Artificial Cells, Blood Substitutes, and Biotechnology

ISSN: 1073-1199 (Print) 1532-4184 (Online) Journal homepage: www.tandfonline.com/journals/ianb19

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To cite this article: K. K. Ong, A. Fakhru'l-Razi, B. S. Baharin & M. A. Hassan (1999) Degumming of Crude Palm Oil by Membrane Filtration, Artificial Cells, Blood Substitutes, and Biotechnology, 27:5-6, 381-385, DOI: 10.3109/10731199909117707

To link to this article: https://doi.org/10.3109/10731199909117707

Published online: 11 Jul 2009.

ART. CELLS, BLOOD SUBS., AND IMMOB. BIOTECH., 27(5&6), 381-385 (1999)

DEGUMMING OF CRUDE PALM OIL BY MEMBRANE FILTRATION

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ABSTRACT

The application of membrane separation in palm oil refining process has potential for energy and cost savings. The conventional refining of crude palm oil results in loss of oil and a contaminated effluent. Degumming of crude palm oil by membrane technology is conducted in this study. The objective of this research is to study the feasibility of membrane filtration for the removal of phospholipids in the degumming of crude palm oil, including analyses of phosphorus content, carotene content, free fatty acids (as palmitic acid), colour and volatile matter. A PCI membrane module was used which was equipped with polyethersulfone membranes having a molecular weight cut off of 9,000 (type ES209). In this study, phosphorus content was the most important parameter monitored. The membrane effectively removed phospholipids resulting in a permeate with a phosphorus content of less than 0.3 ppm. The percentage removal of phosphorus was 96.4% and was considered as a good removal. Lovibond colour was reduced from 27R 50Y to 20R 30Y. The percentage removal of carotene was 15.8%. The removal of colour was considered good but the removal of carotene was considered insignificant by the membrane. Free fatty acids and volatile matter were not removed. Typical of membrane operations, the permeate flux decreased with time and must be improved in order to be adopted on an industrial scale. Membrane technology was found to have good potential in crude palm oil degumming. However, an appropriate method has to be developed to clean the membranes for reuse.

Keywords
Ultrafiltration, degumming, membrane filtration, phosphorus, phospholipids, crude palm oil.

INTRODUCTION

There are two routes to refine crude palm oil; chemical or physical refining (Thiagarajan, 1992). Chemical refining causes oil losses and severe treatments with alkali solution led to chemical damage of the oil. Furthermore, chemical refining also produces a large volume of contaminated wastewater (Rusnani and Affandi, 1995). Hence, physical refining is a more common process in Malaysia (Thiagarajan, 1992). One of the refining processing steps is degumming which is used to remove phospholipids and trace metals from crude palm oil. Normally, phosphoric acid is used as a degumming agent (Rusnani and Affandi, 1995).

According to the US Department of Energy, energy savings of 7.2 - 35.3 trillion Btu/year could be achieved in the US by full implementation of membrane degumming process. In two years, 14.48 million gallons/year of water and 27,670 tonnes/year of solid water will be eliminated with 25% market penetration (Koseoglu, 1996). The use of a membrane technique could reduce oil losses of crude oil (soybean, cottonseed and corn) by 60% due to saponification of neutral oils during chemical refining (Koseoglu and Engelgau, 1990).

The new membrane degumming method is almost a single step operation. This process not only removes all the phospholipids, but also removes the major colour pigments and some of the free fatty acids. Thus bleaching requirements are reduced due to entrapment of some colour pigments by the membrane separation process (Koseoglu, 1996).

This study reports the feasibility of using membrane filtration in crude palm oil degumming particularly phospholipids removal as an alternative to the conventional method.

EXPERIMENTAL PROCEDURES

Palm oil and chemicals

Crude palm oil (CPO) was obtained from Jomalina Sdn Bhd (Teluk Panglima Garang, Selangor, Malaysia). All solvents and chemicals used were of analytical grade.

