The Role of Liquid Entrainment and its Effect on Separation Efficiency in Palm Oil Fractionation

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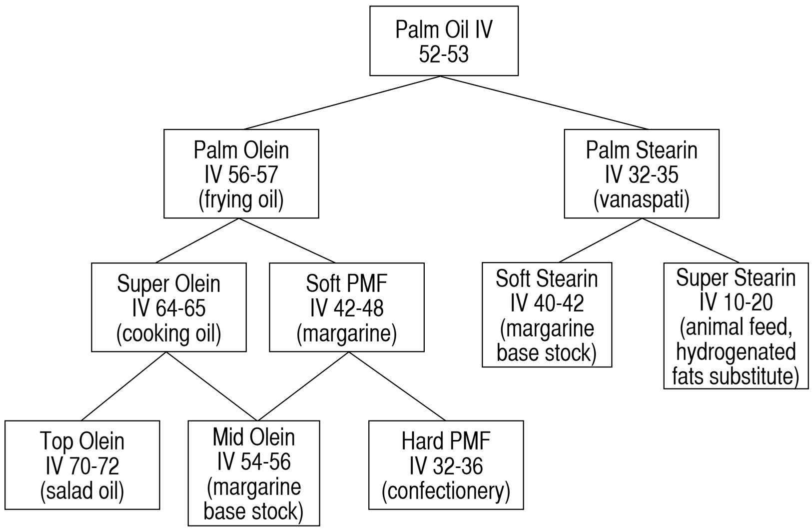
INTRODUCTION

Palm oil is one of the most unique oils and fats. The oil consists of a wide array of triacylglycerols (TAG) which predominantly contribute to its distinct physical and chemical properties. In its natural state, palm oil is semi-solid at room temperature which allows the oil to undergo fractionation to enhance its characteristics while increasing its functionality as an ingredient in a multitude of edible and non-edible applications (Deffense, 1985; Deffense, 1998). Over the last half century, the fractionation process has become the dominant modification process for the Malaysian palm oil industry, together with the steady growth in palm oil production (Kellens *et al*., 2007). Palm oil fractionation produces a liquid fraction (palm olein) and a solid fraction (palm stearin), which are two major palm-based fractions produced and traded from Malaysia (Parveez *et al*., 2020). In 2020, Malaysia produced over 14.2 million tonnes of refined, bleached and deodourised (RBD) palm oil while the production of RBD palm olein and RBD palm stearin were in excess of 10.1 million tonnes and 2.9 million tonnes, respectively, in the same year (MPOB, 2020). Palm oil can also undergo fractionation in multiple stages to further produce various fractions with improved quality and at a higher degree of selectivity, as shown in *Figure 1* (Kellens *et al*., 2007; Deffense, 2009).

In principle, fractionation involves partially crystallising the melted oil under controlled cooling conditions followed by separation of the solid crystals from the crystallising liquid slurry by filtration (Kellens *et al*., 2007). Several types of fractionation processes which have been employed by the palm oil industry with some still operating today are detergent fractionation, solvent fractionation and dry fractionation. Dry fractionation is currently the most preferred fractionation process for palm oil. The advantages of dry fractionation in comparison with other fractionation processes are that it is a purely physical, fully reversible and economical process due to the absence of chemicals or additives, and it has a low environmental impact making it a 'green' technology (Timms, 2005a; Kellens *et al*., 2007). A number of factors which affect the quality of the solid and liquid fractions obtained by fractionation include the origin and quality of raw material, crystalliser design, processing conditions, polymorphism and separation technique (Deffense, 2009). However, one of the main limiting aspects of the fractionation process is the difficulty in clean separation of solid crystals from liquid due to entrainment (Deffense, 2000).

WHAT IS ENTRAINMENT?

Entrainment is the physical trapping or retention of the liquid fraction by the solid crystalline fraction during the separation stage of the fractionation process (Timms, 1994). This happens as a result of the 3D structure of agglomerated crystals where liquid is held within crystal fissures by capillary and viscous forces. Two types of entrainment can occur during the separation stage: (1) intra-particle entrainment, where liquid oil is trapped within a particle or particle agglomerates and (2) inter-particle entrainment, where uncrystallised liquid is trapped between agglomerates (*Figure 2*). Among the factors which have a direct influence on entrainment include the operating conditions applied during crystallisation, the hydrokinetics and design of the crystalliser and the type of separation technique used (Bemer and Smits, 1982). The level of entrainment in the stearin cake consequently determines the yield but not the quality of the olein fraction, whereas for the stearin fraction, both quality and yield are affected (Timms, 2005b). Entrainment is a well-known phenomenon which reduces the separation efficiency of fractionation and complicates enrichment of the filtered products.



