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CONCEPTUAL ALTERNATIVE MILLING FOR 3-MCPD MITIGATION

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ABSTRACT

Most Malaysian refineries employ physical refining to remove objectionable co-constituents in crude palm oil (CPO) with minimal damage and loss to glycerides and desirable components. Unfortunately, carcinogenic 3-monochloropropane-1,2-diol (3-MCPD) esters can form during refining due to high temperatures, the presence of chlorine and an acidic environment. The formation of these esters could theoretically be prevented if these factors were eliminated. A conceptual alternative milling process flow is presented, where degummed, neutralised CPO could be refined. The novelty of the proposed process lies in preventing 3-MCPD formation during conventional refining by eliminating an acid medium. Preliminary results indicated that the optimal degumming conditions for diluted crude oil were 80°C with 0.05% v/v 85% phosphoric acid (H_3PO_4) and a 30-min reaction, reducing phosphorus content from 15.76 to 7.60 ppm. Membrane filtration resulted in 30% free fatty acids (FFA) rejection, decreasing from 4.72% to 3.26%. The contents of 3-MCPD and glycidyl ester in the refined, bleached and deodorised (RBD) palm oil produced were 0.5 and 11.0 mg kg^{-1}, respectively, consistent with conventional products.

Keywords: acid degumming, membrane filtration, scopa bleaching.

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INTRODUCTION

Crude palm oil (CPO) inevitably contains varying amounts of non-glyceridic co-constituents such as free fatty acids (FFA), partial glycerides, phosphatides, sterols, tocopherols, hydrocarbons, pigments, carotenes, sterol glucosides, protein fragments, as well as resinous and mucilaginous materials, traces of pesticides and heavy metals. Even palm oil mills producing CPO that complies with the Palm Oil Refiners Association of Malaysia (PORAM) specifications require further refining to produce high-quality edible oil.

Malaysian CPO has low phosphatide content (15–50 ppm phosphorus, [P]) and high FFA (up to 5%), suitable for physical refining, which offers lower costs, increased production capacity, higher fatty acids recovery and less wastewater generation.

Although various physical refining technologies for vegetable oils are readily available, phospholipids remain a challenge in oil refining, as the quality needs to be compromised, which causes oil waste in production. Phosphatides, known as gums, should be removed in the early refining stage for better oil stability, uncomplicated transportation, improved oil quality and reduced oil loss.

Acid degumming, also known as chemical degumming, is the common degumming practice in present refineries that disperses approximately 0.08%–0.10% of 85.00% phosphoric acid (H_3PO_4) at 80°C–120°C to dissociate non-hydratable phosphatides into phosphatidic acid and calcium or magnesium biphosphate salt in a degumming vessel, equipped with a dynamic mixer for about 15 min. Both components are then absorbed by bleaching earth, except for excess H_3PO_4, which could cause hydrolysis or mono- and di-glyceride phosphorylation during the subsequent physical refining process.

Bleaching earth is a natural clay consisting of naturally occurring complex aluminium silicate minerals such as attapulgite, sepiolite or bentonite.

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Other metals such as iron, magnesium, calcium, sodium and potassium are present in varying degrees as impurities. Long-period natural weathering has rendered the original mineral partly porous with mild adsorption power, which could be dramatically increased by mineral acid leaching, known as acid activation. However, acid-activated bleaching earth would cause undesirable triglyceride-splitting reactions, thus increasing the formation of 3-monochloropropane-1,2-diol (3-MCPD) esters. Spent bleaching earth could be regenerated to 90%–95% activity of the original bleaching earth.

The amount of bleaching earth required depended on the quantity of CPO, typically 0.5%–2.0%. The bleaching process is carried out under vacuum (100 mm Hg) for about 45 min at 80°C–120°C, which should be high enough to maintain low oil viscosity to improve diffusion and mass transfer rates. Among the colour pigments, red is the most difficult to bleach by bleaching earth and thus is usually mentioned in the specification as a 5.25 inch Lovibond cell measurement.

Deodorisation is a multi-step vacuum steam distillation process comprising deaeration, multistage heating, deacidification and oil cooling to enhance the flavour and oxidative stability of palm oil by nearly completely removing FFA and other volatile materials, partially removing tocopherols and destroying thermal peroxides. Process variables that determine the performance of deodorisers are pressure, temperature, stripping medium and retention time. Continuous efforts have been made to enhance the deodorisation technology (Foo et al., 2022), and vendors have introduced several designs of modern industrial deodorisers that prioritise efficiency, yield and safety.

