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# Fish Oil Powder Ingredient

(Nutritional studies on dried functional food ingredients containing *omega-3* polyunsaturated fatty-acids.)

Drs. P.M. Kelly and M.K. Keogh

*A fish oil powder ingredient, with low off-flavour and extended shelf life was successfully manufactured using microencapsulation, and was incorporated into a number of selected food products at a level that would satisfy the recommended daily intake for omega-3 polyunsaturated fatty acids.*

Teagasc
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# Nutritional Studies on Dried Functional Food Ingredients containing *omega-3* Polyunsaturated Fatty-Acids.

(*Fish Oil Powder Ingredient*)

ARMIS No. 4340

*Project Team:*

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## Summary and Conclusions

*The nutritional benefits of fish oils are generally attributed to their content of long chain omega-3 polyunsaturated fatty acids (PUFAs). Diets rich in these fatty acids are known to reduce the risk of coronary thrombosis, and are recommended to those who are susceptible to atherosclerosis. In addition, some of these long chain PUFAs play an important role in early infant nutrition, in the development of vital human organs such as the neural tube.*

*However, practical difficulties arise in achieving an adequate daily intake of fish oils to obtain these physiological benefits. Per capita fish consumption is low in many countries, especially of oily fish with high levels of omega-3 PUFAs. Fish oil, while available as a dietary supplement, is not universally appealing in that form. Attempts to incorporate fish oil into food formulations have had limited success mainly because of 'fishy' flavours coming through in the consumer products. Fish oil is particularly susceptible to oxidation, which results in fishy, painty and metallic flavours.*

*Hence the main aim of this study was the development of a dried ingredient in which the formulation and related processing conditions were optimised to protect the fish oil from oxidation. Protection of any sensitive oil may be achieved by means of microencapsulation, whereby oil is dispersed as very fine droplets in emulsions. During subsequent spray drying the droplets are effectively sealed inside a protective coating of protein surrounded by carbohydrate.*

*The objective was, therefore, to evaluate microencapsulation as a means of extending the shelf-life of fish oil in powder form thus increasing its versatility as a nutritional ingredient in food formulations.*

## Main Conclusions and Achievements

** A fish oil powder with acceptable taste and modest shelf-life expectation (31 weeks at 4°C) was successfully produced and incorporated into a number of food products, including infant formulae, at levels to satisfy the recommended daily intake of omega-3 polyunsaturated acids. The powder was produced by microencapsulating fish oil with dairy ingredients using homogenisation and spray drying.*

** One of the more important determinants of shelf-life of microencapsulated fish oil powders was the level of entrapped air incorporated during the drying process, which in turn was critically dependent on the choice of encapsulating protein used.*

*In this study, the vacuole volume of the powder, which is indicative of occluded air, was reduced to one-third (21 to 7 ml/100 g powder) when micellar casein, in the form of skim milk powder (SMP) was used instead of sodium caseinate as an encapsulating agent.*

** Using the SMP formulation, the shelf-life of the powder was increased by increasing homogenisation pressure. However, increasing homogenisation pressure did not affect the shelf-life of the powder produced from the sodium caseinate formulation.*

# Research and Results

## Aims and Objectives

The overall objective was to develop a powder-based delivery system for oxidation-sensitive oils with unique nutritional properties such as fish oils rich in omega-3 polyunsaturated fatty acids (PUFAs) in order to increase their versatility for use as food ingredients.

A specific objective was to optimise the process of microencapsulation involving homogenisation and spray drying as a means of imparting greater shelf-life stability to dry powder ingredients containing fish oil.

In a final phase, a number of food models (bread, biscuits and infant formulae) were used to evaluate the performance of the selected microencapsulated fish oil powders developed during the course of the work.