Adapted from Kellens *et al*., (2007); Deffense, (2009).

Figure 1. Multi-stage dry fractionation of palm oil and usage of fractions.

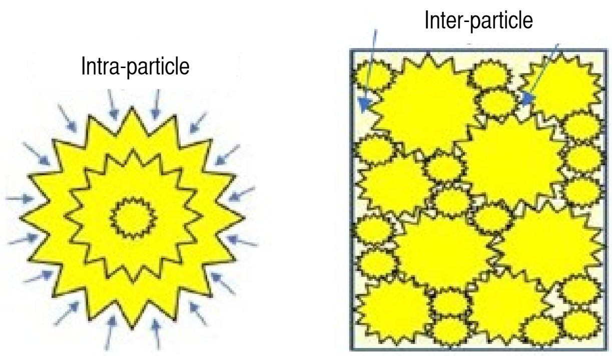


Figure 2. Intra-particle and inter-particle entrainment.

METHODS FOR ESTIMATING ENTRAINMENT

There are several methods that can be employed to measure the level of liquid entrainment within the stearin cake. One of the pioneering studies for entrainment calculation was conducted by Bemer and Smits (1982). Their study incorporated in the mass balance of the filter cake the radioactivity of the samples from the crystal-liquid slurry and from the pure liquid phase using liquid scintillation spectrometer. Other methods which have been proposed since then by several authors include those by Hamm (1986) using the olein fraction composition, applying iodine values of both solid and liquid fractions in the mass balance of the filter cake (Timms, 1994) and correlating entrainment to the degree of filter cake porosity (Hamm, 2005). Nowadays, the most popular method for measuring entrainment is through direct measurement by pulsed nuclear magnetic resonance (pNMR) of the solid fat content (SFC) of the crystallising slurry (Calliauw *et al*., 2007) or SFC of the filter cake immediately following filtration (Arnaud and Collignan, 2008) at the filtration temperature. This method provides a quick way in estimating the separation efficiency of the fractionation process.

A more recent method by Hishamuddin *et al.,* (2020) was developed based on an overall mass balance involving the mass and TAG compositions by high performance liquid chromatography of the olein and stearin products obtained from filtration. The application of this method considers that the melting points of triunsaturated TAG in palm oil which all lie below 0°C allows the them to remain uncrystallised in a liquid state at crystallisation operating conditions. Thus, the presence of triunsaturated TAG in the stearin filter cake is proposed to be ascribed purely to the entrapped olein within stearin crystals. The mass balance equation for the stearin cake is given by:

$$M_E x + M_C y = (M_E + M_C) z \qquad \text{Equation (1)}$$

where M_E is the mass (g) of entrained olein in the stearin cake, M_C is the mass (g) of crystals in the stearin cake, y is the composition of the triunsaturated TAG (g TAG/g crystals) in the crystals, and x and z are the compositions of the triunsaturated TAG in the corresponding olein (g TAG/g olein) and stearin cake fractions (g TAG/g stearin cake) after filtration, respectively, as determined by high performance liquid chromatography. *Figure 3* illustrates M_E and M_C within the cross-section of a stearin filter cake.

