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TRENDS IN  
FRACTIONATION PRACTICE  
FOR EDIBLE OILS

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## TRENDS IN FRACTIONATION PRACTICE

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## 1.0 General features of fractionation

Fractionation has possibly been in use for more years than almost any other edible oil processing technique due to its intrinsic simplicity - an oil can be allowed to cool to a temperature at which part of the oil crystallises, and since the density of the crystalline material is marginally greater than that of the liquid phase a separation, though very imprecise, can be obtained by allowing the heavier crystalline phase to settle.

Although the fractionation process has progressed since such early days it remains a basically simple process, and the conditions used can truly be described as mild. Due to this simplicity the process is also a relatively low-cost operation. Technically, on the other hand, the large number of triglycerides present in most oils and the characteristics of liquid-solid separation techniques mean that it is difficult to obtain a sharp separation by fractionation. A further disadvantage of the process is the formation of a secondary product - in the case of separation by filtration this is either the cake or the filtrate - the value of which can have an important effect on the economics of the process as a whole.

## 1.1 The oils fractionated

The dominant position of palm oil in the field of fractionation is too obvious to require much elaboration, with the amount of palm oil fractionated in S E Asia (Malaysia and Indonesia) far outstripping the overall quantity of oils fractionated elsewhere. Palm oil is also fractionated in other regions of the world where palm oil production has been developed in recent years, e.g. Central / South America. The production of the liquid fraction, palm oleine, which is used as an industrial frying oil in many parts of the world, is the objective of most palm oil fractionators, but the production of palm mid-fraction for use in the confectionery fats industry has for many years been a very profitable operation, until recently almost always carried out in solvent. The interest in producing palm mid-fraction by dry fractionation has grown appreciably in the last decade, and the eagerness of processors to be seen to be using mild, or even 'green', processes has been a significant incentive in this shift.

Palm kernel oil and anhydrous milkfat (AMF) are the other major oils and fats undergoing fractionation. Increasing quantities of palm kernel oil are fractionated in Malaysia for export to countries requiring confectionery fats, and palm kernel oil fractionation is also undertaken in various European countries.

The fractionation of anhydrous milkfat is undertaken almost exclusively in Europe, with a small quantity being produced in New Zealand.

Other oils and fats subjected to fractionation are tallow, fish oil and some partially hardened vegetable oils. The quantities of these oils processed by fractionation are believed to be limited.

Information on fractionation plant capacities available is far from complete, and the statistics on the movements of fractionated products between producer and consumer countries is not sufficiently specific to allow a detailed map of fractionation capacity and its utilisation to be constructed. However, the fact that most fractionation plants are supplied by only three suppliers of such plants makes it possible to estimate capacities installed in recent years. The information made available by equipment suppliers also facilitates monitoring of the geographical location of new installations.

Of fractionation capacity installed in the last three years more than 80% was installed in S E Asia and therefore used for the fractionation of palm oil. Furthermore, the average size of plant installed in S E Asia was in the range of 300 - 400 m.t. per day (TPD), with a maximum capacity of 700 TPD being noted, equivalent to an annual capacity of over 200 000 m.t. Plants installed elsewhere and used to fractionate other oils, including AMF, seldom exceed 100 TPD (Table 1).

**TABLE 1**

**FRACTIONATION PLANT INSTALLATIONS**

(as installed by major plant suppliers)

**CAPACITIES IN TPD**

		<u>1981 - 85</u>	<u>1991 - 93</u>
<i>PALM OIL</i>	S E ASIA	8495	7200
	C / S AMERICA	796	905
	OTHER	410	180
REGIONS			
<i>ANHYDROUS MILK FAT</i>	EUROPE	354	120
OTHERS	WORLDWIDE	634	200

LARGEST PLANT INSTALLED : 700 TPD  
(INDONESIA, 1992)

NOTE 1000 TPD = 300 000 M.T./A.