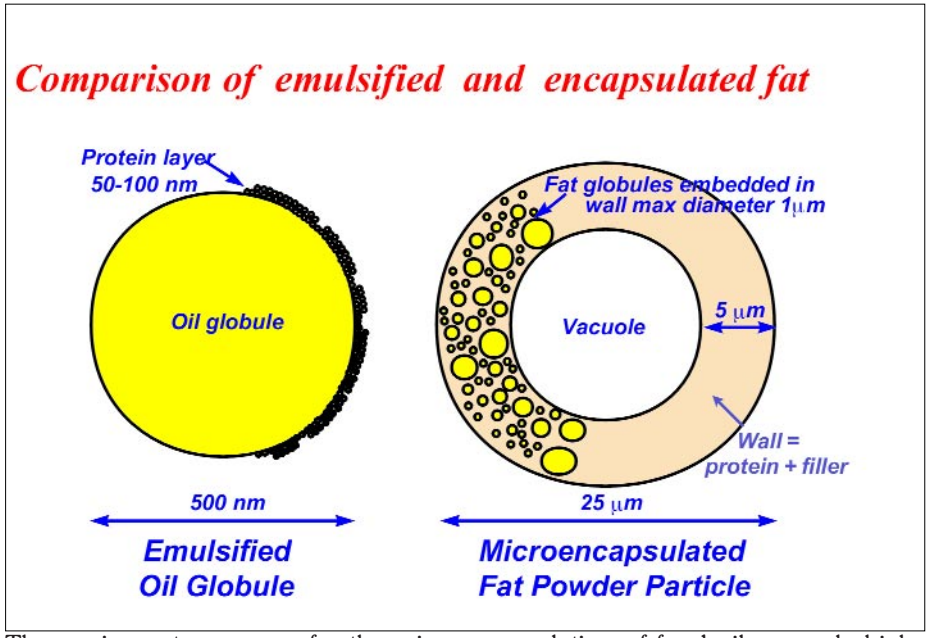
## Assessment of Sensory Properties

The detection of fish oil oxidation was readily monitored by sensory analysis. In conjunction with a trained taste panel, novel sensing technology based on the use of the so-called 'electronic nose' *(eNose)* was also evaluated. Chemical analysis of oxidised monomers and polymers by one of the project's partners and of volatiles by a second partner complemented the work of the sensory panel and helped with the interpretation of the results.

## Microencapsulation

Typically, the microencapsulation process involves dispersal and fixing of micro-sized oil globules < 1 $\mu$m in a solid particle matrix of typically 25 $\mu$m diameter.






The equipment necessary for the microencapsulation of food oils, namely high-pressure homogenisation for dispersing oil globules and spray-drying for fixation of the globules is readily available in the dairy industry.

A test of microencapsulation efficiency is the extent to which the solid matrix or wall material of the powder particles protects the microparticles or core material from deterioration before release on dissolution. Any microparticle can be encapsulated, but with oil as core material, the wall material must be an effective emulsifier and be capable of dehydration. To date, the main microencapsulating agents used are gum arabic, modified (succinylated) starches or milk proteins. All have good emulsification and dehydration properties.

The general hypothesis supporting the use of microencapsulation as a technology is that the smaller the oil globule size (achieved by homogenisation of an emulsion), the lower the amount of free oil that forms on the surface of each powder particle, the lower the amount of oil exposed to air (i.e. oxygen) and, therefore, the lower the rate of oxidation of the powder during storage.

It is felt that the coverage of surface fat on powder particles is a critical parameter of the oxidative stability of dried powder more so than traditionally used, solvent extractable 'free fat'. It is suggested that surface fat could derive from instability to coalescence over time. Coalescence instability in emulsions is assumed to increase the diffusion of fat onto powder particle surfaces during drying. Thus, fat

globule diameter and size stability over time may be of fundamental importance as indicators of microencapsulation efficiency.

## Preparation of Emulsions

Fish oils were prepared and refined by the Danish Institute for Fisheries Research (Lyngby, Denmark) from sand eel (Ammodytes species) and stored at -18°C on delivery. Prior to processing, fish oil was warmed from -18°C to 4°C overnight, and then to 25°C in a water-bath at 40°C until the oil became clear before mixing into the aqueous suspension at 20 - 25°C.