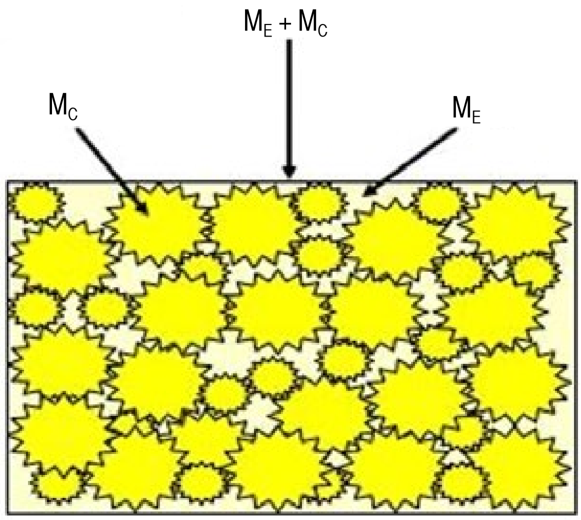
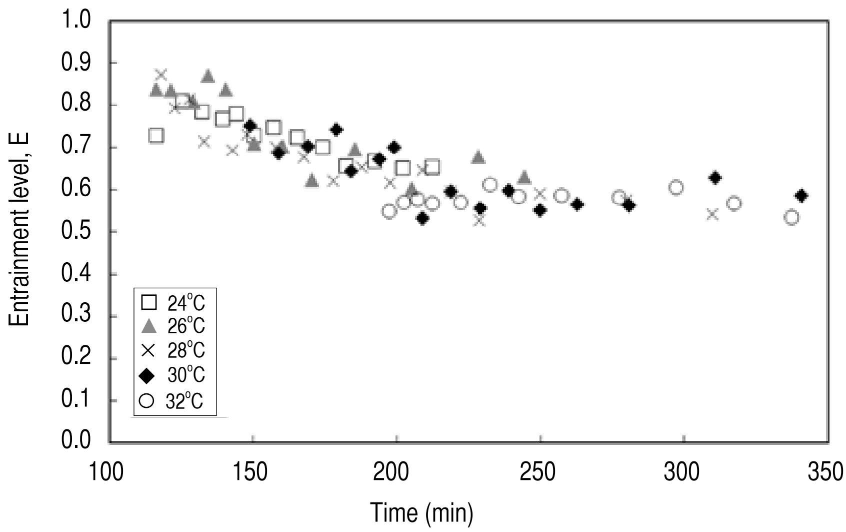


Figure 3. Distribution of mass of crystals (M_C) and mass of entrained olein (M_E) within a stearin filter cake.

The method proposed by Hishamuddin *et al*. (2020) found that entrainment levels in the stearin cake during the early stages of palm oil crystallisation at isothermal temperatures between 24°C and 32°C ranged from 70%-90% on the basis of filter cake mass. Their study concluded that the large surface to volume ratio of the high number of nuclei during this stage caused intra-particle occlusion in the growing crystals structure, in addition to the inability of uncrystallisable TAG species to rapidly diffuse away from the surface of crystals. A gradual decrease in entrainment values to between 54% and 65% was observed at the end of the crystallisation stage, owing to the reduced surface area to volume ratio of the crystals as a result of crystal growth (*Figure 4*). The entrainment values presented in their study were consistent with those reported in earlier observations (Hamm, 1986; Timms, 2005a; Kellens *et al.*, 2007). The proposed method provides an alternative for measuring the SFC in the absence of NMR facilities and avoids the need for strict sample temperature control which is usually required in SFC measurements using NMR.

SEPARATION PROCESSES AND THEIR EFFICIENCIES

In the last few decades, various types of separation processes have been commercially applied in palm oil fractionation. These include vacuum filtration using rotary drum or belt filter, decanting, centrifugation, hydraulic press and membrane filter press (Timms, 2005b). Earlier studies on palm oil fractionation have reported a substantial increase in the SFC of the stearin cakes produced by the membrane filter press to 55% compared to those obtained through vacuum filtration at 41% (Kellens, 1994; Hamm, 2005). Higher olein yields can be obtained with significant reductions in entrainment levels when using membrane filter press compared to vacuum filtration and centrifugation, as presented in *Table 1* (Timms, 2005a; Kellens *et al.*, 2007). The application of pressure during the filtration stage has also been shown to significantly affect the quality of fractionated products. During the operation of membrane filter presses, squeezing pressure is applied to compressible filtration chambers to expel entrapped olein from the slurry. This produces a better quality 'dry' stearin cake and greater olein yield due to the increase in the separation efficiency (Willner, 1994).



