises efficient water recycling and high quality water reclamation by producing high quality of water. In addition to beneficial prospects towards environmental pollution, the process also has potential economic benefits to the local communities. This recycling activity will contribute to the cost savings for the plant's budget as well as to lead towards the natural resources preservation. Through this

treatment the water discharge parameters will always comply with the standard discharge limit imposed by Department of Environment (DOE)⁶.

Up to date, chemical coagulants are widely used such as aluminium sulphates (alum), hydrate lime, aluminium hydroxide, ferric chloride and poly aluminium chloride (PAC) in the pre-treatment of wastewater⁷. The additional alum has been shown to be effective in pre-treatment. Sometimes, excess dosage of alum tends to produce sludge difficult to dispose to the environment because it contains aluminum. To avoid using chemical coagulant, green environmental friendly coagulant such as Moringa Oleifera (MO) are used for pre-treatment to achieve the ultimate goal of wastewater treatment towards zero discharge system. MO can be classified as cost effective, biodegradable, no physical hazard, metal free and non-toxic coagulants involved during the pre-treatment. In addition, natural coagulant could replace chemical coagulant to have much better performance.

POME itself has its own values to be recovered as it contains healthy antioxidants such as oil and β -carotene. Palm oil contains 300 times more carotenes than tomatoes and 15 times more retinol equivalents (vitamin A) than carrots⁸. Carotenes working as anti-cancer agents for certain types of cancer, reduce night blindness and also enhance the immunity together with protection against toxin, colds, flu and infections^{9, 10}. In addition, carotenoids have an industrial use in food products, cosmetics as vitamin supplements and health food products as well as feed additives for poultry, livestock, fish, and crustaceans¹¹. Besides that, carotenes also work in feed industries as colorant and antioxidant properties^{9, 12}. In fact, it is being used safely as an oral sunlight blocking agent for the prevention of sunburn and has been shown to be effective either alone or in combination with other carotenoids or antioxidant vitamins. Assumption can be made that the carotenes in the oil separated from the centrifuge sludge is similar to the commercial palm oil which is also similar to the CPO^{13, 14}. Since POME is abundant, the carotenes contained in POME can be used as a valuable source of vitamin A if reclaimed using adsorption chromatography. Besides that, oil, water and solids from the POME could be further treated to be converted into valuable products such as carotenes, drinking water and fertilizer.

This study serves triple purposes as shown in figure 1. Firstly the drinking water was recovered from integrated membrane process, secondly the wastewater was converted into valuable products such as carotenes, and lastly the agricultural waste was exploited to generate fertilizer, solving the environmental problem facing palm oil mill.

MATERIAL AND METHODS

Material

Dry Moringa Oleifera seeds were taken from trees around Nibong Tebal area in Penang state, Malaysia. Palm oil mill effluent (POME) was taken from United Palm Oil Mill, Nibong Tebal, Penang at a temperature range of 80–90°C. The study used *n*-hexane and Petroleum ether as the solvents. The solvents used were analytical grade. *N*-hexane, Petroleum ether and Ethanol were bought

