New Developments in Palm Oil Fractionation

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INTRODUCTION

Fractionation, a precursor of the modern edible oil and fat processing industry, is the oldest separation process. It plays an important role, especially in the palm oil industry, owing to the composition of palm oil which contains about equal amounts of saturated and unsaturated fatty acids. The physical nature of palm oil, exhibiting a semi-solid state in the Malaysian tropical climate, allows its separation into a low-melting fraction, olein, and a high-melting fraction, stearin (Deffense, 1985). Fractionation can be defined as the separation of a mixture into its component fractions. Generally, the concept of a physical separation process can be based on a few parameters such as differences in solidification, solubility and volatility of the different compounds. The common techniques used for fractionation are fractional crystallisation, fractional distillation, short-path distillation, supercritical fluid extraction, liquid-liquid extraction, adsorption, complexion and membrane separation. (Kellens *et al*., 2007). In the oils and fats industries, fractional crystallisation is the process used for separating oils and fats into two or more components, and it involves two steps: selective crystallisation and filtration. There are three fractionation processes used to fractionate palm oil, namely, dry fractionation, detergent fractionation and solvent fractionation.

Dry Fractionation

Dry fractionation is the simplest and cheapest process, which is most commonly used in palm oil refineries. As the name suggests, it is a dry process using direct filtration of triacylglycerol crystals after controlled programmed cooling. This process is simple because it does not depend on any chemical and no effluent is produced during the process. Hence, this process has an advantage of minimum product losses (Kellens *et al*., 2007). In dry fractionation, the oil is partially crystallised by controlled cooling of the molten feed to the desired fractionation temperature, holding the partially crystallised slurry for crystal growth, followed by filtration using a membrane filter press (Gunstone, 2001). *Figure 1* shows various palm oil products obtained from single-, double- and triple-stage dry fractionation processes. Single-stage dry fractionation of palm oil can be used to produce palm oleins with IV of 56 and 62. The saturation content of palm olein can be further reduced by using multiple stage fractionation in which double- and triple-stage fractionation can be used to produce oleins with IV of 65 (super olein) and IV 70 (top olein), respectively (Gijs *et al*., 2007; Kellens *et al*., 2007).

Detergent Fractionation

Detergent fractionation, first developed by Lanza, involves the addition of a detergent as a wetting agent to improve the separation of the crystals from the liquid phase (Deffense, 1985). Sodium lauryl sulfate is usually used as the wetting agent, in combination with magnesium sulfate as the electrolyte (Kellens and Hendrix, 2000).

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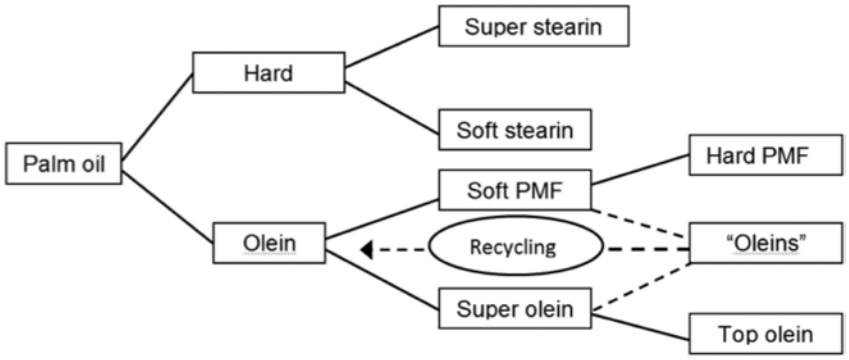

Figure 1. Dry fractionation of palm oil and its products from single-, double- and triple-stage dry fractionation.

When the partially crystallised slurry is mixed with the detergent solution, the crystals are wetted by the detergent and are easily suspended in the aqueous phase; the mixture is then separated by centrifugation (Kellens *et al*., 2007). The aqueous phase is then heated and the melted stearin is recovered through a second centrifugation step. The olein and stearin fractions are washed with water and dried to remove the trace amounts of detergent (Deffense, 1985). Detergent fractionation has lost its appeal due to detergent contamination of the end products and the high production cost (Kellens *et al*., 2007).