## 2. Trends in technology

### 2.1 Historical

Even before the explosive growth of S E Asian palm oil production began in the 1970's the demand for a relatively low-cost frying oil provided a clear incentive for upgrading the technology of palm oil fractionation and this led, on the one hand, to the use of the rotary vacuum filter for the liquid-solid separation and, on the other, to the application of the concept first suggested by Lanza for facilitating separation of the crystallised fat from the liquid by transferring it to an aqueous phase using a surfactant. In the mid-1950's fractionation of palm oil in acetone solution had also been developed for the production of a mid-fraction suitable for use in cocoabutter equivalents. For two decades from the mid-1950's fractionation of oils therefore comprised three basic processes - dry fractionation using rotary vacuum filtration, Lanza (or Lipofrac) fractionation and solvent fractionation. Acetone was the solvent commonly used in the latter process, but 2-nitropropane was used in at least one plant and hexane was extensively studied as a fractionation solvent in view of the lower cost of solvent recovery. Aqueous isopropanol was also recommended as a solvent for this process.

The principal advantages of the Lanza/Lipofrac process are the improved separation when compared with dry fractionation using a rotary vacuum filter as well as the ability to accelerate crystallisation in view of the fact that the formation of large crystals is not required. The relatively large density difference between the aqueous phase containing the suspended crystallised material and the oleine makes it possible to use disk centrifuges for the phase separation. The process has the disadvantage of incurring additional processing costs in the form of wetting agents and, more recently, generating an effluent problem.

Solvent fractionation obviously gives a sharper solid-liquid partition, with reduced viscosity and the possibility of washing the cake additionally facilitating better separation when filtering. This is of particular importance when the process is operated for the purpose of producing a solid fraction with minimum oleine entrainment. This advantage is however bought at a considerable cost, both in terms of capital investment but also in operating cost. Moreover, recent trends towards more 'consumer-friendly' processes in the food industry have generated pressures to move away from processes requiring solvents such as acetone and hexane.

## 2.2 Dry fractionation

### 2.2.1 Crystallisation

The various efforts to persuade the industry to adopt continuous crystallisation have been successful in the case of Lanza/Lipofrac crystallisation, where both high-speed (Votator) and low-speed scraped surface heat exchangers have been successfully applied to give a continuous process, as well as in solvent-based fractionation. Dry fractionation, on the other hand, continues to use batchwise crystallisation. The design of crystallisers for dry fractionation has very much concentrated on the need to control the polymorphic changes experienced by fat crystals and at the same time to minimise the formation of secondary nuclei, as these have an adverse effect on filter performance. This has led to the use of very slow rates of crystallisation, applying extremely low temperature differentials between coolant and oil. This obviously results in lengthy crystallisation times. Control systems for batch crystallisers have become far more sophisticated but the industry still appears to be divided over the best method of controlling the fat crystallisation process.

Although a number of equipment suppliers have indicated an ability to supply continuous crystallisation plant for dry fractionation of edible oils there is no evidence of any such plant actually being in operation. It is however clear from another contribution to this meeting<sup>1</sup> that further work on this topic is in progress.

### 2.2.2 Separation

The first step in the modernisation of the filtration of crystallised fat slurries saw the introduction of the rotary vacuum filter - a low-cost device capable of handling the quantities being fractionated in the 1960's. However, the relatively low pressure differential and the short time available for separating liquid from solid on the drum meant that entrainment of liquid oil in the cake was high, with palm oleine yields not exceeding 65 % when fractionating to produce an oleine having a Cloud Point of approximately 10 °C. Entrainment of the liquid fraction in the cake was estimated to be in the range 65 - 77 %<sup>2</sup>. The introduction of the Florentine stainless steel endless belt filter by Fractionnement Tirtiaux improved the separation by allowing more time for liquid to be drained from the cake, but even then the oleine residue in the cake was substantial, with the cake from palm oil fractionation estimated to contain 55 - 60 % liquid, i.e. oleine. Although separation by filtration must obviously lead to some loss of liquid fraction in the filter cake the liquid : solid ratio of the cake produced left considerable room for improved separation.

Bemer and Smits<sup>3</sup> showed that entrainment consists of oil occluded within the crystallised agglomerates as well as oil trapped between the agglomerates, and showed that by crystallising the oil in a carefully controlled liquid flow system it is possible to avoid oil occlusion.