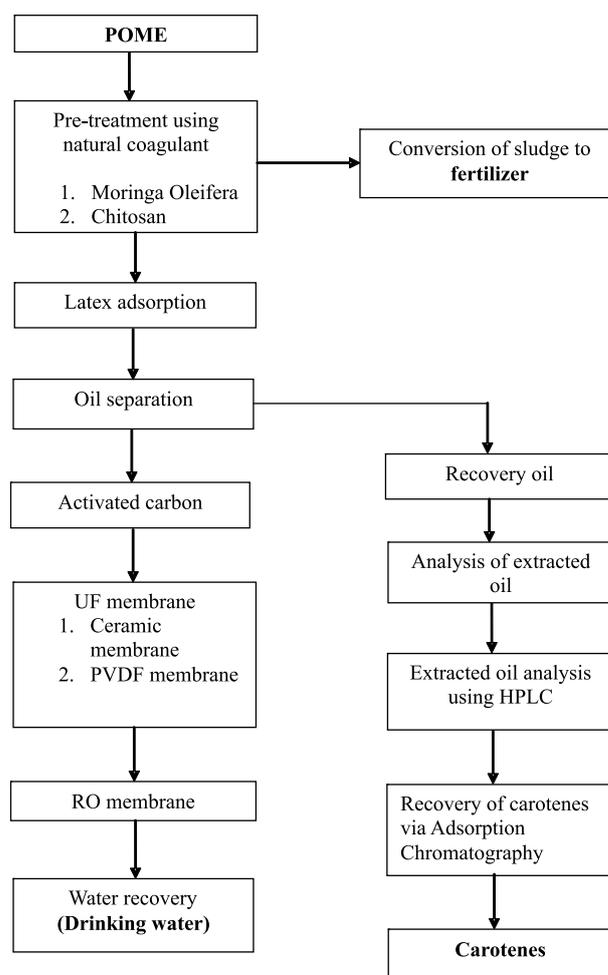


Figure 1. Flowchart process diagram

from Merck, Germany. β -Carotene standard solutions for calibration curve were purchased from Sigma-Aldrich and Merck. Silica gel bought from Sigma Aldrich Sdn. Bhd and florisil and aluminium oxide bought from Merck, Germany were used as adsorbents in this study.

Solvent extraction

200 mL of POME was mixed with *n*-hexane with 40 mL increments for each of the experiment until the ratio of *n*-hexane achieved the ratio of 1:1. The experiment was conducted at 28°C. Both mixtures were blended in a flocculator for 25 min at 500 rpm. Then it was transferred into the separating funnel and left to separate into two layers. The supernatant was poured into a conical flask and the solvent was separated using rotary evaporator model Buchi R-215, Switzerland. The flask was dried in an oven at 102°C for 15 minutes and left to cool in dessicator for 30 minutes. The weight was measured three times to get accurate results. The drying and cooling processes were repeated until the conical flask became constant. The measured weight was taken as extracted oil value.

Carotenes recovery

10 grams of adsorbent was packed and used in chromatography column at room temperature. The column was filled in with *n*-hexane until it achieved steady state. Then 2.5 g extracted oil from POME was heated to 40°C and loaded onto the column. The heated oil was allowed to come in contact with the adsorbent and

allowed to melt before being loading into the column. 100 ml of non-polar solvent, *n*-hexane was then poured into column, followed by 100 ml polar solvent, ethanol. Mass loading between oil and adsorbent ratio was 1:5. 25 ml of the sample was collected regularly using flask. Rotary evaporator was used to remove the solvent from the carotenes. 0.1 g of sample was weighed and directly diluted with 25 ml of *n*-hexane, then the samples were measured using spectrophotometer (model Spectroquant® Pharo 300, Merck, Germany) at 446 nm. The experiments were repeated using different temperatures; 30, 40 and 50°C and different types of adsorbents and solvents. The carotenes recovery in extracted oil was determined using equation:

$$R_q = \sqrt{\frac{1}{n} \sum_{i=1}^n y_i^2}$$

Where, W_{extract} is the weight of oil extract (eluted) from the experiment (mg), C_{extract} is the carotenes concentration in the extracted oil (ppm), W_{POME} is the weight of oil loaded onto the column and C_{POME} is the carotenes concentration (ppm) in the oil loaded onto the column.

Bench scale study

Jar test apparatus, benchscale coagulation studies were determined by 500 mL of POME was filled up in 600 mL beaker. The pH value was adjusted to the desired value by using either sulphuric acid (3M) or potassium hydroxide (5M) within the range of 4 to 9. MO as coagulants was used for pre-treatment study. MO were added in a varying range from 500 mg/l to 6000 mg/l. the resulting mixture was agitated at 150 rpm for 5 minutes and continued with slow speed at 30 rpm for 30 minutes. After mixing, the content of each beaker were then allowed to settle with settling time of 0 to 180 minutes. Several parameters were varied to identify the best combination as the guidelines for pilot plant study. The optimum values of the operating parameters obtained was pH 5 and the MO dosage is 6000 mg/L.