Solvent Fractionation

Solvent fractionation is the most efficient fractionation process compared with the other fractionation processes (Kellens and Hendrix, 2000). It was initially developed to overcome some bulk crystallisation problems, such as slow heat transfer and high viscosity which limit nuclei movement. In solvent fractionation, the oil is diluted in organic solvents such as acetone and hexane in certain proportions to reduce its viscosity, which is higher when using dry and detergent fractionations (Kellens *et al*., 2007). In dry fractionation, it is not possible to remove all the liquid from the solid phase, as some liquid will remain entrapped in the solid fraction. Solvent fractionation is more efficient in reducing liquid oil entrapment, thus enhancing the liquid oil yield as well as increasing the hardness of the solid fraction. Similar to detergent fractionation, solvent fractionation has also lost its lure due to its high production cost and higher risk of fire and to human health (Kellens *et al*., 2007).

CURRENT DEVELOPMENTS IN PALM OIL FRACTIONATION

Recent developments in palm oil fractionation are mainly focused on areas related to crystallisation and solid-liquid separation. The quality of the olein fraction depends only on the crystallisation step, whereas the quality of stearin fraction depends on both the crystallisation and separation steps (Timms, 2005). A few stages are required for the fractionation of fats. They are supercooling of the melt, nucleation and crystal growth (Kellens *et al*., 2007). At temperatures much lower than the supercooling temperature, nuclei can be formed in the system (Lawler and Dimick, 2002). Nuclei are defined as the smallest crystals that can exist in a solution at a particular concentration and temperature (Timms, 2005). There are three types of nucleation phenomena, *i.e*. primary nucleation including homogeneous nucleation, heterogeneous nucleation, and secondary nucleation (Kellens *et al*., 2007). Homogeneous nucleation is nucleation that occurs in the bulk mother phase, while heterogeneous nucleation refers to nucleation onto foreign substances in the crystalliser such as dirt, walls of crystallisers, *etc.* Secondary nucleation happens when tiny crystallites are removed from the surface of existing crystals which will then act as new nuclei in the crystallisation system (Kellens *et al*., 2007). Hence, secondary nucleation is undesirable in any fractionation process (Timms, 2005).

Once nuclei are formed, they will start to grow by incorporating the TAG molecules from the adjacent liquid layer. Crystal growth is affected by internal and external factors. External factors include the degree of supercooling, the presence of inhibitors, *etc.*, while internal factors are polymorphic form, crystal morphology, crystal defects, *etc.* (Foubert *et al*., 2007). To ensure uniform crystal growth, crystallisation must proceed continuously in a system that allows homogeneous contacts of the nuclei with the surrounding supersaturated liquid (Timms, 2005). Therefore, sufficient agitation with a non-destructive feature is important to ensure continuous and uniform crystallisation (Kellens *et al*., 2007).

MODIFICATION OF CRYSTALLISER DESIGN

Modification of crystalliser design is one of the areas receiving most

attention in the development of fractionation technology. A typical crystalliser is fitted with a cooling coil and an agitator. The agitator design can affect the mass and heat transfers from the crystallising oil to the cooling surface (Kellens and Hendrix, 2000). In recent years, there have been some modifications in crystalliser design. These modifications serve to reduce utilities consumption, and to increase yield and production of higher purity fractions, thereby reducing processing cost. A good combination of efficient cooling and agitation systems is important to ensure the formation of highly filterable crystals during membrane filtration.

Some crystallisers are equipped with double-layered cooling coils instead of a single cooling coil. Double-layered cooling coils are used to provide faster heat transfer and thermal homogeneity within the oil while maintaining minimal disturbance to crystal growth (Lipico, 2008). In the Oiltek dry fractionation plant technology, hybrid cooling coils are used to provide higher yields and shorter cooling times. Tall and slim crystallisers with programmed agitator speed are designed to provide efficient heat transfer between the coils and the oil. In addition, an agitator with a lower dish end and a radial lower paddle is used to facilitate slurry circulation and prevent the slurry from settling to the bottom of the crystalliser. Agitators with speed variators and a programmable rate of agitation are used to ensure agitator speed control and efficient homogenisation of the slurry with minimum disturbance to the process (Oiltek, 2012). The Alfa Laval fractionation technology uses a modular crystalliser design. This design enables increases in production capacity by the addition of more crystallisers and filter capacity (Alfa Laval, 2014). Today, fractionation technologies incorporate fully automated control of the crystallisers and membrane filter press via programmed temperature controllers and programmes for filter press feeding and squeezing, respectively, to ensure consistent productivity.