The aqueous phase was made by adding caseinate and lactose or skim milk powder (SMP) to de-ionised water at 50 - 60°C, pre-adjusted to give a final solution pH of 7.0 ± 0.1. The ratio of ingredients used to prepare a standard emulsion composition was 10: 10: 10: 70 in respect of fish oil: caseinate: lactose: water. All ingredients were stirred using a laboratory mixer/emulsifier at the lowest speed of 200 rpm (Silverson Mixer Model AXR, Silverson Machines Ltd, Chesham, UK), and the coarse emulsions were then homogenised at 20 - 25°C at a pre-selected single-stage pressure (150 - 500 MPa) and number of passes (1 - 5) in a Gaulin Mini-Lab homogeniser (APV, Silkeborg, Denmark, 60 kg/h).

## Spray Drying

A pilot-scale dryer (Anhydro F1 Lab dryer, Copenhagen, Denmark) using a 2-fluid nozzle was used. Inlet and outlet air temperatures were 177° ± 2°C and 75° ± 2°C, respectively. The powders were vacuum-packed, sealed in aluminium bags and temporarily stored at -18°C before being subjected together to storage assessment.

# Results

Initial trials indicated that a microencapsulated ingredient with only poor oxidative stability could be made. It was felt that the poor stability may have been due in part to:

- *the absence of an antioxidant in the fish oil,*
- *a high level of unencapsulated fat on the surface of the powder particles and*
- *the exposure of particles to oxygen during spray drying, and from the air present in the powder vacuoles.*



Because of surface chemistry studies being undertaken by one of the project's partners, an antioxidant was not included in the fish oil during the initial trials in order to exclude any effects on the properties of the powder particles caused by surfactant properties associated with certain antioxidant blends.

It was not possible to explain the unexpectedly high level of unencapsulated fat on the surface of the powder particles made with sodium caseinate. This was all the more perplexing given that lower surface fat values had been obtained previously using vegetable oils rather than fish oil on a laboratory scale drier by one of the project's partners.

In the absence of a more plausible explanation, it was felt that the much larger Anhydro spray drier now being used in the trials may be generating a different type of particle structure with more fat on the particle surface. Mechanical damage to the particles, either in the drier or during the packaging process may also be a possibility. A potential contribution to this physico-chemical defect due to variation in quality of sodium caseinate was also checked out and eliminated.

Alternative encapsulating agents to sodium caseinate/lactose were also evaluated. These materials are widely used for microencapsulating a range of highly unstable components such as flavouring agents. However, the results of preliminary trials were not encouraging and this aspect was discontinued. The sodium caseinate powders were significantly ($p<0.05$) less oxidised and the powder protein level significantly ($p<0.05$) reduced off-flavour at 3 weeks' storage. The lowest off-flavour was obtained at 7% fat, 6% lactose and 7% powder protein. Other participants showed using chemical tests that the shelf-life of these powders ***without*** antioxidants was only 0 - 2 weeks.

## Olfactory Analysis

The use of the *eNose* sensor for detecting differences between fish oil powders over time was a partial success. The interpretation of the differences was hindered by the absence of a standard fish oil powder, the changing composition of the fish oil wall material and the possibility of the antioxidant contributing to the array of volatiles present. Further refinement of the sample preparation may be necessary in order to reduce interpretation problems.

*Multiple Discriminant Analysis* of the *eNose* data from the powders could differentiate between samples on an age basis. There were significant differences between relatively fresh powders and aged powders. Using these results from the

*eNose*, it could be deduced that the volatiles characterised over time from the same powder were different and using the sensory and chemical analysis carried out in parallel, it may be concluded that these differences were due to oxidation.

This experiment was difficult to interpret using volatile analysis by *eNose*, due to the variation in powder composition and the likelihood of volatile species changing on ageing. A comparison between whey protein-stabilised powders and sodium caseinate-stabilised powders indicated that there were differences between these two groups of powders, especially in powders after 2 - 6 weeks storage.