The batch and cross-flow deodorisation processes should be carried out at an absolute pressure as low as possible and cannot completely remove volatile components (Dijkstra, 2007). Refined, bleached and deodorised (RBD) palm oil consists of solid-phase stearin and liquid-phase olein, which need to be further separated through a thermo-mechanical fractionation process classified as dry fractionation, solvent fractionation and surfactant fractionation to enhance the appearance, acceptability and marketability of the products. Palm stearin, palm olein and palm middle fraction would be produced in a fractionation plant. The selection and application of fractionation methods depended on the required physical and chemical properties of stearin and olein (Kellens et al., 2007).

Refined Palm Oil Contaminants

Physically refined palm oil contains various undesirable minor components due to chemical reactions during the refining process. The 3-MCPD esters have been classified as possibly carcinogenic by the International Agency for Research on Cancer (IARC) and later were concluded to cause kidney failure by the European Food Safety Authority (EFSA) in May 2016.

The European Union (EU) has shown concern for the fatty acid esters of glycidol and has enforced Regulation (EU) No. 2018/290 amending Regulation (EC) No. 1881/2006 and 835/2011 published on 27 February 2018 (Phytocontrol Group, 2018). Limits for various contaminants in refined oil have been specified. Polycyclic aromatic hydrocarbons (PAH) consist of benzo(α)pyrene, benzo(α)anthracene, benzo(β)fluoranthene and chrysene. The limit for dioxins and polychlorinated biphenyls is based on the toxic equivalent of the World Health Organization (WHO). Thus, palm oil refineries must adopt mitigation to reduce the ester level in refined palm oil.

The MCPD esters formation pathway study showed that the major formation factors for the 3-MCPD are high temperature (above 140°C), the presence of chlorine and acidic medium (Rushworth, 2020). An excessive amount of H_3PO_4, a strong acid used in conventional CPO degumming, could not be eliminated by bleaching earth, thus forming an acidic medium that catalyses the 3-MCPD formation in the presence of chloride at high deodorisation temperature. The chloride in the oil originated from various sources, mainly fertiliser muriate of potash (KCl) and groundwater. Ester formation during the refining process could be theoretically prevented if any of the mentioned factors were irrelevant.

Various efforts have been made to mitigate the formation of 3-MCPD (Elisabeth, 2023). *Figure 1* shows the correlation between the content of 3-MCPD in refined palm oil and the total chlorine content in CPO (Chew & Saparin, 2022). Fresh fruit bunches (FFB) washing before extraction, dilution water control and CPO dechlorination agent sodium metabisulfite application reduce inorganic water-soluble chlorinated compounds in CPO. Other recommendations are CPO washing before refining, using low chloride high pH bleaching earth, combining acid degumming with water degumming, reducing deodorisation temperature to 230°C and using chemical refining (Ibrahim et al., 2016). However, the proposed mitigation strategy involves some capital expenditure (CAPEX) investment. Furthermore, a substantial amount of wastewater will be generated, which may overload the existing effluent treatment plant in the refinery.

CPO degumming using enzyme phospholipases (NS 40138) with a minimum amount of weak chelating acid, such as citric acid, could also mitigate the formation of 3-MCPD (Rushworth, 2020). Thus, eliminating acidic deodorisation theoretically would mitigate the 3-MCPD issue.

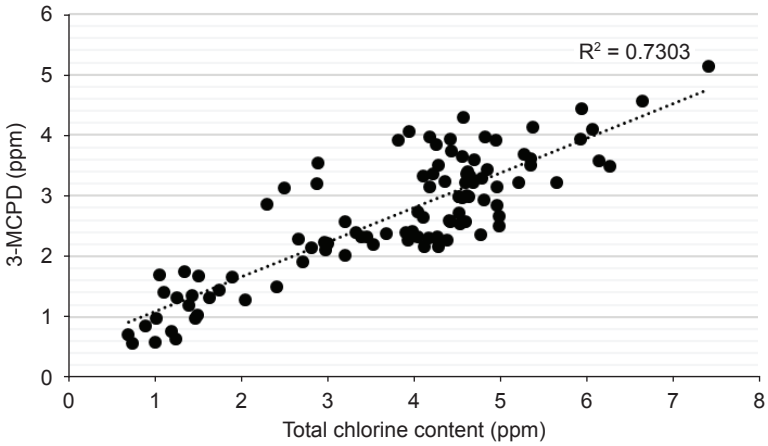

Figure 1. Refined palm oil 3-MCPD content against the CPO total chlorine content.