CONTINUOUS FRACTIONATION

Continuous fractionation involves modifications to both crystallisation and process flow. In 2013, the Desmet Ballestra Group filed a patent for the continuous fractionation of triacylglyceride oils (Kellens *et al*., 2013). The process involves the use of one or more crystallisers in series. In the process, molten fat is fed continuously to the crystallisers in series, during which time the fat is cooled gradually using heat exchangers to initiate crystallisation and to allow the formation of crystal growth. Each of the crystallisers exhibits a temperature gradient. The crystallising slurry is continuously withdrawn from the last crystalliser and filtered (Kellens *et al*., 2013).

MODIFICATION OF COOLING PROGRAMME

Modification of fractionation conditions is another research area that is crucial for improvement of the fractionation process. A basic understanding of the crystallisation of palm oil products is necessary before it can be applied to the fractionation process. A lot of fundamental research studies on palm oil crystallisation have been carried out by MPOB (Chen *et al*., 2004; Zaliha *et al*., 2005; Chong *et al*., 2007; Norizzah *et al*., 2012) over the last 10 years. Chong *et al*. (2015) offered a technology for adoption which promises higher olein yield production. The technology shows that the fractionation process can be improved by modifying the fractionation cooling programme alone, while additional steps are incorporated mid-way during the fractionation process.

In 2014, two process patents were filed, one for crude palm oil and the other for RBD palm oil fractionation in which the additional steps were incorporated (Chong *et al*., 2014a, Chong *et al*., 2014b). These are named the MPOB modified fractionation programmes for crude palm oil and RBD palm oil fractionation, respectively. The patented processes produce smaller mean diameter crystals with greater size homogeneity compared with the conventional cooling programme. This results in easier filtration leading to a reduction in olein entrainment, thus increasing the olein yield. The increase in olein yield is achieved solely through an alteration to the fractionation cooling programme used by the refinery. No capital investment is involved in this technology because it does not require any equipment upgrade on the existing fractionation process. The increase in olein yield will benefit the refinery due to the price differential between the olein and stearin.

ADDITION OF CRYSTALLISATION AIDS

The use of crystallisation aids is another interesting area of development in palm oil fractionation. Over the last few years, research collaboration between MPOB and Sakamoto Yakuhin Kogyo has

shown that the fractionation process can be improved by the use of polyglycerol ester additives. Kuriyama *et al*. (2011) reported that GRAS status additives can be used to aid the fractionation process for improved yields and fraction characteristics.

Laboratory-scale fractionation conducted by MPOB has also shown a significant increase in olein yield. The laboratory experiments were conducted using low concentrations of the PGEmix-8 additive at 0.1%, 0.3%, 0.5% and 0.7% w/w. A control run was carried out without addition of the additive. The crystallisation was conducted at an isothermal condition of 24°C for 100 min. The slurry was filtered using vacuum filtration.

Figure 2 shows the olein yield of the laboratory-scale fractionations at different dosages of PGEmix-8, which is a polyglycerol ester made up of a mixture of palmitic, oleic and stearic acids. It was observed that olein yield was significantly raised with increasing levels of PGEmix-8 used in the fractionation process. The olein and stearin fractions were analysed for their triacylglycerols and fatty acids compositions. *Table 1* shows a comparison of the acylglycerol composition of olein fractions after the isothermal crystallisation. It was found that the acylglycerol composition of all oleins was similar and comparable. As expected, fatty acid composition (FAC) of the oleins, shown in *Table 2*, also produced similar findings, *i.e.* FAC of oleins with the use of the additive was similar to that of the control. This indicates that the use of the additive in the fractionation process did not significantly change the overall composition of the olein products.

Unlike the olein fractions, FAC of the stearins was significantly altered by the PGEmix-8 additive. *Figure 3* shows the total FAC of the stearin fractions when the PGEmix-8 additive was used. It was found that the total saturated fatty acids (SFA) of the stearins were increased while the monounsaturarated fatty acids (MUFA) were reduced with increasing amounts of PGEmix-8 added into the fractionation process. The polyunsaturated fatty acids (PUFA) content remained unchanged for all the stearins. This indicates that the stearins produced with the additive were purer and contained less amounts of unsaturation components, as a result of less olein entrainment with the use of the additive. The result is similar to the findings of Kuriyama *et al*. (2011), who reported that olein yields can be improved by including additives into the process without compromising olein quality.