Differences were not apparent between the powders after 10 weeks. The main problem was the absence of a true reference sample which could be defined as good and available for comparison purposes throughout. While the difference between powders using different proteins could be detected, it was not possible to determine their relative degree of oxidation.

## Antioxidant Effects

Two antioxidants were screened: (i) ALT 1 - a patented natural antioxidant combination containing tocopherol, ascorbyl palmitate, with lecithin as carrier prepared by the Danish Fisheries Laboratory providing the oil; and (ii) GRINDOX 117 - a commercially-sourced synthetic antioxidant containing propyl gallate. The antioxidants were added to the fish oils by the Danish Laboratory before shipment. The effect of drying fish oil emulsions using nitrogen ($N_2$) gas pressurisation of a 2-fluid atomiser nozzle as well as its use as a drying medium, $N_2$ + ALT 1 and $N_2$ + GRINDOX 117 on the oxidative stability of fish oil powders stored at 16°C was compared to drying under air.

The air and $N_2$-dried powders had a shelf-life of 4 - 6 weeks. The natural antioxidant ALT 1 extended the shelf-life marginally to 4 - 8 weeks but the synthetic antioxidant GRINDOX 117 further extended the shelf-life to 6 - 8 weeks. However, for reasons relating to the nutritional aspects of the study, a decision was made in favour of an all-natural antioxidant (ALT 1) as formulated by the Danish partner.

All subsequent experiments were conducted on fish oils containing ALT 1.



## Encapsulant Selection

A limited number of non-protein encapsulants were also evaluated. Hydrocolloids such as ***N-Lok*** and ***acacia gum*** did not perform as well as sodium caseinate/lactose in terms of emulsion particle size reduction. In fact, ***β-cyclodextrins*** presented the additional difficulty of not being able to homogenise the emulsions due to excessively high viscosity. As a result, the free fat values of powders made using the latter were very high (33 - 38%), while those of the controls were under 1%. Furthermore, the ***β-cyclodexdrin***-encapsulated powders formed a sediment on reconstitution.

## Homogeniser Type, Particle Size and Surface Fat

Performance comparisons were carried out on 4 different homogenisers, operated at 45 MPa x 4 passes, in order to identify the most effective type for reduction of emulsion particle size. According to the D(v,0.5) and D(v,0.9) indices, the large Niro homogeniser performed better than the Gaulin model with values of 0.39 and 0.51 $\mu$m, respectively. However, the benefit in terms of reduced surface fat content in the subsequent powders was only marginal, 39% compared to 43%, respectively.

## Number of Homogenisation Passes

The effect of up to 10 homogenisation passes at maximum pressure (65 MPa, Gaulin homogeniser) was compared using concentrates at either 30% (standard formulation) or 13% total solids. No benefit in terms of globule size reduction was gained by exceeding 5 passes, and there was only marginal improvement at the higher solids concentration. Increasing homogenisation pressure to 65 MPa from 45 MPa appeared to be more effective in producing emulsions of finer particle size.

It was, therefore, decided to produce a series of fish oil powders, containing the antioxidant ALT 1, with different compositions under various homogenisation conditions in order to obtain reduced surface fat and oxidation rates. In the same experiment surface fat was increased by spraying onto the dried powder to test an hypothesis put forward by the Spanish partner (CSIC) that the rate of oxidation was not necessarily related to the level of surface fat and could be determined by conditions in the interior of the powder particles.