The switch from vacuum to pressure filtration in the form of the membrane filter press in the mid-1980's provided the next milestone in the development of the filtration step in fractionation, and made it possible to increase the yield of oleine in palm oil fractionation further to near 80%. Although the increased yields obtained by the introduction of the membrane filter press are significant, it must be remembered that the true saturated triglyceride content of a palm oil fractionated for the production of oleine is less than 10%, which means that even when using a membrane filter press the filter cake produced contains substantial proportions of liquid fraction. Wesdorp<sup>4</sup> has reported that when fractionating palm oil at 29°C and filtering the resulting slurry at 12 bar the saturated triglycerides content of the filter cake is only 45 %.

Results on AMF fractionation using a membrane filter press show a trend towards lower solids contents in the filter cake at lower fractionation temperatures, solids content (by NMR) being 55 - 60% when fractionating at 20°C or above but only 40 - 45% when fractionating in the temperature range 7 - 10°C. The data in Table 2 were obtained in a wide-ranging trial using various operating conditions and different process systems.

**TABLE 2**

#### ENTRAINMENT IN AMF FRACTIONATION AND FILTRATION

(Based on Solid Fat Content measurements at filtration temperature)

at 20 °C and above	SFC = 55 - 65 % (membrane filter) Entrainment = 35 - 45% SFC = 50 - 55% (vacuum filter) Entrainment = 45 - 50%
at 15 °C	SFC = 55 % (membrane filter) Entrainment = 45 % SFC = 45 % (vacuum filter) Entrainment = 55 %
at 5 - 10°C	SFC = 40 - 45% Entrainment = 55 - 60% (membrane and vacuum filter)

The membrane filter press used in the fractionation of edible oils such as palm oil and anhydrous milkfat has normally been operated at a pressure of approximately 6 bar, although in other applications this type of press has been operated at pressures of up to 16 bar. The question whether use of higher press pressures can lead to further reduction of oleine entrainment in the filter cake is in part answered by the development of the Hydrofilter press by Krupp, where pressures of up to 50 bar can be applied to the cake. (It should be noted that other filter suppliers also offer high-pressure filtration but it appears that their filters have not to date been tested for the filtration of fat slurries.)

Willner<sup>5</sup> has reported that whereas an increase in filter squeeze pressure from 6 to 50 bar reduces the liquid fraction content of the cake from approximately 60 to approximately 47% of the total cake in the case of palm oil fractionation, in the case of palm kernel oil the same change in squeeze pressure reduces oleine entrainment from approximately 45 % to approximately 20 % of the cake.

These observations demonstrate that filtration at higher pressure can offer benefits where it is important to produce a cake containing the lowest possible amount of liquid fraction. The observations also suggest that the liquid retention of the cake is a function not only of the pressure applied but also of the oil being fractionated. Furthermore, the work by Bemer and Smits referred to earlier (ref. 2) shows that the fluid regime in the crystallisation stage can play a considerable role in facilitating entrainment reduction.

The presses suitable for operation at pressures above 16 bar are significantly more costly than the membrane press in use in edible fat fractionation at present [which is already the largest single item in the capital cost of the plant and can be expected to account for more than 30 % of the total capital cost]. The use of high-pressure membrane presses is therefore only justified when the alternative route to low liquid entrainment in the cake involves using a substantially more costly process, e.g. solvent fractionation.

### 2.2.3 Economics of dry fractionation

The capital cost of a dry fractionation plant will be related to the scale of the operation as well as to the type of fat being fractionated, with stainless steel crystallisers being used for the fractionation of anhydrous milkfat. Experience has shown that for a processing capacity of 40 000 - 60 000 m.t. oil per annum the capital cost of a plant using the membrane filter press principle (for the upper end of the range it is likely that two presses would be required) would be equivalent to approximately US \$ 20 / m.t. capacity / annum (1993 estimates) when using mild steel crystallisers, or up to 10% higher when using stainless steel for the crystallisers. Use of a vacuum

filter, e.g. the Tirtiaux Florentine filter, in place of the membrane filter should, on the other hand, reduce the total capital cost by approximately 10 %, but would also lower the yield of liquid fraction. Obviously a plant designed for a lower throughput, as is likely to be the case when fractionating anhydrous milkfat, would show a higher capital cost per unit of throughput.