Pilot Plant experimental set-up

In pilot plant, the processes were divided into two parts, pre-treatment processes and membrane system. Pre-treatment consists of several processes such as coagulation-flocculation, latex treatment, oil separation treatment, and activated carbon treatment, while for membrane system the processes involved are ceramic ultrafiltration membrane, PVDF ultrafiltration membrane and reverse osmosis treatment. Transfer tank with diameter of 760 mm (D) x 500 mm (H) was filled up with 200 L raw POME using SEA-LAND pump. The raw POME was then pumped into pre-treatment tank via centrifugal transfer pump. A pre-determined amount of coagulant was added automatically using prominent-Alpha 0419 with capacity of 1–10 L/hr. The effluent was allowed to settle. Supernatant from pre-treatment was pumped into the latex treatment tank. Latex powder was manually added into the tank to absorb oil. The solution was mixed roughly, and transferred into oil separation tank. Manually addition of *n*-hexane aided solvent extraction in the oil separation tank. Envilab agitator using compressed air was introduced from the bottom of the tank for mixing. The solution allowed to

separate into two layers after thorough mixing, *n*-hexane is on the top layer and water at the bottom layer. Grunfos CRN-4-50 centrifugal pump was used to pump out water to the next treatment, carbon filter 10 μm , while *n*-hexane was collected for carotenes analysis. The water was then passed through the carbon filter with dimension of 250 mm (D) x 1300 mm (L) at a flowrate 4 L/min for adsorption process. After undergoing these series of pre-treatment processes, filtrate from carbon filter was drained into UF system. The ceramic ultra filtration membrane area was 0.36 m^2 , pore size was 0.005 μm and cross flow was 8–20 m/h, while operating conditions for PVDF ultrafiltration membrane area was 0.9 m^2 , pore size was 0.1 μm , and cross flow was in the range of 0.8–1.6 m/h. The Grunfos CRN-8-50 pump at the pressure of 2 bar with capacity of 150 L/min was used to transfer water into the cross flow batch concentration process mode, which means the permeate from UF membrane can be diverted into UF permeate tank while retentate flow was returned to the UF feed tank. UF permeate was later pumped into TFC RO system unit. The area for RO composite membrane was 0.9 m^2 and operating pressure was 0–64 bars. Trans-membrane pressure was maintained at 45 bars by regulating the pressure control valve.

Fertilizer preparation

The aqueous phase containing sludge from the pre-treatment was used to prepare the fertilizer. It mainly consists of water and suspended solids. The aqueous phase was filtered through sieve of 45 μm mesh size. The coarse particles or sludge that retained on the sieve was used in this experiment. The sludge was mixed with the empty fruit bunch (EFB) ash and was dried in the oven for 4 hours at 90°C. The sludge mixture contained 40% of EFB and 60% POME sludge. Nutrient analysis was then carried out.

RESULTS AND DISCUSSION

Carotenes recovery

Figure 2 shows the result obtained from the adsorption chromatography for separation of carotenes from extracted oil. The experiments studied the effect of temperatures, adsorbents, and solvents effect on the recovery of carotenes. The temperatures studied were varies between 30°C, 40°C and 50°C, while the adsorbents used were Silica gel, Florisil and Alumina oxide. *n*-hexane was selected to be used as a solvent to study the effect of temperatures and adsorbents. Based on preliminary study, *n*-hexane extracted more oil from POME sample than Petroleum Ether. Therefore, *n*-hexane was used as the initial eluting solvents. According to Loconto¹⁷ the polarity indexes for *n*-hexane is 0.1 whereas the polarity index for ethanol was 4.3. The larger the polarity index, the more polar the solvent. The sample which are very polar, strongly interacted with the column and tends to elute at later retention time. However, molecules that are more non polar tend to interact weakly with the normal phase support and elute quickly from the column at earlier retention time.

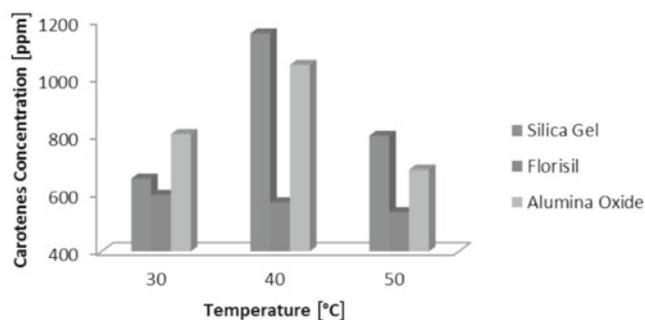


Figure 2. Effect of adsorbent at various temperatures using adsorption chromatography

The graph shows the effect of adsorbents at various temperatures using adsorption chromatography. The carotenes concentration obtained at 40°C is higher than carotenes concentrations at 30°C and 50°C for silica gel and alumina oxide. The highest carotenes concentration was 1154.55 ppm at the temperature 40°C using silica gel followed by alumina oxide and florisil with values of 1045.39 ppm and 570.81 ppm respectively. However at lower temperature (30°C) the extracted oil did not completely liquefy and hindered the carotenes elution. However, at temperature 50°C, the viscosity of oil was lower and thus it increased the rate of diffusion of the adsorbate to the adsorbent. Furthermore, the significant reduction on carotenes concentrations occurred at temperature 50°C and resulted in unsuccessful separation of carotenes into high concentrations.