## Formulation Effects

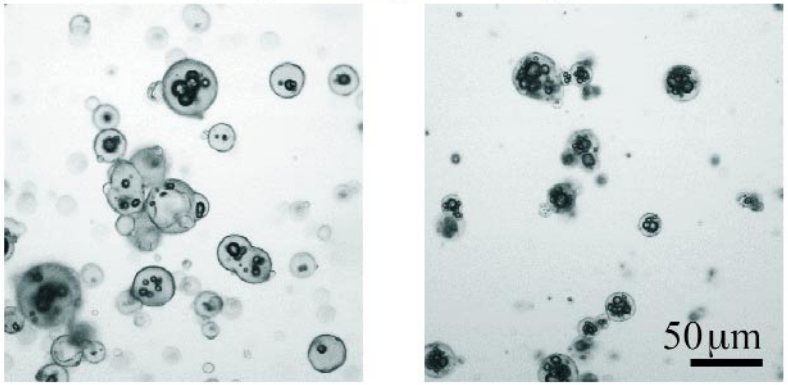
Three levels of each of the composition variables fat, protein and lactose were set out, namely 6, 10 and 14%. However, maximum protein content in the emulsion was restricted to 12% due to the viscosity limitations. A high fat - high free fat powder was also made together with a fish oil powder where 1% of the fish oil was sprayed on to the surface of the powder using surface coating equipment. The homogenisation conditions used were 5 - 65 MPa x 5 passes with the exception of a negative control where 45 MPa x 4 passes was used.

Differences in emulsion oil globule diameter due to variations in the protein:lactose ratios were not significant. The free fat levels of the high fat - high free fat and 1% fish oil sprayed-on powders were higher, as expected.

Altering the protein:lactose ratio of the emulsions did not affect the surface fat significantly, but surface fat values of the low fat (18%) powders were lower (by 12.8%), while those of the high fat (42%) powders were higher (by 8.1%), as expected.

**Transmitted laser light micrographs of low and high vacuole volume powders**

*Vacuoles are shown as the dark circles within the powder particle spheres*




*Low vacuole volume using SMP*

*High vacuole volume using Na caseinate*



The reconstituted emulsion oil globule diameter increased by about 0.03 µm typically, but the increase ranged from 0.01 for the low fat powder to 0.09 µm for the powder homogenised at the lowest pressure and the sprayed on sample.

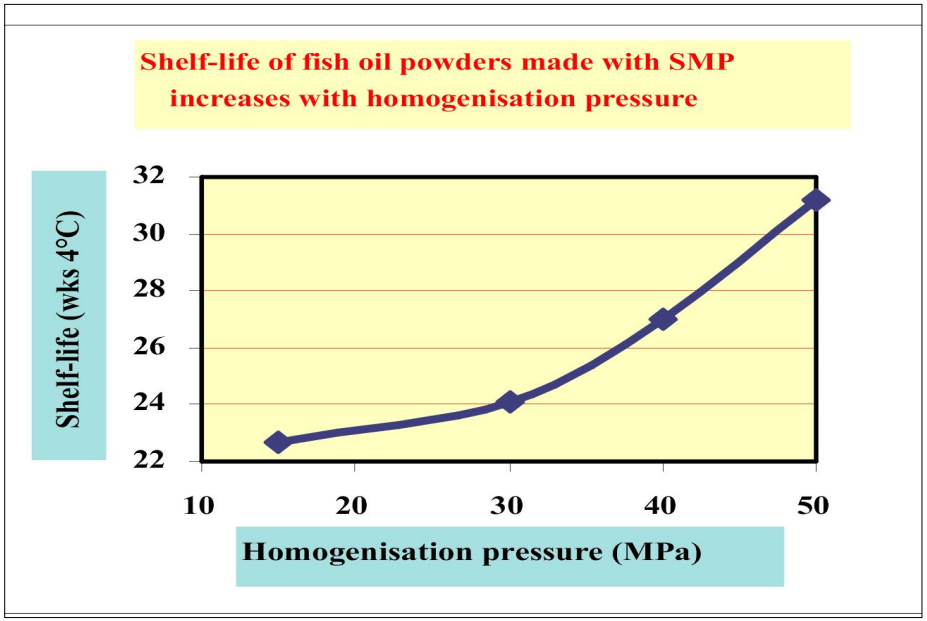
The vacuole volume of the powders ranged from 22.0 to 30.8 ml/100 g for the high and the low fat powders, respectively. An exception was the very high fat powder, which had a low vacuole volume of 5.3 ml/100 g. High fat resulting in high free fat levels would be expected to result in low vacuole volumes due to foam depressing effects. Nevertheless, the high vacuole volumes obtained reflect high levels of air entrapment which may explain what appears to be a relatively higher rate of oxidation in oil trapped within a powder particle matrix rather than at the exterior on powder surfaces.