Membrane and membrane module

Polyethersulfone membranes were used and membrane module consists of six single tube testers (ROP 1557) and polythersulfone membranes were supplied by PCI Membrane System (UK), in line with a positive displacement pump (Model 1051) and pressure gauge. The membranes have a specified molecular weight cut-off (MWCO) of 9,000 (ES209) and total area of 0.271 m^2. The internal diameter and length of the membrane were 12 mm and 120 cm respectively. The recommended maximum pressure of the membrane is 30 bar and maximum operating temperature is 80°C. The recommended pH range of the membrane is 1.5 -12.

Experiment and Analysis

The ultrafiltration experiment was performed on single tube tester (ROP 1557). About 17 kg of crude palm oil was melted at 60°C to homogenise the oil. A few hundred millilitres of the sample was collected. The remaining oil was used as feed. Initially, the membrane process pressure was increased to 26 bar by adjusting the valve. Permeate flux was measured for three hours with intervals of half an hour. The retentate was recycled to the feed tank. After the first hour, a few hundred millilitres of retentate was collected and another 17 kg of crude palm oil (feed) was changed to ensure as minimum as possible the moisture content in the membrane. A few hundred millilitres of crude palm oil was collected for analysis. Another retentate was collected at the third hour after the first hour collection. The temperature was controlled at 63 ± 2°C. Figure 1 shows the schematic flow diagram of membrane filtration. Finally the sample, retentate and permeate were analysed for the phosphorus content, colour, free fatty acid, carotene content and volatile matter based on PORIM Test Methods (1995). Water flux was also measured at 26 bar.

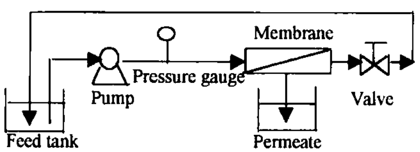

FIG. 1. Schematic flow diagram of membrane filtration

Scanning Electron Microscope (SEM)

The clean membrane, used membrane before cleaning and used membrane after cleaning were mounted onto the aluminium specimen stub by double sided tape separately. The specimens were then coated with gold using a Polaron E5100 SEM Coating Unit (UK) at the plasma current of 18 mA for 2 minutes. The specimens were top viewed by JEOL JSM-6400 (Japan) which was operated at 15kV with a working distance of 16 mm.

RESULTS AND DISCUSSION

Permeate Flux

The water flux of membrane determined at a pressure of 26 bar, was 607.4 L/m^2.h. According to Fane and Radovich (1990), the typical fluxes are 10-200 L/m^2.h for operating pressures ranging from 100 to 500kPa for membranes with pore sizes of 1-20 nm in ultrafiltration process. Thus, the water flux obtained was higher than the typical fluxes because the pore size of the membrane used was larger and the operating pressure used was higher. In this study, permeate flux was measured every half an hour within three hours at 26 bar. Permeate flux variation with time is displayed in Figure 2.

Obviously, the evolution of the permeate flux follows the general law of membrane fouling because the flow rate of permeate decreased with time. Fouling phenomenon was also observed by Prádanos *et al.* (1996) in the study of mechanism of protein fouling. In the study, Prádanos *et al.* have found that the fouling process started as a pore blocking phenomenon. Possibly this could be attributed to a blockage of the pore size by particle diameter similar to or below the protein size. Another possible reason for this occurrence was formation of concentration polarisation on the membrane surface. This phenomenon was shown by the deposition of oil globules on the membrane surface. By increasing crossflow velocity, the flux can be improved because higher shear rates remove components deposited on membrane surfaces (Fukumoto *et al.*, 1998).