Membrane for Fatty Acids Separation

The membrane is an intervening phase separating two phases and acts as an active or passive mass transfer barrier between adjacent phases (Gekas, 1986). Membrane filtration for fatty acid separation from edible oil is a viable classical process (Keurentjes, 1991). Three configurations could be envisaged for membrane separation. Direct filtration with molecular size differences retention is the most common approach. Extraction mode is another feasible concept; however, the extraction medium for separating fatty acids in previous studies was mostly alcohol, which also led to oil losses (Shah & Venkatesan, 1989). Polyamide membranes showed better selectivity for fatty acid separation than cellulose acetate and polysulfone membranes (Kumar & Bhowmick, 1996). Dispersion mode involves hydrophilic and hydrophobic membranes in series based on the preferential wetting phase, in which the non-membrane wetting phase would be retained.

Molecular Size Estimation

Assume the molecule is a spherical shape, and volume is the amount of space any mass takes up in three-dimensional space. Using algebra and the density formula discovered by Archimedes of Syracuse, a specific molecular size could be estimated. The total amount of palmitic acid, oleic acid, stearic acid and linoleic acid is 97.8% of the total fatty acids in CPO. Since the molecular size of triglyceride is bigger than the molecular size of FFA, ranging from 9.846 to 10.003 nm, the FFA in CPO theoretically could be separated via membrane filtration.

This study explored the potential of a proposed palm oil milling enhancement that prevents 3-MCPD formation during conventional refining through the elimination of an acid medium as a feasible alternative for CPO refining to produce RBD palm oil that complies with the EU regulation requirements on the 3-MCPD ester content.

MATERIALS AND METHODS

Materials

Diluted crude oil samples were courtesy of a palm oil mill in Selangor, Malaysia. All technical-grade chemicals were locally purchased.

The organic solvent nanofiltration (OSN) membranes known as PuraMem® Flux (MCWO of 280Da), PuraMem® Selective (MCWO of 600Da), DuraMem® 300 (MCWO of 300Da) and DuraMem® 500 (MCWO of 500Da) were courtesy of Evonik Resource Efficiency GmbH, Germany. OSN membrane sheets are asymmetric polymeric membranes with a shiny yellow active membrane surface. Flat sheet membranes were provided in A4 size (210 × 297 mm) and were cut to suit the respective membrane housing.

Crude oil deacidification was performed using laboratory dead-end and crossflow membrane filtration systems.

Dead-end filtration experiments were carried out using the Sterlitech® HP4750 High-Pressure Stirred Cell (Sterlitech Corp., USA), as shown in *Figure 2*. The membrane cell assembly was partly immersed in a water bath to keep the crude oil temperature at approximately 50°C, and nitrogen gas maintained the membrane filtration operating pressure at 20 bar (g).

The Sterlitech® CF042 (Sterlitech Corp., USA) bench-scale modular crossflow filtration system, shown in *Figure 3*, was commonly used in the research and development of small batch processes. The unit comprises a pump, a membrane holder fitted with a valve for adjusting the pressure and devices for temperature control.

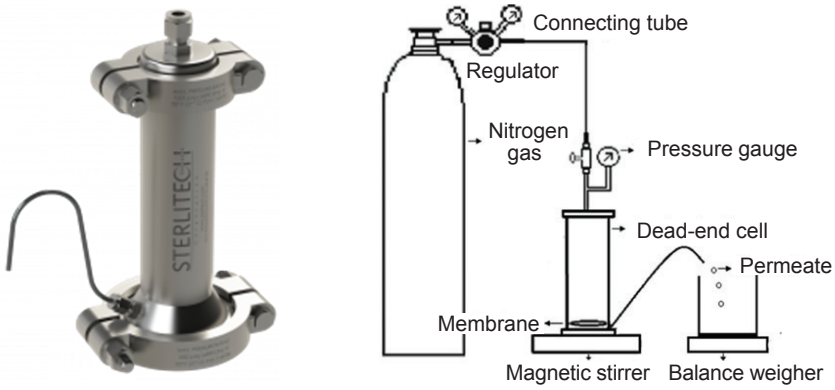

Figure 2. Sterlitech® HP4750 High-Pressure Stirred Cell with 14.6 cm² active membrane area.