## Sensory Evaluation and Shelf-life

The fishy off-flavour score of the powders after manufacture ranged from 10 - 27 on a 0 - 100 hedonic scale. The fishy off-flavour score reached a maximum at 3 - 12 weeks after which it decreased. None of the powders exceeded a score of 40 for fishy off-flavour during storage.

The metallic and painty off-flavour scores increased during storage (at 16°C) at different rates. A score of 40 was considered objectionable by the taste-panel. On an arbitrary basis, a score of < 12.5 was considered acceptable and < 25 was considered intermediate. The off-flavour scores were zero after manufacture, except where 1% fish oil was sprayed on. As homogenisation pressure increased, the time taken to reach an off-flavour score of 12.5 increased from 3 to 7.5 weeks. This result is in agreement with the surface fat - oxidation hypothesis, even though the surface fat values only decreased marginally from 46.3 to 40.0%. The storage stability was further increased to 12 weeks at the high protein:low lactose ratio but decreased to 6 weeks at the low protein:high lactose ratio.

Shelf-life was further increased to 15 weeks in the low fat powders but decreased to 3 weeks in the case of the high fat, high free fat powder. Both these results were expected, since they were related to changes in surface fat levels and also given that higher fat contributes to higher levels of off-flavour compounds. The time taken to reach an off-flavour score of 25 ranged from 12 to 18 weeks in the powders made from emulsions at various homogenisation pressures. However, storage stability was not related to homogenisation pressure.




## Shelf-life after Spray-Coating

When 1% fish oil was sprayed on to powder particles, shelf-life stability was expected to be low due to an anticipated increase in oxidation arising from a high surface fat coverage of 60%. However, this did not prove to be the case, and further studies are needed to investigate this anomalous observation.

## Co-Drying

Co-drying was carried out by simultaneously dry-dosing either starch or sodium caseinate during atomisation of the fish oil emulsion using the larger NIRO Tall-form spray drier (nominal capacity 100 kg/h). The objective was to enhance coverage of the rapidly drying atomised droplets in order to protect the subsequently dried particles against oxidation. No differences in shelf-life were later observed between the co-dried and control powders. However, microstructure examination of the resulting powders proved interesting.

Laser microscopy showed that the sodium caseinate samples had a more agglomerated appearance compared to the control. This was partly due to the



presence of large (up to 800 $\mu$m diameter), glassy, irregularly-shaped particles of protein, probably sodium caseinate. Starch was present mostly as discrete rounded particles, although some starch was present in powder agglomerates. Neither sodium caseinate or starch appeared to be covering the encapsulated fish oil particles.

Confocal scanning laser microscopy revealed some views of the internal powder particle structure, including the size and distribution of oil within the matrix. Oil droplets were evenly distributed in all powders and were less than 3 $\mu$m diameter. Very little surface or free oil was observed.

## Process Optimisation - Vacuole Volume and Shelf-life

Accordingly as numerous processing strategies failed to reduce surface fat content, it became apparent that factors other than the 'high surface fat - high oxidation rate' hypothesis may be at play in influencing shelf-life. A new approach examined the issue of air entrapment during spray drying on the basis that it could be a contributor to oxidation within the powder particle matrix. It quickly became apparent that sodium caseinate/lactose based emulsions gave rise to powders with higher than normal levels of vacuole volume compared to those in typical milk powders.

A subsequent study using microencapsulated fish oil powders with varying levels of vacuole volume confirmed that greater shelf-life could be achieved by adopting process conditions that favoured the creation of lower vacuole volumes.

Emulsification of fish oils using skim milk powder in place of sodium caseinate/lactose proved more successful, and led to the development of a novel process which was provisionally patented by the authors (Keogh et al. 1998). This is based on the hypothesis that vacuole volume in powders is lowered by decreasing the tendency of concentrates to foam through use of emulsifying proteins with higher levels of aggregation (as satisfied by the micellar state of casein in skim milk powder).