MODIFICATION OF FILTRATION

To complete the fractionation process, the solid phase needs to be separated from the liquid phase at the fractionation temperature. Efficient separation is essential to ensure production of a purer stearin fraction with minimum entrainment of the olein. There are a few separation methods used in fractionation plants. These include centrifugation, vacuum filtration and membrane filtration (Timms, 2005). The principal of centrifugation is based on the density difference between the solid and liquid phases of the slurry. It is only useful for separation of a slurry system with a density difference of 10% or more between the solid and the liquid phases. Therefore, this separation method is only applicable to detergent fractionation (Timms, 2005).

For dry fractionation, vacuum filtration and a membrane filter press are used. The design of the vacuum filtration is similar to a laboratory vacuum filtration system in which a vacuum is applied to suck the liquid oil through a filter (Timms, 2005). There are two types of vacuum filtration used industrially, the belt filter (Tirtiaux) and the drum filter (Desmet) (Kellens and Hendrix, 2000). Belt filters give better separation in terms of producing stearin with a lower level of entrainment than drum filters (Timms, 2005).

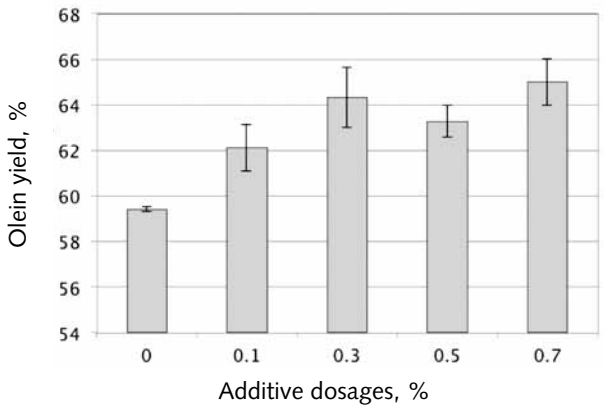

Figure 2. Olein yields of laboratory scale fractionation with different dosages of PGEmix-8.