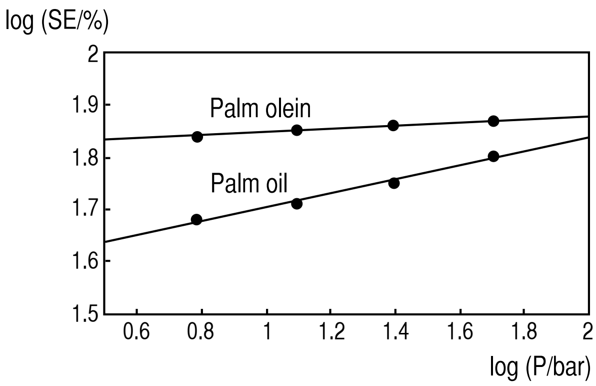
Source: Hishamuddin *et al*. (2020).

Figure 4. Entrainment levels in stearin cakes at different isothermal temperatures as a function of time.

TABLE 1. COMPARISON OF FILTRATION DATA BETWEEN DIFFERENT FILTRATION TECHNIQUES IN A PALM OIL FRACTIONATION PLANT

Filtration data	Vacuum filtration (drum/belt)	Centrifugation (nozzle)	Membrane press (6 bar/16 bar)
Entrained oil in filter cake (%)	59	53	45 / 35
Olein yield (%)	71-72	74-76	78 / 82

Source: Adapted from Timms (2005a), Kellens *et al*. (2007).



Source: van den Kommer and Keulemans, (1994).

Figure 5. Effect of pressure on separation efficiency (SE) in palm oil and palm olein fractionation.

Various studies in the past have demonstrated that separation efficiency or the amount of solid crystalline phase in the stearin cake depends on the level of filtration pressure applied. Increasing filtration pressure has a more pronounced effect on palm oil fractionation compared to palm olein fractionation, as illustrated in *Figure 5* (van den Kommer and Keulemans, 1994). The production of oleins (normal and superoleins) through single and double stage fractionations typically requires a filtration pressure of 15 bar, while higher pressures of about 30 bar are essential to produce cocoa butter equivalent products with qualities similar to those obtained by solvent fractionation (Kellens, 1996). It has also been reported that stearin cakes with entrainment levels reduced to 20% can be achieved with the application of filtration pressures of up to 50 bar (Willner, 1994; Hamm, 1995). Despite the substantial entrainment reduction, high filtration pressures may result in the passage of the stearin cake through the filter cloth. One way to overcome this problem is by applying a lower pressure on a thinner stearin cake. *Table 2* shows the effect of filter chamber width and squeezing pressure on separation efficiency in palm oil fractionation. Similar entrainment levels and olein yields obtained when using a 50 mm filter chamber at 30 bar squeezing pressure can also be achieved by employing a lower pressure of 15 bar on a 25 mm filter chamber (Kellens *et al.*, 2007).

The polymorphism and morphological aspects of crystals also play important roles in achieving good olein-stearin separation. Crystal spherulites in the *β'* form are easier to filter due to their uniform size and firmness, and have the tendency to remain suspended in the liquid oil due to the higher degree of stability compared to the

TABLE 2. EFFECT OF SQUEEZING PRESSURE AND FILTER CHAMBER WIDTH ON THE SEPARATION EFFICIENCY IN PALM OIL FRACTIONATION

Chamber plate width (mm)	Squeezing pressure (bar)	Liquid oil entrained in filter cake (%)	Yield (%)	
			Stearin	Olein
50	6	45	23.6	76.4
	15	39	20.0	80.0
	30	35	18.3	81.7
25	6	40	20.6	79.4
	15	34	18.8	81.2
	30	30	16.7	83.3

Source: Adapted from Kellens *et al*. (2007).

more metastable α-form crystals (Deffense and Tirtaux, 1989; Kellens *et al*., 2007). The use of additives such as polyglycerol fatty acid esters in palm oil crystallisation has been shown to produce smaller and even-sized crystals, leading to increased olein yields and entrainment reduction (Kuriyama *et al*., 2011; Saw *et al*., 2017; 2020). Moreover, in the case of palm olein fractionation, better separation efficiency and higher superolein yield can be attained when a small number of nuclei and similar crystal sizes exist during crystallisation (Deffense, 1998; Yoong *et al*., 2019). Seeding is often practiced on an industrial scale in palm olein fractionation to induce nucleation and generate uniform, dense and hard crystals with excellent filterability, which can withstand squeezing pressures of up to 30 bar (Deffense, 2009).