Silica gel showed good performance among other adsorbents in the recovery of carotenes. Therefore, we

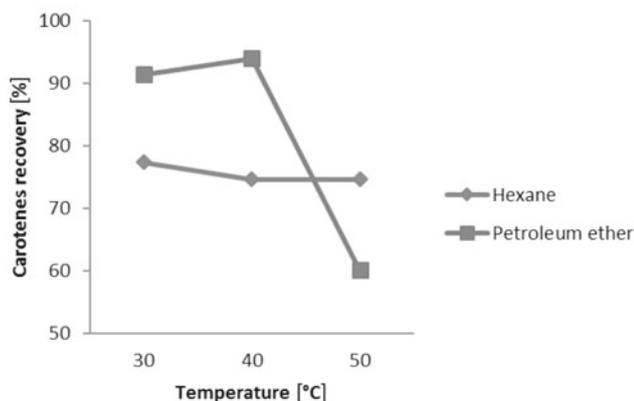


Figure 3. Effect of solvents on percentage recovery of carotenes at various temperatures

carried out further studies to identify the optimal solvent for recovery of carotenes. Two solvents were used; *n*-hexane and Petroleum ether (PE). Figure 3 shows the effect of solvents on percentage recovery of carotenes at various temperatures.

At 40 °C, PE exhibited highest percentage recovery at 94.03% than that of *n*-hexane at 74.63% respectively. The

carotenes recovery for PE was higher than *n*-hexane at temperatures 30°C and 40°C. However, carotenes recovery by PE dropped unexpectedly with sudden decrease to 60.13% at 50°C. This is because the boiling point for PE is 40–60°C, thus it might have evaporated at 50°C. At higher temperature, the oil viscosity was lower thus increased the rate of diffusion of the adsorbate to the adsorbent. Hence, reducing the average carotenes concentration and resulting ineffective separation of carotenes into high concentration. In addition, at this temperature, the vapour pressure of the solvent is good enough to overcome atmospheric pressure and to enhance the solvent to form bubbles inside the bulk of the solvent. This eventually would reduce the amount of solvent and reduce the diffusion rate. As a result, the solubility of carotenes recovery dropped in PE. On the whole, the carotenes recovery in *n*-hexane is more stable and maintained compared to PE.

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Pilot plant test

The main objective of the research is to recover clean water from POME that can be used as drinking water. Integrated membrane process comprises two processes involved; pre-treatment and membrane process. During the process, water sample was analyzed to assess the quality of treated POME discharged from every integrated process in the overall treatment system. Table 1 shows the analysis of sample after different operations.

The pre-treatment processes played a significant effect in reducing almost 99% of turbidity and suspended solids content from POME¹⁵. Conversely, natural coagulants removed almost 95% suspended solids in POME before it goes further to the other treatments. It can be seen that the value of suspended solids before enter the series of membrane treatment must be less than 50 mg/L respectively and it was achievable using a series of pre-treatment processes. Evidently, further reduction in suspended solids was successfully achieved after passing several series of membranes. However, from the results obtained almost 98% and 99% suspended solids were removed after passing through the PVDF membrane and ceramic membrane respectively. Thus, these particles would only deposit on the membrane surface and never penetrate into permeate side. The final treatment from RO membrane produced crystal clean water that is free from suspended solids.