Since casein aggregation increases in the order:

*sodium caseinate* < *calcium caseinate* < *micellar casein*

the vacuole volumes of microencapsulated fish oil powders prepared using these three forms of casein corresponded to 21, 14 and 7 ml/100 g powder, respectively.

As a result of this reduction in vacuole volume, the shelf-life of the microencapsulated fish oil powders increased.

This indicates that vacuole volume and, therefore, the level of occluded air in the powders had a greater effect on oxidation during storage than most other physico-chemical properties measured.

Maximum shelf-life was estimated by extrapolation to be 36 weeks at zero vacuole volume and an homogenisation pressure of 50 MPa x 5 passes.

## Concluding Experiment:

A shelf-life comparison was made between fish oil powders which were:

- freeze dried (by another partner),
- spray dried with sodium caseinate + lactose,
- spray dried with SMP and
- two commercial powders A and B.

Each of the five powders was also stored under vacuum + $N_2$ or under vacuum only. There were no significant differences in shelf-life between the samples stored under these conditions, the main differences occurring in those samples stored in air where the skim milk powder based microencapsulated fish oil formulation excelled.

However, other notable differences between ***freeze-dried*** and ***spray-dried*** powders on the one hand, and between ***commercial*** and ***experimental spray-dried*** powders were observed (See *Appendix 1*).

## Food Product Application Study:

The main objective of the food product application study was to incorporate the equivalent of 0.9 g of *omega-3* PUFA (3 g of fish oil) in the form of ~ 9 g of microencapsulated fish oil powder/day into one of a number of selected consumer food products.



Bread, biscuits and soup powder were identified as the most suitable consumer foods for the incorporation of the microencapsulated fish oil powder, and were then selected for use in a bio-availability study carried out by the nutritional partners. A follow-up study examined the technological challenges associated with adaptation of food formulations for incorporation of microencapsulated fish oil powder, as well as the sensory and quality impacts on the resulting processed consumer food.

It was found that when the microencapsulated fish oil powder off-flavour score was less than the value of 25 (See *Sensory Evaluation*), the off-flavours were not detected in the bread and reconstituted soup powder were at acceptable levels. Successful application in bread was thought to be due to the presence of a carbon dioxide atmosphere. Soup powder reformulation was a simple dry-blending of microencapsulated fish oil powder without the necessity of any heating step until before consumption, and thus, did not compromise the stability of the microencapsulation system.

### Bread

*Bread volume yield:* Dough incorporation of experimentally-produced microencapsulated fish oil powders resulted in a more open crumb texture. On the other hand, a closed crumb texture arising from the use of commercial powders contributed to lower volume yield.

*Oxidation state as measured by peroxide value (PV):* There was no significant difference in PVs of bread, prepared from experimentally-produced spray-dried powders stored for 24 weeks. Breads prepared with commercial fish oil powders had lower PVs than the experimental, microencapsulated powders. Storage of breads under ambient conditions for 3 days showed little increase in PVs.

## Infant Formula

Spray dried whey-based infant formulae with added fish oil in the form of an experimental microencapsulated fish oil ingredient or a commercial fish oil ingredient were produced. The dry ingredients were reconstituted, combined with a vegetable oil mix, heat treated and spray dried. Sensory and chemical evaluation of these fish oil enriched infant formulae were compared with a control infant formula (no added fish oil) and a commercial infant formula according to the following experimental plan

| Code | Treatment | Explanation |
|---|---|---|
| 1 | Control | Spray dried whey-based infant formula without fish oil |
| 2 | Experimental micro-encapsulated fish oil powder | Spray dried whey-based infant formula incorporating experimental microencapsulated fish oil powder |
| 3 | Commercial micro-encapsulated fish oil powder | Spray dried whey-based infant formula incorporating commercial microencapsulated fish oil powder |
| 4 | Commercial infant formula | Commercially available infant formula |

The quantity of fish oil powders added to the infant formula was based on the recommended level of *omega-3* fatty acids for infants (30 mg DHA/day).