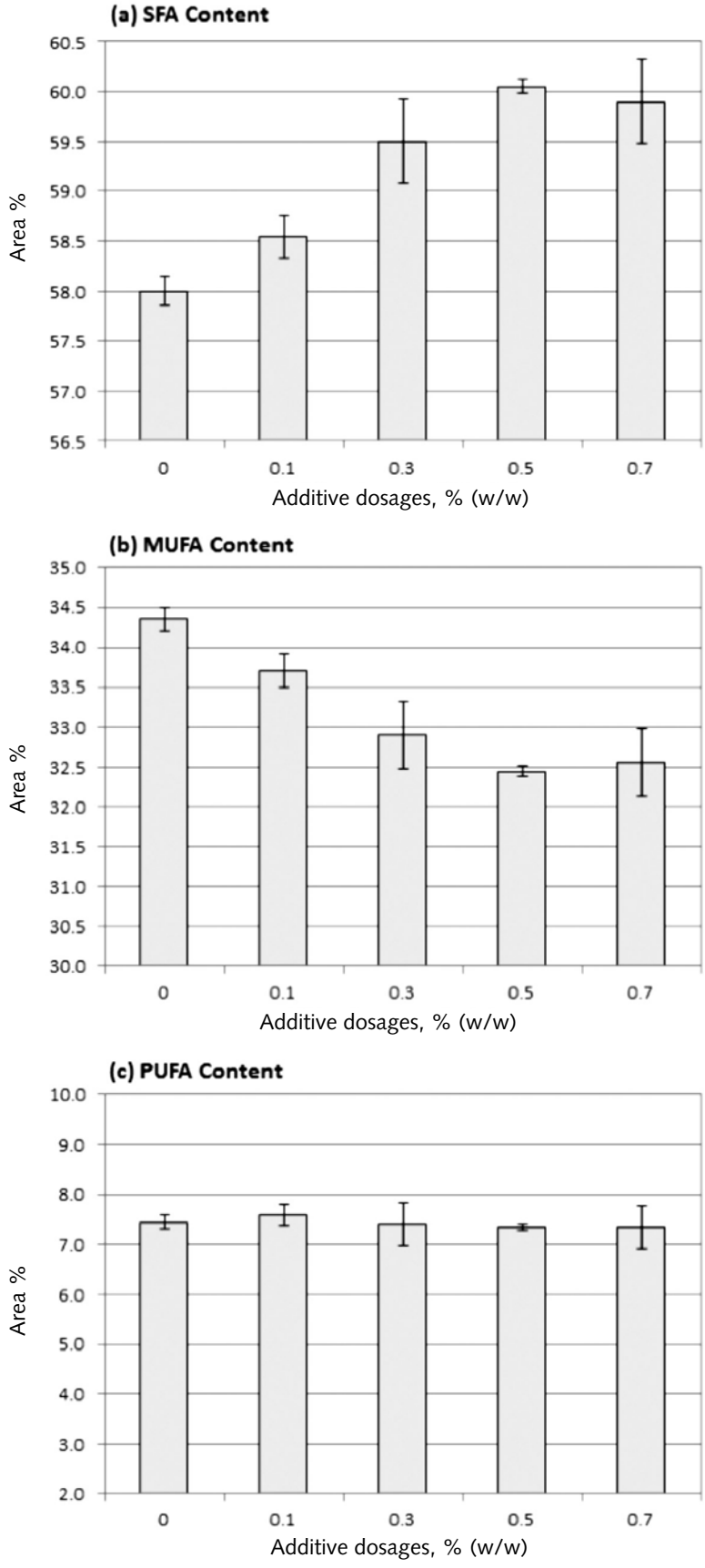

Figure 3. Total (a) SFA, (b) MUFA and (c) PUFA contents in the stearin fractions with the use of PGEmix-8 additives.

TABLE 1. COMPARISION OF ACYLGLYCEROL COMPOSITION OF OLEIN FRACTIONS AFTER ISOTHERMAL CRYSTALLISATION AT 24°C FOR 100 MIN

Acylglycerol, Area %	Dosage of PGEmix-8, %				
	0.0	0.1	0.3	0.5	0.7
FFA + MAGs	**0.3 ± 0.1**	**0.1 ± 0.0**	**0.2 ± 0.0**	**0.2 ± 0.0**	**0.3 ± 0.1**
Total DAGs	**6.4 ± 0.0**	**6.2 ± 0.2**	**6.2 ± 0.1**	**6.1 ± 0.0**	**6.1 ± 0.1**
OLL	0.4 ± 0.0	0.5 ± 0.1	0.5 ± 0.0	0.5 ± 0.0	0.5 ± 0.0
OLO	2.0 ± 0.1	2.1 ± 0.1	2.1 ± 0.1	2.1 ± 0.1	2.1 ± 0.0
OOO	5.3 ± 0.1	5.2 ± 0.1	5.2 ± 0.0	5.2 ± 0.0	5.3 ± 0.2
Triunsaturated TAGs	**7.7 ± 0.1**	**7.8 ± 0.2**	**7.8 ± 0.0**	**7.8 ± 0.1**	**7.8 ± 0.2**
PLL	2.4 ± 0.0	2.4 ± 0.1	2.5 ± 0.0	2.5 ± 0.1	2.4 ± 0.0
PLO	10.3 ± 0.0	10.4 ± 0.0	10.3 ± 0.0	10.2 ± 0.1	10.3 ± 0.0
POO	26.7 ± 0.1	26.8 ± 0.4	26.4 ± 0.2	26.4 ± 0.1	26.6 ± 0.0
SOO	3.1 ± 0.2	3.3 ± 0.3	3.5 ± 0.0	3.2 ± 0.0	3.3 ± 0.1
Diunsaturated TAGs	**42.5 ± 0.2**	**42.9 ± 0.1**	**42.7 ± 0.2**	**42.4 ± 0.1**	**42.5 ± 0.0**
MLP	0.6 ± 0.0	0.6 ± 0.1	0.7 ± 0.1	0.7 ± 0.1	0.6 ± 0.0
PLP	9.8 ± 0.2	9.5 ± 0.1	9.5 ± 0.0	9.5 ± 0.1	9.6 ± 0.1
POP	26.8 ± 0.1	26.8 ± 0.3	26.5 ± 0.3	26.8 ± 0.1	26.8 ± 0.0
POS	4.8 ± 0.1	4.8 ± 0.1	5.0 ± 0.1	4.9 ± 0.0	5.0 ± 0.1
SOS	0.7 ± 0.1	0.7 ± 0.1	0.7 ± 0.0	0.9 ± 0.3	0.6 ± 0.0
Disaturated TAGs	**42.6 ± 0.1**	**42.4 ± 0.0**	**42.4± 0.1**	**42.7±0.1**	**42.5 ± 0.0**
PPP	0.5 ± 0.1	0.6 ± 0.2	0.6 ± 0.0	0.7 ± 0.0	0.5 ± 0.1
PPS	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.1	0.1 ± 0.1	0.3 ± 0.2
Trisaturated TAGs	**0.5 ± 0.1**	**0.6 ± 0.2**	**0.8 ± 0.2**	**0.7 ± 0.1**	**0.8 ± 0.0**
Total TAGs	**93.2 ± 0.2**	**93.8 ± 0.2**	**93.7 ± 0.1**	**93.7 ± 0.0**	**93.7 ± 0.2**