Figure 4 presents the permeate flux over time for PVDF membrane. The membrane's separation process was operated using pre-treated POME for 24 hours

Table 1. Analysis of sample at different operation

Sampling points	Suspended solids [mg/L]	COD [mg/L]	Oil & grease [mg/L]
Raw POME	18.975	43.155	3.172
Coagulation	948.75	12.650	383
Filter press	45.54	12.359	30
Activated Carbon	24.72	9.321	3.89
Ceramic membrane	0.27	5.767	1.22
PVDF	0.00	258	0.00

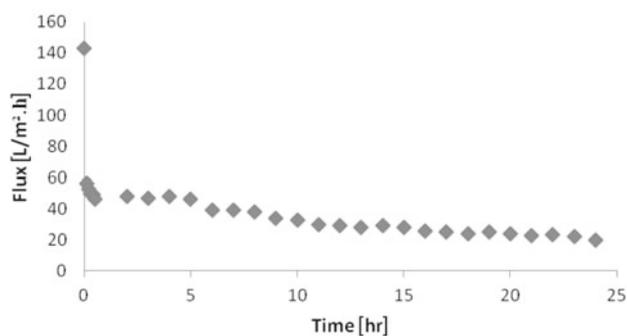


Figure 4. The permeate flux vs. time for PVDF

with transmembrane pressure of 2 bars. The graph was observed for first 20 minutes. It showed a significant reduction in permeate flux, in which the permeate flux declined from $143 \text{ L/m}^2 \cdot \text{hr}$ to $46 \text{ L/m}^2 \cdot \text{hr}$ during the first phase. Permeate flux reduced slowly in the second phase during the operating period within 20 minutes to 8 hours. The permeate flux obtained was $46 \text{ L/m}^2 \cdot \text{hr}$ to $34 \text{ L/m}^2 \cdot \text{hr}$ during this period. During the third phase, between 9 to 24 hours constant flux was attained ranging from $20 \text{ L/m}^2 \cdot \text{hr}$ to $30 \text{ L/m}^2 \cdot \text{hr}$, which indicated that the constant in reduction flux was achieved. We concluded that permeate flux decreased slowly as time increases. In addition, during steady state, cake layer formed on the membrane surface. This phenomenon was observed through similar study conducted by Liu et al., (2004)¹⁶.

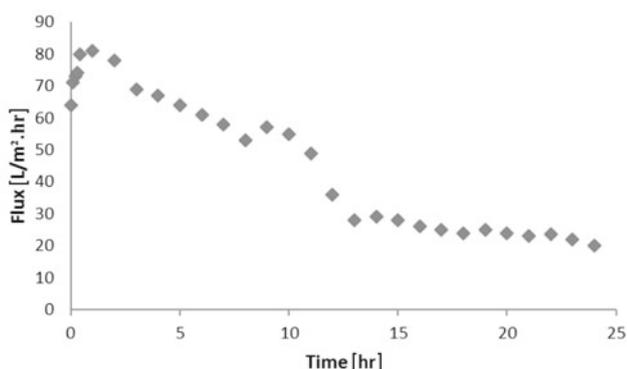


Figure 5. The permeate flux vs. time for RO membrane

RO membrane is best known with its high rejection of suspended solids, bacteria, viruses, multivalent ions and monovalent ions and is used widely in industrial processes and production of potable water. It has the possibility to produce clean water and drinking water. RO is the last process in integrated membrane process, in which permeate from UF became the feeder for RO membrane. Figure 5 showed the permeate flux over time for RO membrane. RO membrane is efficient in reclaiming water for safe drinking. The RO unit was operated at the inlet volumetric flowrate of $15 \text{ m}^3/\text{hr}$ and the average transmembrane pressure of 46.5 bars for 24 hours. The process involves three subdivisions in

Figure 5. Increase in permeate flux was observed for the 1 hour operating time with flux value increasing from $64 \text{ L/m}^2 \cdot \text{hr}$ to $81 \text{ L/m}^2 \cdot \text{hr}$.

There are a few reasons behind this phenomenon. One is due to increase in temperature ($15\text{--}33^\circ\text{C}$) with time during the operation. The temperature raise during the experimental run might be due to high resistance and friction that occurred during the membrane operation. This was difficult to control especially at high transmembrane pressure. On the second phase, flux reduces from 78 to $49 \text{ L/m}^2 \cdot \text{hr}$ could be seen from 2 to 11 hour of operating time. During this time, the temperature was almost constant at $33\text{--}35^\circ\text{C}$. The gradual reduction in permeate flux was ascribed to formation of fouling layer at the membrane surface. The third phase displayed almost constant flux attained from 12–24 hour of operating time with average flux value of $44.5 \text{ L/m}^2 \cdot \text{hr}$. During the steady state of flux, fouling layer formed on the membrane surface. Through RO process, RO permeates amounted to 78 L/hr water was generated as the final discharge effluent.