Table 3 tabulates the estimated entrainment levels in palm stearin from various fractionation processes employing different separation techniques to produce palm olein IV 56-57 over the last half century (Timms, 2005b). With the continuous developments and improvements in separation techniques over the years, the level of olein entrainment in stearin has seen a significant reduction from about 70% in the 1970s to 30% in the 2000s. The most effective separation process has been reported as solvent crystallisation in combination with belt filtration and washing. However, the process is too costly and is only used for producing high quality solid fractions for the confectionery industry. Currently, a combination of dry crystallisation and membrane filter press is capable of producing palm mid-fractions equivalent to those produced by solvent fractionation (Timms, 2005a).

CONCLUSION

The palm oil industry has seen significant advancements in the fractionation process over the last half century. Various research studies have demonstrated that a combination of dry crystallisation and membrane filter press with reduced chamber width and higher squeezing pressure is ideal in increasing olein yields and keeping entrainment to a minimum, rivalling that of solvent fractionation. Although solvent fractionation is still used in the production of specialty fractions required by confectionery manufacturers, current developments in fractionation would need to carefully consider evolving global consumer demands for safer and healthier ingredients that are free from chemicals and additives. As dry fractionation adopts a green technology approach, further enhancement on existing techniques could possibly look into optimising process conditions with

TABLE 3. ESTIMATED ENTRAINMENT IN PALM STEARIN FROM VARIOUS INDUSTRIAL FRACTIONATION PROCESSES EMPLOYING DIFFERENT SEPARATION TECHNIQUES

Year	Company	Process	Yield (%)	IV of stearin	Entrained olein (% of stearin)
Mid 1960s	Unilever	Continuous Acetone Tube Crystalliser + Belt Filter + washing with pure solvent	10-11	~8	~0
Mid 1970s	Bernardini	Batch Hexane Crystalliser + Drum Filter	37-40	44-46	70-73
Early 1980s	De Smet	Batch Dry Crystalliser + Drum Filter	37-40	45-47	70-73
Mid 1970s	Tirtaux	Batch Dry Crystalliser + Belt Filter	28-33	28-33	60-67
Mid 1970s	Alfa Laval	Batch Crystalliser + Detergent + Centrifugation (Lanza/Lipofrac Process)	17-23	25-35	35-52
Late 1980s	De Smet	Batch Dry Crystalliser + Membrane Filter Press	20-21	32-33	47-50
2000	De Smet	Batch Dry Crystalliser + Membrane Filter Press (50mm chamber width and 6 bar squeezing pressure)	~24	~40	~45
2000	De Smet	Batch Dry Crystalliser + Membrane Filter Press (25 mm chamber width and 30 bar squeezing pressure)	~17	~32	~30

Source: Timms (2005b).

the aim for higher separation efficiency and maximising entrainment reduction. Newer technologies in crystalliser and filtration designs can be explored to sustainably produce even higher quality and niche oleins and stearins down the multistage fractionation route, thereby ensuring increased value addition and functionality of palm oil and its fractions in the future.

REFERENCES

Bemer, G G and Smits, G (1981). Industrial Crystallization of Edible Fats: Levels of Liquid Occlusion in Crystal Agglomerates. *Proc. of the 2nd World Congress of Chemical Engineering, 1*: 369-371.

Calliauw, G; Gibon, V; De Greyt, W; Plees, L; Foubert, I and Dewettinck, K (2007). Phase composition during palm olein fractionation and its effect on soft PMF and superolein quality. *J. Am. Oil Chem. Soc.*, *84(9)*: 885-891.

Deffense, E (1985). Fractionation of palm oil. *J. Am. Oil Chem. Soc.*, *62(2)*: 376-385.

Deffense, E and Tirtiaux, A (1989). Fractionation: A fast growing technology, 80th AOCS Meeting, 3-7 May, Cincinnati, USA.

Deffense, E (1998). Dry fractionation and selectivity: Innovations technologiques. *OCL-Ol, Crops Gras, Li.*, *5(5)*: 391-395.

Deffense, E (2000). Dry fractionation technology in 2000: Fractionation of fats. *European J. Lipid Sc. Technol.*, *102(3)*: 234-236.

Deffense, E (2009). From organic chemistry to fat and oil chemistry, *OCL*, *16(1)*: 14-24.