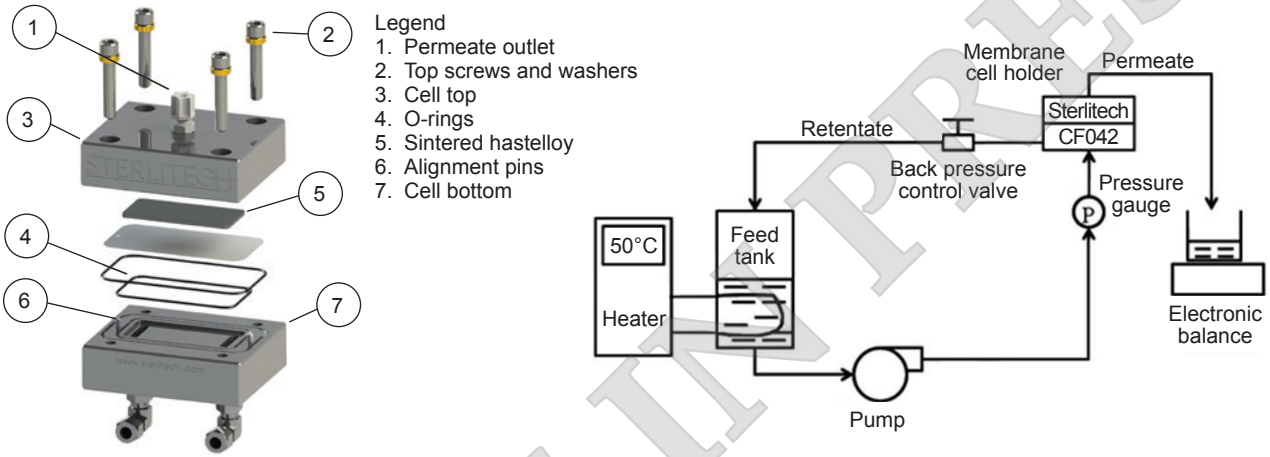

Figure 3. Sterlitech® CF042 Crossflow Cell with 42 cm² active membrane area.

A Lovibond tintometer was used to match the oil colour with a set of standard coloured number glasses, ranging from 0 to 70 red and 0 to 70 yellow. Results were expressed in R and Y values.

Method

A new conceptual approach, shown in *Figure 4*, has been proposed to improve the milling process. The modifications involved diluting crude oil during degumming and using membrane filtration to lower the levels of phospholipids and FFA, making the CPO suitable for deodorisation in the refinery without further treatment. This also helps in reducing the formation of 3-MCPD esters. The main principle of this technology is to eliminate an acid medium, thus preventing 3-MCPD formation during conventional refining.

Diluted crude oil degumming process optimisation. Degumming is the initial step in oil refining, which involves removing phospholipids. Gum removal was examined by adding different amounts of 85.00% H_3PO_4 to the crude oil collected after the vibrating screen, up to 0.10% (v/v). The mixture was stirred with a magnetic stirrer for a period up to 40 min at temperatures ranging from ambient to 90°C. Subsequently, 30.00% hot water (85°C) was added to the solution and thoroughly mixed for dilution. Excess H_3PO_4, phospholipids and water-soluble gums would be removed simultaneously. Finally, the degummed crude oil was separated using a centrifuge (*Figure 5*).

The phosphorus content in the oil layer was determined based on the standard phosphorus content determination method. The temperature, time and acid degumming dosage were evaluated to determine the optimum conditions.

FFA removal by membrane filtration. The second step involved removing FFA from degummed oil through membrane filtration, preferably without using any solvents as wetting agents. Membranes are generally classified as polar or nonpolar. PuraMem® membranes are stable in mild and non-polar solvents such as hexane and heptane, whereas DuraMem® membranes are stable in polar organic solvents like methanol.