The operating costs of a fractionation plant are relatively low, with utilities accounting for less than US \$ 5 per m.t. Allocation of labour costs is largely a matter of operating company policy.

## 3. An alternative to fractional crystallisation ?

### 3.1 Principles of supercritical fluid extraction

Extraction with supercritical fluids has been explored as an alternative to fractional crystallisation in the case of edible fats. Since supercritical fluid extraction relies on solubility in the fluid phase rather than melting point for separating ability it is unlikely that the fractions produced will exactly match those obtained by crystallisation. The particular merit of the use of this separation process is the avoidance of the problem of entraining liquid in the cake, which, as has been shown in the preceding sections, is particularly relevant when fractionation is used to produce a high-purity cake.

A considerable volume of work has been carried out and published on equilibrium data and separations achieved in various supercritical fluid extraction processes involving edible fats, including seed extraction, deacidification and, in recent years, triglyceride partitioning. Although these studies have included a number of solvents as well as mixtures of solvents and entrainers, the solvent most commonly investigated has been carbon dioxide. Carbon dioxide has a number of advantages, both from a technological as well as a product safety point of view. It is also a relatively low-cost solvent, although recovery of the solvent after processing remains essential. The principal disadvantage of carbon dioxide is its low solubility for triglycerides, and in this respect propane is distinctly superior to carbon dioxide. The solubility of triglycerides in carbon dioxide is primarily a function of gas density and therefore both pressure and temperature can be used as process parameters in the extraction and in the subsequent fractional separation.

Whereas in fractional crystallisation separation depends basically on a difference in melting point of the different triglycerides separation in supercritical fluid extraction is primarily related to molecular size and polarity. In the case of triglycerides the carbon number is the most important characteristic governing the separation effect of solubilities for triglycerides of different chain lengths at 40°C, but pressure also has an important bearing on the separation<sup>6</sup>. The solubility gradient as a function of pressure is

obviously an important criterion in choosing conditions for the extraction and the subsequent separation stage.

It has been shown<sup>7,8</sup> that continuous fractional separation of anhydrous milkfat is feasible, using a conventional packed column as the fluid-liquid contactor and separators operated at different pressures to produce fractions having the required composition (Fig. 1). Results on batch fractionation of anhydrous milkfat have also been published<sup>9</sup>, and the mathematical modelling of the process for anhydrous milkfat has been investigated<sup>10</sup>.

The triglyceride distribution of anhydrous milkfat is complex, with peaks at C38 and C50. The latter peak principally reflects the presence of mono-unsaturated triglycerides containing oleic acid. The work of Kankare and Antila<sup>9</sup> shows that these mono-unsaturated triglycerides tend to be retained in the extraction residue, and that the second peak in the triglyceride distribution can therefore be eliminated, particularly when extracting at the lower end of the range of extraction pressures used. Enrichment of the extract fractions in triglycerides containing short-chain fatty acids, which are predominantly saturated, is also demonstrated in this work. The range of triglycerides present in the fat is however too widespread in terms of carbon number to enable sharply delineated fractions to be produced.

Buening-Pfaue and colleagues<sup>8</sup> compared the fractions obtained by extraction with supercritical carbon dioxide with those obtained by fractional crystallisation. Their work highlighted the fact that in the extraction process the triglycerides containing oleic acid are selectively retained in the non-volatile fraction (which is at the same time depleted in short-chain fatty acids) whereas in fractional crystallisation the oleic acid-rich triglycerides are enriched in the liquid fraction. A direct comparison of the processes from the point of view of fractions produced is therefore difficult.

The selectivity of the extraction is seen more clearly in the case of palm kernel *stearine* extraction with supercritical carbon dioxide, as shown in Fig. 2<sup>11</sup>. Here the reduction in the content of the longer-chain triglycerides is much more marked, and an estimate of the partitioning effect further emphasises the separation achieved, as can be seen in the following table.