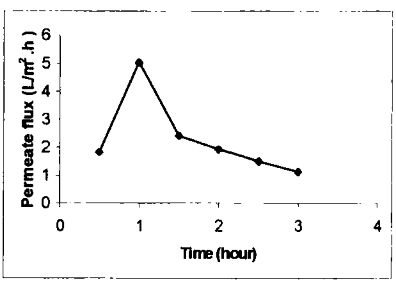

FIG. 2. Permeate flux versus time. Operating pressure, 26 bar. Polyethersulfone, 9,000 MWCO

Scanning Electron Microscope (SEM)

Figure 3 shows the clean polyethersulfone membrane with a magnification of 9,000. Figure 4 shows the used polyethersufone membrane before cleaning. Figure 5 shows the used polyethersulfone membrane after cleaning with tap water, followed by 0.1 N NaOH, 0.1 N H_2SO_4 and tap water. Oil globules were observed on the membrane surface in Figure 4 and Figure 5. By comparing these two plates, it was found that less oil was observed in Figure 5 but oil deposits continue to dominate the membrane surface. Hence, a more effective cleaning agent should be used in future studies.

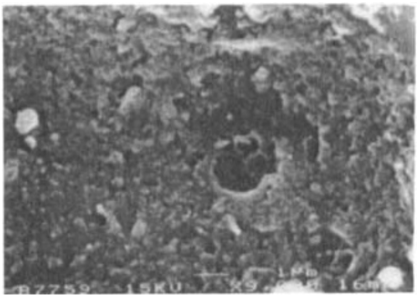

FIG. 3. Top view of SEM micrograph of the 9,000 MWCO clean membrane (magnification: 9,000X).

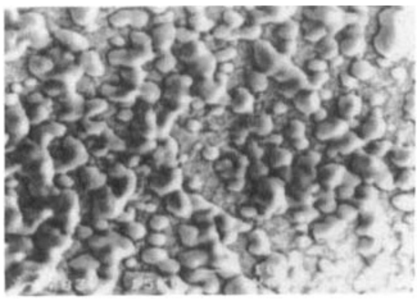

FIG. 4. Top view of SEM micrograph of the fouled 9,000 MWCO membrane before cleaning process (magnification: 800X).

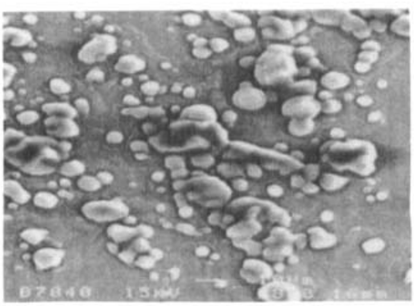

FIG. 5. Top view of SEM micrograph of the fouled 9,000 MWCO membrane after cleaning process (magnification: 800 X)

Phosphorus Content

The performance of the membrane process was expressed in terms of permeate flux, percent rejection of phospholipids, and phospholipids content in permeates (Subramanian and Nakajima, 1997).

Table 1 shows the phosphorus content of crude palm oil before membrane degumming and after degumming by membrane filtration (MWCO of 9,000) after three hours. The percentage removal of phosphorus was 96.4% (i.e. 0.3 ppm in permeate) in this study. For a good quality refined palm oil specification, the maximum value of phosphorus content is 4 ppm (Goh, 1993). Hence, a better quality palm oil in terms of its phosphorus content was produced. This process approaches waste minimisation concept because all the streams within the membrane process were recycled and only little is discharged as effluent.

The average molecular weight of phospholipids is around 700 Dalton (Subramanian and Nakajima, 1997). However, the membrane used which had a MWCO of 9,000 rejected phospholipids. According to (Subramanian and Nakajima, 1997), this is probably the result of the formation of reverse micelles because phospholipids have both hydrophilic and hydrophobic groups. The hydrophilic polar heads are inward in the reverse micelles and interact with other polar compounds. Goh *et al.* (1985) also reported that phospholipids cause reverse micelle, vessicle or emulsion droplet formation. Hence the larger size of complex formed can easily be separated by membrane (Subramanian and Nakajima, 1997). The percentage removal of phosphorus achieved was 96.4% and hence, use of polyethersulfone membrane seems to be a potential alternative to the conventional degumming method.