A laboratory stainless steel dead-end filtration module system was used to investigate the feasibility of the OSN membrane for removing FFA from degummed crude oil. Each membrane

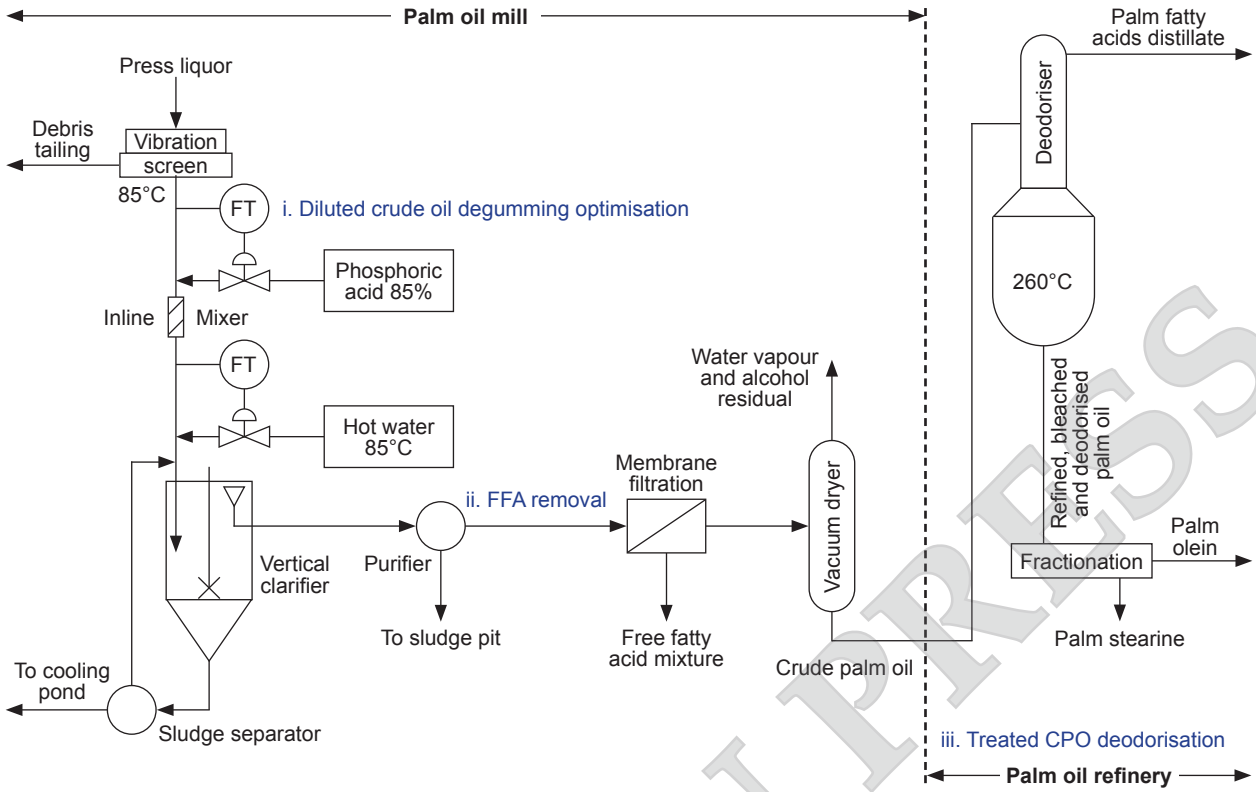

Figure 4. Conceptual milling enhancement process flow diagram.

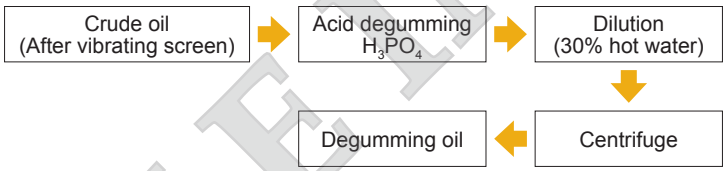

Figure 5. Diluted crude oil degumming process flow chart.

was cut into a circular disc with an approximate area of 20.43 cm^2 and fitted in the membrane cell such that the active surface encountered the feed material. The temperature and stirring speed were 50°C and 400 rpm, respectively. A total of 30% w/w of wetting agents (hexane, methanol or ethanol) was added to the degummed crude oil before filtration if necessary. Filtrations of 100 g of degummed crude oil were conducted in batch mode for 1 hr each at different pressures, ranging from 20 to 40 bar (g) under a nitrogen blanket. Triglycerides (TAG) were retained as retentate inside the cell, while FFA permeated through the OSN membrane (permeate) and were collected through a port beneath the membrane support (*Figure 2*).