TABLE 3

# SUPERCRITICAL FLUID EXTRACTION

## Enrichment in short-chain triglycerides

Composition in terms of Triglyceride Carbon Numbers  
(expressed as Ratio C40 and below : C42 and above)

	FEED	EXTRACT	RESIDUE
AMF	0.857	1.487	0.472
PALM KERNEL STEARINE	3.18	15.12	n.a.
<u>SELECTIVITY</u> (EXTRACT / FEED C40 - / C42 +)			
AMF		32	
PALM KERNEL STEARINE		4.75	

Data for above estimates from Refs. 7 and 9

In the case of the extraction of palm kernel *stearine* extraction with supercritical carbon dioxide has greatly enhanced the short-chain triglyceride content of the extract, almost certainly resulting in a significant reduction in the content of mono-unsaturated fatty acids (principally oleic acid) in the extract.

It therefore appears that extraction with supercritical carbon dioxide offers an interesting alternative process to the conventional fractional crystallisation, at least in those cases where it is desirable to effect a separation based on chain length. Moreover, since the unsaturation present is often due to the presence of the C<sub>18</sub> fatty acids supercritical fluid extraction also offers the possibility of producing a fraction with reduced levels of unsaturation. The degree of fractionation will of course depend on the process conditions used, i.e. temperature, pressure and gas flow rate, as well as on the number of contacting stages available in a continuous contactor. The comparison of fraction compositions using the two processes highlights the fact that the user should not expect to produce fractions of identical composition.

Entrainment in supercritical fluid extraction is controlled by avoiding fluid phase densities, i.e. pressures, approaching that of the liquid phase and

maintaining fluid phase linear velocities in the extractor consistent with design criteria for gas-liquid contactors.

### 3.2 Economics of supercritical fluid extraction

The recent paper by Chidambara Raja and colleagues<sup>6</sup> provides a basis for comparing the cost of fractionation by supercritical fluid extraction with fractionation by crystallisation. The authors, who studied and reported on the extraction of anhydrous milkfat using carbon dioxide at 240 bar and 40°C, estimated the cost of installing and operating a plant for a maximum throughput of 800 m.t. per annum. Their calculations show that the carbon dioxide compressor, which is required to handle almost 50 kg. carbon dioxide per kg. AMF, is responsible for the largest single contribution to the capital cost estimate. When their cost estimates are adjusted to facilitate comparison with a single-stage fractional crystallisation, the capital cost of an extraction plant capable of processing 12 000 m.t./annum AMF appears to be greater than that of a comparable fractional crystallisation plant by a factor of approximately 3.

The operating costs for the extraction plant (not including labour costs) are also far higher than those commonly quoted for fractional crystallisation, the reason for this being the cost of carbon dioxide compression and circulation. Power consumption for the process is reported to be 3 MJ/kg. AMF. This compares with power consumption in the case of fractional crystallisation of slightly under 0.1 MJ/kg. AMF, and constitutes a major charge against the process and consequently an important disadvantage in any comparison of operating costs. Thermal energy requirements for the two processes being compared are likely to be modest in both cases.

Useful discussions of various aspects of the process engineering of extraction with supercritical fluids, including process economics, are given in<sup>12</sup>.

**TABLE 4**

#### COST COMPARISON : AMF FRACTIONATION (Single-stage)

	<u>FRACTIONAL CRYSTALLISATION</u>	<u>CARBON DIOXIDE EXTRACTION</u>
Capacity (m.t./a.) of plant costed	12 000	800
Capital Cost, '000 US \$	510	560
Equivalent capacity Capital Cost '000 US \$ (1993)	600	2 000

#### OPERATING COST / M.T.

Power, kWh	25	750
Steam, kg	80	< 100
Carbon dioxide, kg (make-up)	not used	155
LABOUR, men per shift	2	2

#### OPERATING CONDITIONS

Temperature, °C	28	40/80
Pressure, bar		247
Solvent Ratio, kg CO <sub>2</sub> / kg fat	not applicable	46

SOURCES    Supercritical fluid extraction :  
              adapted from  
              Trans. I ChemE , 71 (Part C), 3 (1993)

Fractional crystallisation  
1988 quotation updated

#### 4.0 Conclusions

Fractionation (fractional crystallisation) has grown considerably in importance in edible oil processing in the last quarter century, largely as a result of the growth in palm oil availability. The fractionation of anhydrous milkfat has also become an important application of the process. The continuing growth in palm oil production and the consumer-friendly nature of the dry fractionation process suggest good prospects for further expansion of process applications.