Table 2 shows the comparison with the quality of boiler water, government water and treated river water taken from the palm oil mill operated at Nibong Tebal. The quality of the reclaimed water from the integrated membrane process was compared with the quality of boiler water, government water, and treated river water taken from palm oil mill operated at Nibong Tebal. From the results obtained, the treated water from integrated membrane was as good as government water quality which indicated that this water can easily be utilized for the plant operation. The integrated membrane system promotes a zero discharged system to the palm mill industries. As an overall view, the system has successfully reduced operation time to less than 2 days compared with conventional treatment which takes around 80–120 days.

Conversion of sludge into fertilizer

After pre-treatment process, POME sludge was collected after using sieve. Table 3 showed the characteristics of POME sludge and other wastes. When the POME sludge was dried, the reduction in moisture content increased the nutrients percentages. The dried POME sludge contained an appreciable amount of metals such as potassium, calcium, magnesium and iron which are the essential nutrient elements for plant growth¹⁷. Ma et al. (1996) claimed that the solid concentrate is a good feed material for fertilizer manufacturing, hence suitability of the dried POME sludge prepared in this study is also suitable to be used as fertilizer. The dried POME sludge showed notable levels of total organic matter, total nitrogen, potassium, magnesium and iron compared to those found by Parades et al. (1999) in olive mill wastewater (OMW) sludge for agricultural purposes and also reported by Guerrero et al. (1995)

Table 2. Characteristic of different sources of water

Parameter	Membrane treatment water	Boiler water	Government water	Treated river water
Zinc [ppm]	4.10	22.50	3.70	8.70
Iron [ppm]	0.28	2.86	0.22	1.17
Calcium [ppm]	1.77	1.88	1.79	1.76
Suspended solids concentration [mg/L]	0.00	106.77	2.09	14.07
BOD [mg/L]	73.00	236.00	29.00	68.00
pH	6.63	10.73	7.00	6.47

Table 3. Characteristics of POME sludge and other wastes

Parameter	POME sludge [wet]	Calculated result for dried POME sludge [%]	Dried POME sludge ^c [%]	OMW sludge ^d [%]	Peel orange wastes ^e [%]
Total organic matter	6.5	88.09	90.42	71.06	84.30
Total nitrogen	0.2	2.71	3.16	1.74	1.0
Phosphorus	0.04	0.54	<0.5	0.14	0.2
Potassium	0.2	2.71	5.39	1.41	1.3
Calcium	308 ^a	0.42	0.51	2.87	0.8
Magnesium	864.6 ^a	1.17	0.58	0.36	0.1
Iron	186.9 ^a	0.25	0.21	0.45	40.0 ^b
Boron	46.3 ^a	0.06	<0.21	NA	NA
Moisture content	93.4	10.00	9.94	NA	NA
Total organic carbon	3.8	51.5	52.6	47.90	48.90

^a Unit in mg/L,^c Ma et al., 1996,^b Unit in ppm,^d Paredes et al., 1999,

NA: not available,

^e Guerrero et al., 1995.

in peel orange wastes for production of lettuce. The application of peel orange wastes increased the quantities of organic matter, nitrogen, phosphorus and potassium present in the soil. Therefore, POME sludge could be used as fertilizers in agricultural soils for improvement of soil fertility. Dried POME sludge showed the highest organic matter value which was probably due to lignocellulosic character or plant residues in various stages of processing¹⁸. Adding the dried POME sludge to the soil could increase the total nitrogen content, organic matter content and moisture retention, thus crop yields could be increased¹⁹. On the whole, the dried POME sludge obtained in this study could be used as fertilizer in plantation.

CONCLUSIONS

Adsorption chromatography was shown to be an effective for recovering high carotene contents from extracted oil. Silica gel showed superior performance in separation of carotenes resulting in the highest carotenes concentration and high carotenes recovery percentage. The integrated membrane used in the treatment process for POME resulted in a good quality of water which can be recycled back to palm oil mill. The advantages and value added offered by this new approach might lead to cost saving and alternative treatment for this industry. 78% of water with turbidity value of 20 NTU present in POME was successfully recovered as high quality water through the integrated system. The sludge generated from coagulation treatment is also suitable to be converted into fertilizer with further treatment to the sludge. POME sludge was possible to be used as fertilizer due to its high organic matter content, notably levels of nitrogen, potassium, calcium and magnesium and suitable C:N ratio.

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