The OSN membrane filtration for FFA removal from degummed crude oil was expanded to a laboratory stainless steel crossflow system due to the capacity limitation of the dead-end filtration system. Crossflow filtrations using PuraMem® membrane were operated in batch mode with continuous retentate recycling at 50°C and pressure ranging from 1 to 3 bar (g) for 1 hr each, as shown in *Figure 3*. The degummed crude oil was diluted with (0%, 10%, 20%, 25% and 30%) hexane.

The content of FFA in the retentate samples was determined based on the Malaysian Palm Oil Board (MPOB) test method (MPOB, 2005). The flux and pressure influence the separating process but were not considered in this study.

Deodorisation of treated CPO. The treated CPO was placed on a heating mantle at 105°C and pressure ranging from 2 to 4 mmHg. Bleaching earth was then added and left for 15 min. A vacuum Buchner funnel has been used to filter the oil, removing spent bleaching earth.

The deodoriser performance is subjected to pressure, temperature and retention time. 100 g of treated CPO was heated to 90°C with agitation using a magnetic stirrer under a nitrogen blanket in a three-neck round-bottom flask. The oil was then subjected to FFA removal at selected temperatures ranging from 200°C to 260°C for 30 to 40 min, as shown in *Figure 6*. The RBD oil samples were analysed for the quality parameters.

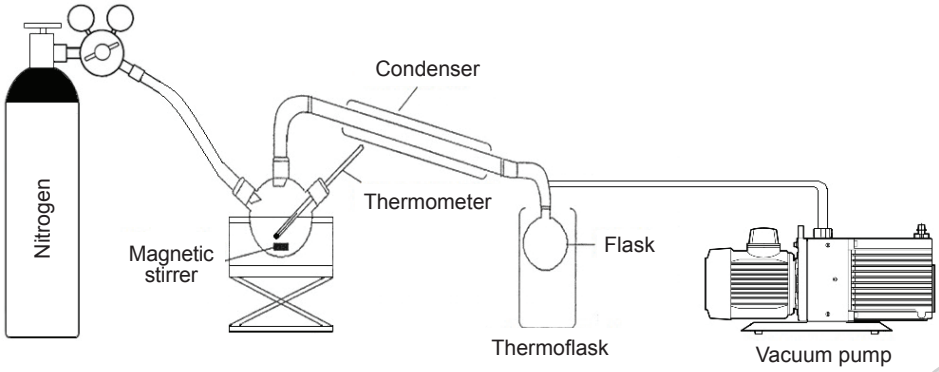

Figure 6. SCOPA laboratory deodoriser.

Oil quality. The FFA and Lovibond of redness have been determined according to the MPOB Test Method (MPOB, 2005). The content of 3-MCPD and GE were determined according to the American Oil Chemists' Society (AOCS, 2013) official method Cd 29a-13.

All the presented data is the average of three replicates. The precision of the related test methods has been presented in the relevant reference (MPOB, 2005).

RESULTS AND DISCUSSION

Diluted Crude Oil Degumming Process Optimisation

Optimal processing conditions in the degumming process for diluted crude oil were determined to be 80°C, 0.05% v/v H_3PO_4 (85%) and 30 min duration (*Table 1*). The optimal degumming process reduced the phosphorus content in diluted crude oil from 15.76 to 7.60 ppm.

TABLE 1. PHOSPHOROUS CONTENT IN ACID-DEGUMMED DILUTED CRUDE OIL

Variable condition	Phosphorus content (ppm)
Time (min)	
0	37.66
20	23.68
30	19.93
40	25.65
Temperature: 80°C; H_3PO_4: 0.05% v/v	
H_3PO_4 (% v/v)	
0.00	15.76
0.05	7.60
0.07	7.64
0.10	7.97
Temperature: 80°C; Time: 30 min.	
Temperature (°C)	
Room	35.43
70	20.77
80	19.46
90	22.44
Time: 30 min; H_3PO_4: 0.05% v/v	

Enzymatic degumming was first introduced in the year 2000 and showed an increase in oil yield. Phospholipases classification PLA_1, PLA_2, PLC and PLD are based on the cleaving position of the fatty acids. The temperature and pH in the water phase are crucial parameters. The enzyme substitution mechanism known as the "ping-pong" model describes the reaction pathway.