TABLE 2. COMPARISON OF FATTY ACID COMPOSITION (FAC) OF OLEIN FRACTIONS AFTER ISOTHERMAL CRYSTALLISATION AT 24°C FOR 100 MIN

Olein samples, %(w/w) of PGEmix-8	FAC, Area %											
	C12:0	C14:0	C16:0	C18:0	C20:0	SFA	C16:1	C18:1	MUFA	C18:2	C18:3	PUFA
Olein-control	0.2±0.0	1.0±0.0	39.7±0.1	4.0±0.0	0.4±0.0	**45.3±0.1**	0.2±0.0	44.1±0.0	**44.3±0.0**	10.1±0.1	0.3±0.0	**10.4±0.1**
Olein-0.1%	0.2±0.0	0.9±0.0	39.5±0.1	4.1±0.0	0.4±0.0	**45.1±0.1**	0.2±0.0	44.1±0.1	**44.3±0.1**	10.3±0.0	0.3±0.0	**10.6±0.0**
Olein-0.3%	0.2±0.0	0.9±0.0	39.4±0.0	4.1±0.0	0.4±0.0	**45.0±0.0**	0.2±0.0	43.9±0.0	**44.1±0.0**	10.4±0.0	0.3±0.0	**10.7±0.0**
Olein-0.5%	0.2±0.0	0.9±0.0	39.5±0.0	4.1±0.0	0.4±0.0	**45.1±0.0**	0.2±0.0	43.9±0.1	**44.1±0.1**	10.4±0.1	0.3±0.0	**10.7±0.1**
Olein-0.7%	0.2±0.0	1.0±0.0	39.6±0.1	4.1±0.0	0.4±0.0	**45.3±0.1**	0.2±0.0	43.8±0.1	**44.0±0.1**	10.4±0.1	0.3±0.0	**10.7±0.1**

Note: SFA = saturated fatty acids; MUFA = monounsaturated fatty acids; PUFA = polyunsaturated fatty acids.

The most common filtration method used at present is the membrane filter press. During operation, less than one bar pressure is applied to fill the filter press with the crystallised slurry. Higher pressure is then applied to squeeze the entrained liquid oil. A standard filter press normally applies a pressure of 5-6 bars. A high-pressure membrane filter press can operate up to 30 bars (Kellens and Hendrix, 2000). In general, membrane press filtration is the most efficient separation method with the lowest liquid oil entrainment in the stearin fraction, as well as producing the highest olein yield.

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CONCLUSION

The above is a brief review of developments in palm oil fractionation over the last 30 years. Although the use of the PGEmix-8 additive in palm oil fractionation has shown great potential in improving the fractionation process, it is not feasible at present due to the high cost of the additive. In contrast, the MPOB technology for increased olein yield shows promise as it is a zero investment technology with low to no risk, and the promise of financial gains which is dependent mainly on the price differential between olein and stearin and the quantum of increase in olein yield that can be obtained based on the operational constraints of the refinery concerned.

ACKNOWLEDGEMENT

The authors would like to thank the Director-General of MPOB for permission to publish this article.

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