Despite the scale of palm oil fractionation continuous crystallisation has not made any inroads into the process technology used. Work on continuous countercurrent crystallisation of fats is now in progress, and is the subject of a contribution to the present set of papers.

Entrainment of liquid fraction in the filter cake remains a drawback in fractional crystallisation, thus setting limits to cake purity. This is especially important where the cake is the principal fraction produced. The development of high-pressure filtration, involving pressures up to 50 bar, has helped to reduce entrainment further.

Extraction of fats with supercritical carbon dioxide has been shown to produce a significant fractionation, with triglycerides of lower Carbon Number being preferentially extracted. The concept has attracted considerable attention, particularly amongst those working in the field of milkfat fractionation, but the most significant selectivities have been found when fractionating palm kernel fat using this process. Present indications are that this type of process is likely to be significantly more costly to instal and operate than conventional fractionation.

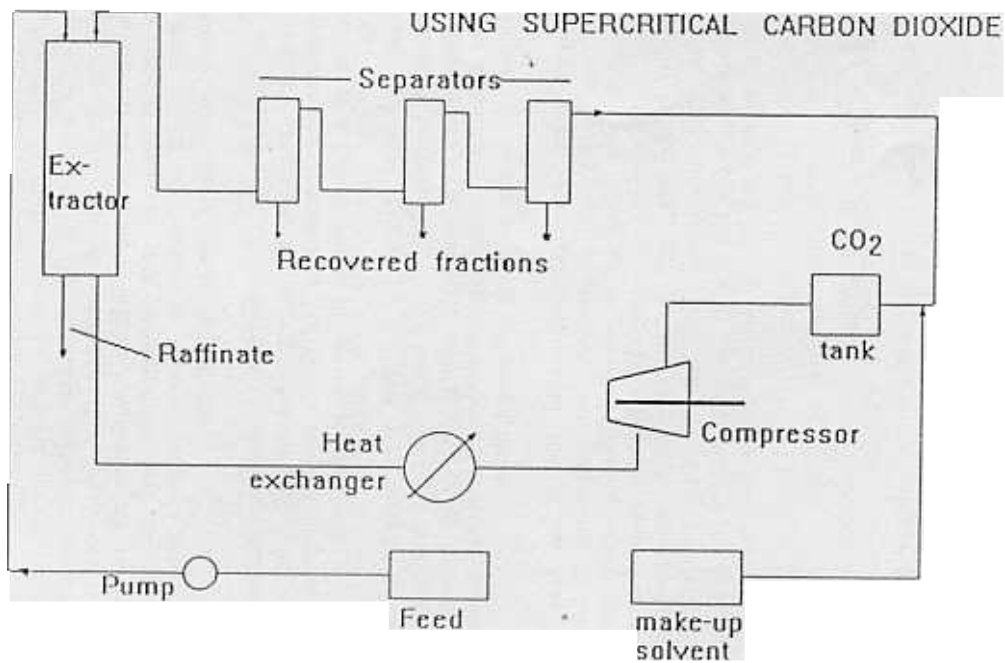
#### *Note added in preparation for publication*

It has been pointed out to the author that if the pressure conditions in the separator can be chosen so as to permit condensation of *liquid* carbon dioxide after separation of the extracted fraction(s) the capital- and energy-intensive recompression of gaseous carbon dioxide can be substantially reduced. If this were shown to be feasible the capital and operating costs of the extraction process would be reduced dramatically.

#### REFERENCES

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- 2 In this context entrainment is defined as % liquid fraction in the cake as recovered from the filter. The values are obtained by calculation using the filtrate and cake compositions. Alternatively an approximation to entrainment can be obtained by noting the Solid Fat Content at the filtration temperature (see Table 2).
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**FIG. 1** EXTRACTION OF ANHYDROUS MILKFAT



*Based on : Figure 1, Ref. 6*

**FIGURE 2** SUPERCRITICAL FLUID EXTRACTION OF PALM KERNEL FAT