FFA Removal by Membrane Filtration

The effectiveness of FFA separation from crude oil for respective membranes was evaluated. An ideal membrane allows all FFA to pass through while retaining or rejecting all TAG. Results from laboratory dead-end filtration experiments for FFA removal from treated crude oil using various OSN membranes are shown in *Table 2*. Results showed that no filtrate was obtained unless the treated crude oil was mixed with hexane, and there was no observable enhancement in selectivity when alcohol was used as a cosolvent.

FFA removal from treated crude oil using OSN membrane filtration was expanded to a crossflow system due to the capacity limitations of the dead-end filtration system. The experimental results are shown in *Table 3*. No filtrate could be obtained unless more than 30% hexane was added to the degummed oil. The FFA content in the oil was reduced as solvent concentrations increased from 30% to 40%. The reduction percentage ranged from 26% to 31%. Similarly, increasing the filtration pressure from 1 to 3 bar (g) improved FFA rejection. Further pressure increases could not be performed due to equipment limitations. The evaluation showed that DM300 and PMF (MWCO <300) exhibited the best performance, achieving a 31% reduction in FFA in the treated oil.

Membrane filtration is a scalable industrial process. The membrane reusability, flux decline and potential scaling could be determined via membrane modelling of a selected membrane for the specific application.

TABLE 2. RESULTS FROM LABORATORY DEAD-END FILTRATION

Membrane type	Feed	Observation
PMF	Crude oil	No filtrate could be obtained
	Crude oil + hexane	Medium pass-through totally
PMS	Crude oil	No filtrate could be obtained
	Crude oil + hexane	Medium pass-through totally
DM 300	Crude oil	No filtrate could be obtained
	Crude oil + methanol/ethanol	No filtrate could be obtained
	Crude oil + hexane	Medium pass-through totally
DM 500	Crude oil	No filtrate could be obtained
	Crude oil + methanol/ethanol	No filtrate could be obtained
	Crude oil + hexane	Medium pass-through totally

Note: PMF - PuraMem® flux, 280 Da MWCO; DM 300 - DuraMem®, 300 Da MWCO; PMS - PuraMem® selective, 600 Da MWCO; DM 500 - DuraMem®, 500 Da MWCO.

TABLE 3. RESULTS FROM LABORATORY CROSSFLOW FILTRATION

Condition	Membrane	Solvent	Final FFA	Remark
Temperature: 50°C	DM300	Without solvent	-	No filtrate
Pressure: 1 bar (g)		10%–30% Methanol	-	No filtrate
Initial FFA: 4.71%		10%–30% Ethanol	-	No filtrate
		10%–20% Hexane	-	No filtrate
		30% Hexane	3.65	
		40% Hexane	3.51	
Temperature: 50°C	DM300	30% Hexane	3.36	
Pressure: 3 bar (g)		40% Hexane	3.25	
Initial FFA: 4.76%				
Temperature: 50°C	DM 500	Without solvent	-	No filtrate
Pressure: 1 bar (g)		10%–30% Methanol	-	No filtrate
Initial FFA: 4.70%		10%–30% Ethanol	-	No filtrate
		30% Hexane	3.68	
		40% Hexane	3.64	
Temperature: 50°C	DM 500	30% Hexane	3.36	
Pressure: 3 bar (g)		40% Hexane	3.34	
Initial FFA: 4.76%				
Temperature: 50°C	PMF	Without solvent	-	No filtrate
Pressure: 1 bar (g)		10%–20% Hexane	-	No filtrate
Initial FFA: 4.72%		30% Hexane	3.41	
		40% Hexane	3.52	
Temperature: 50°C	PMF	Without solvent	-	No filtrate
Pressure: 3 bar (g)		10%–20% Hexane	-	No filtrate
Initial FFA: 4.72%		30% Hexane	3.36	
		40% Hexane	3.26	
Temperature: 50°C	PMS	Without solvent	-	No filtrate
Pressure: 1 bar (g)		10%–20% Hexane	-	No filtrate
Initial FFA: 4.71%		30% Hexane	3.56	
		40% Hexane	3.48	
Temperature: 50°C	PMS	Without solvent	-	No filtrate
Pressure: 3 bar (g)		10%–20% Hexane	-	No filtrate
Initial FFA: 4.71%		30% Hexane	3.41	
		40% Hexane	3.36	

Note: PMF - PuraMem® flux, 280 Da MWCO; DM 300 - DuraMem®, 300 Da MWCO; PMS - PuraMem® selective, 600 Da MWCO; DM 500 - DuraMem®, 500 Da MWCO; FFA - free fatty acid.