TABLE 1
Phosphorus Content

	Average phosphorus Content (ppm)	% Removal of average phosphorus content (%)
Crude palm oil (Feed)	8.3	-
Retentate	8.7	-
Permeate	0.3	96.4

Colour

Table 2 shows Lovibond colour of crude palm oil before and after membrane degumming. The percentage removal of R was 25.9%. The colour of Y was removed more than R, i.e. 40%. Based on the standard specification of PORAM (Palm Oil Refiners' Association of Malaysia), maximum R value for neutralised bleached palm oil is 20 (Thiagarajan, 1992). Therefore the efficiency of colour removal was considered good because the permeate has fulfilled the standard specification. Hence, the membrane filtration is suitable for pretreatment of crude palm oil in that colour was removed according to the standard specification. The colour reduction was due to the reduction of β-carotene content after membrane degumming because β-carotene is responsible for the red colour of the unrefined palm oil (Idris, 1995).

TABLE 2
Lovibond Colour

	Average of Lovibond colour	% Removal of average R value (%)	% Removal of average Y value (%)
Crude palm oil (Feed)	27R 50Y	-	-
Retentate	28R 50Y	-	-
Permeate	20R 30Y	25.9	40.0

Carotene Content

Carotene content before and after membrane filtration is given in Table 3. Carotene removal was only 15.8%. *The removal efficiency of carotene was considered not* significant and should be improved further.

TABLE 3
Carotene Content

	Average of carotene content as β-carotene (ppm)	% Removal of average carotene content as β-carotene (%)
Crude palm oil (Feed)	596	-
Retentate	629	-
Permeate	502	15.8

Free Fatty Acid Content

The percentage of free fatty acids as palmitic acid before and after membrane degumming is shown in Table 4. This membrane process could not remove free fatty acids. Hence smaller MWCO of membrane should be used for crude palm oil deacidification.

TABLE 4
Percentage of Free Fatty Acid as Palmitic Acid

	Average of % free fatty acid as palmitic acid (%)	% Removal of free fatty acid as palmitic acid (%)
Crude palm oil (Feed)	2.62	-
Retentate	2.68	-
Permeate	3.18	0

Volatile Matter

The volatile matter content of crude palm oil before and after membrane degumming is given in Table 5. The volatile matter could not be removed by this membrane process. The results showed that the volatile matter in the permeate and retentate were higher than the feed where the volatile matter content were 0.096%, 0.099% and 0.0695% respectively. The moisture content may be contributed by moisture presence in the membrane, which was incompletely removed by the first batch of crude palm oil used.

TABLE 5
Volatile Matter

	Average of volatile matter (%)	% Removal of average volatile matter (%)
Crude palm oil (Feed)	0.0695	-
Retentate	0.099	-
Permeate	0.096	0

CONCLUSIONS

The percentage of phosphorus removal was 96.4%. Hence, there appears to be good potential for the use of membrane technology in degumming of crude palm oil. A better quality palm oil in terms of its phosphorus content was produced.

Although the membrane effectively removed the phosphorus, the removal efficiency of carotene and *colour was less than the conventional method*. The removal of carotene achieved was 15.8% and colour was reduced from 27R 50Y to 20R 30Y. Thus, the colour removal was considered good but the carotene removal was considered insignificant. Free fatty acid and volatile matter were not removed by the membrane filtration.

All the streams within the membrane process were recycled, and little was discharged as effluent. Thus, an advantage of this concept is that it provides waste minimisation.

ACKNOWLEDGEMENTS

Appreciation is extended to the laboratory assistants from the Chemical and Environmental Engineering and the Biotechnology Departments, UPM (Universiti Putra Malaysia), and PORIM (Palm Oil Research Institute of Malaysia) for helping to carry out this project. This study was carried out with financial support from IRPA (Intensification for Research in Priority Area) Grant 03-02-04-0062.

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