Gupta, M (2017). Chapter 11 – Winterization and fractionation of selected vegetable oils. in *Practical Guide To Vegetable Oil Processing* Gupta M K AOCS 2nd ed., Press. P. 291-322.

Hamm, W (1986). Fractionation-With or without Solvent? *Fette, Wiss. Technol. 88(S1)*: 533-537.

Hamm, W (1995). Trends in edible oil fractionation. *Trends Food Sc. and Technol.*, *6(4)*: 121-126.

Hamm, W (2005). Entrainment: Are we making progress? In *Society of Chemical Industry (SCI) Oils and Fats Group Meeting on Fractionation-Current Status and Future Prospects in a Low-Trans World*. *Society for Chemical Industry - Oils and Fats Group Meeting.*, 22-23 November 2005, Ghent University, Ghent, Belgium.

Hishamuddin, E; Nagy, Z K and Stapley, A G F (2020). Thermodynamic analysis of the isothermal fractionation of palm oil using a novel method for entrainment correction. *J. Food Eng. 273*: 109806.

Kellens, M (1994). Developments in fat fractionation technology. (*Society of Chemical Industry Lecture Paper Series No. 0042*). Society of Chemical Industry, London, UK.

Kellens, M J (1996). Developments in fractionation technology: Use of high pressure membrane filter presses in dry fractionation. *PORIM International Palm Oil Congress. Competitiveness for the 21st Century (Chemistry and Technology),* 23-28 September, Kuala Lumpur, Malaysia.

Kellens, M; Gibon, V; Hendrix, M and De Greyt, W (2007). Palm oil fractionation. *European J. Lipid Sc. and Technol.*, *109(4)*: 336-349.

Kuriyama, J; Miyaji, Y; Tamura, K; Zaliha, O and Chong, C L (2011). Improved sustainable fractionation of palm oil using polyglycerol fatty acid esters. *J. Oil Palm Res.*, *23*: 1141-1145.

MPOB (2021). Production of selected processed palm oil. http://bepi.mpob.gov.my/index.php/en/production/production-2020/production-of-processed-palm-oil-2020.html, accessed on 10 February 2021.

Parveez, G K A; Hishamuddin, E; Loh, S K; Ong-Abdullah, M; Salleh, K M; Bidin, M N I Z; Sundram, S; Hasan, Z A A and Idris, Z (2020). Oil palm economic performance in Malaysia and R&D progress in 2019. *J. Oil Palm Res.*, *32(2)*: 159-190.

Saw, M H; Hishamuddin, E; Chong, C L; Yeoh, C B and Lim, W H (2017). Effect of polyglycerol esters additive on palm oil crystallization using focused beam reflectance measurement and differential scanning calorimetry. *Food Chem.*, *214*: 277-284.

Saw, M H; Hishamuddin, E; Fauzi, S H M; Yeoh, C B and Lim, W H (2020). Influence of polyglycerol ester additive on palm oil fractionation in relation to the crystal size distribution. *J. Oil Palm Res.*, *32(2)*: 303-312.

Timms, R E (1994). Principles of fat fractionation, Society of Chemical Industry (SCI) Oils and Fats Group Symposium on Fractional Crystallisation of Fats. London, United Kingdom March 1994. *SCI Lecture Paper Series,* Paper No. 0039.

Timms, R E (2005a). Fractional crystallisation-the fat modification process for the 21st century. *European J. Lipid Sc. and Technol.*, *107(1)*: 48-57.

Timms, R E (2005b). The history and current status of fractionation. Fractionation- Current Status and Future Prospects in a Low-*trans* World,. *Society for Chemical Industry - Oils and Fats Group Meeting*, 22-23 November 2005, Ghent University, Ghent, Belgium.

van den Kommer, M and Keulemans, C N M (1994). Developments in dry fractionation, (*Society for Chemical Industry Lecture Paper Series No. 0040*), Society for Chemical Industry, London, United Kingdom.

Willner, T (1994). High-pressure dry fractionation of fats, (*Society of Chemical Industry Lecture Paper Series No. 0038*), Society of Chemical Industry, London, United Kingdom.

Yoong, J H; Saw, M H; Yeoh, C B; Kanagaratnam, S; Abdullah, L C and Choong, S Y (2019). The effects of polyglycerol esters on palm olein fractionation. *J. Oil Palm Res.*, *31(2)*: 294-303.