Although the principle of membrane separation is commonly based on a size exclusion mechanism, earlier work indicated that the main limitations of FFA separation by membrane filtrations were small molecular-weight difference between triacylglycerols (>800 Da) and FFA (<300 Da), low selectivity of available membranes, low permeate flux and incompatibility with industrial needs (Kumar & Bhowmick, 1996). The ineffective membrane in crude oil deacidification is primarily ascribed to the small molecular weight difference between the TAG (oil molecules) and FFA, which upsets the membrane separation's capability. A small molecular weight cut-off (MWCO) membrane usually has high permeative resistance.

Furthermore, low FFA concentration in crude oil, usually less than 5%, was found to hinder the membrane's ability to separate the trace amount of FFA from the bulk. Many experiments have been conducted on FFA removal (Keurentjes et al., 1991; Raman et al., 1996), but there is not enough commercial information available on the membrane technology for vegetable oil deacidification, because FFA separation via a standard pressure-driven membrane process is practically challenging. Subramanian et al. (1998) reported that a single-step membrane filtration process was only suitable as an alternative to chemical refining for degumming and clarification stages, not for deacidification.

Deodorisation of Treated CPO

Filtration with an OSN membrane was only able to reduce about 30% of FFA in crude oil, lowering it from 4.72% to 3.26% in the retentate. The membrane-filtered oil was refined using Scopa Bleaching without acid degumming to produce refined, bleached, and deodorised palm oil (RBDPO). The FFA content in the RBDPO was slightly higher than the PORAM specification due to the limitations of the vacuum pump in providing the necessary pressure below 5 mmHg. The levels of 3-MCPD and glycidyl ester were 0.5 and 11.0 mg kg^{-1}, respectively, which are comparable to those in conventional deodorisation, as shown in *Table 4*.

FFA is the main impurity affecting RBDPO quality and must be reduced as low as reasonably possible. The removal of FFA during milling was insufficient; therefore, thermal deodorisation during refining became necessary. The formation of 3-MCPD and glycidyl ester theoretically occurs when FFA is eliminated from diacylglycerol at high temperatures, ranging from 200°C to 260°C (Craft et al., 2012). Lower deodorisation temperatures could reduce 3-MCPD formation, but would also decrease FFA removal efficiency.

TABLE 4. COMPARISON OF COMMERCIAL AND RBDPO PRODUCED

Parameters	Commercial RBDPO	RBDPO produced
FFA (%)	0.1	1–2
Colour (5.25" Lovibond Cell)	Below 3.0 R	Below 3.0 R
3-MCPD (mg kg^{-1})	0.2–2.0*	0.5
Glycidyl ester (mg kg^{-1})	10–15*	11
Hexane residue (mg kg^{-1})	Not applicable	Not detected

Note: * - source from Hew et al. (2021).

Colour is an important characteristic of commercial vegetable oil. PORAM specifications have fixed the maximum 5.25" Lovibond Cell colour value at 3.0 R. Results showed that the proposed new milling process approach can produce CPO that is good for thermal deodorisation without acid degumming.

CONCLUSIONS

The optimal conditions for diluted crude oil degumming were 80°C, 0.05% v/v 85.00% H_3PO_4 and a 30 min reaction time. Diluted crude oil with an initial phosphorus content of 15.76 ppm could be reduced to 7.60 ppm following the optimal degumming process. OSN membrane filtration showed low rejection of FFA, with a 30.00% reduction from 4.72% to 3.26% in the retentate. The removal efficiency of FFA was not satisfactory and should be further improved. The contents of 3-MCPD and glycidyl ester in the produced RBD palm oil were 0.5 and 11.0 mg kg^{-1}, respectively, comparable to conventional products. Therefore, the proposed milling enhancement could prevent the formation of the mono-chloro-propane-diol ester by eliminating medium acidity during the CPO refining process. The additional CAPEX and operating expenditure (OPEX) should be minimal, with an attractive investment return rate (IRR), especially through business-to-business negotiations for a premium price for the enhanced quality of CPO and the recovered FFA filtrate as downstream oleo-chemical feedstock.

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