## Sensory Evaluation

* Chemical measurement of oxidation by PV showed that the commercial infant formula (4) had the highest level of oxidation initially and during the 16-week storage period.

* The infant formula produced with the commercial microencapsulated fish oil powder (3) had the lowest PV. However, these infant formula powders were 'least liked' in sensory tests over the storage period. This dislike was not due to a 'fishy' taint but rather an objectionable background flavour.

* The infant formula produced from microencapsulated powder produced in the pilot-process plant (2) did not deteriorate in sensory shelf-life properties over a 16-week storage period even though there was an increase in PV during storage.

*In summary, a fish oil powder ingredient, with low off-flavour and extended shelf-life was successfully manufactured in the pilot process plant using microencapsulation.*

*This ingredient was successfully incorporated into infant formula at 100% RDA for omega-3 polyunsaturated fatty acids.*



Appendix 1

Differences between ***freeze-dried*** and ***spray-dried*** powders:

**- Powder particle diameter values [D(v, 0.5)]** were lower (17 $\mu$m) *than the spray-dried* powders (33 - 43 $\mu$m).

- **Vacuole volumes** were lower (1.5 ml/100 g) than the *spray-dried* powder made with SMP (6.5 ml/100 g) and the *spray-dried* powder made with sodium caseinate + lactose (21.5 ml/100 g).

**- Wettability** time of all *experimental* powders was acceptable (60 s) but the *freeze-dried* and *spray-dried* powders made with SMP had shorter wetting times than the *spray-dried* powders made with sodium caseinate.

**- Shelf-life (12.5 score)** was lower (4 weeks) than *spray-dried* powder made with sodium caseinate + lactose (8 weeks) and the *spray-dried* powder made with SMP (12 weeks).

**- Shelf-life (25 score)** was lower (16 weeks) than *spray-dried* powder made with sodium caseinate + lactose (20 weeks) and the *spray-dried* powder made with SMP (22 weeks). It should be noted that the homogenisation conditions used for the *spray-dried* powder made with SMP were less than the optimum for the shelf-life (25 score) of 50 MPa x 5 passes.

*Differences between* ***commercial*** *and* ***experimental spray-dried*** *powders:*

**- Oil globule size D(v,0.9) values** of the powders after reconstitution were very large in the *commercial* samples (38 and 18 $\mu$m) compared to the *experimental spray-dried* samples (0.8 and 1.0 $\mu$m).

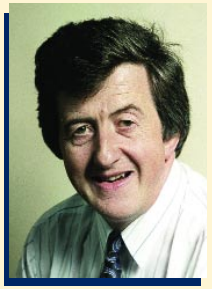
**- Free fat** levels were lower (1.4 g/100 g fat) than the *experimental spray-dried* powder (3 - 6 g/100 g fat).

**- Powder particle diameter D(v, 0.5) values** were higher (244 - 286 mm) than *experimental spray-dried* powders (33 - 43 $\mu$m).

**- Vacuole volume** levels were lower (0.0 ml/100 g) than the *experimental spray-dried* powder made with SMP (6.5 ml/100 g) and the *experimental spray-dried* powder made with sodium caseinate + lactose (21.5 ml/100 g).

**- Wettability** times were notably inferior to the *experimental spray-dried* powders. The *commercial* powders dissolved only when the aqueous suspension reached 35°C and agitation was used, whereas the *experimental spray-dried* powders had wettability times of < 60 s at 20°C.

**- Off-flavour** and **shelf-life** were distinctly different to the *experimental spray-dried* and *freeze-dried* powders. The *commercial* powders had an undefined, objectionable off-flavour at zero time, initially, and this hardly increased during storage. On the other hand, the level of metallic + painty off-flavour was less than the pilot-scale *experimental* powders, and did not reach a mean score of > 12.5 until 20 weeks storage at 16°C.



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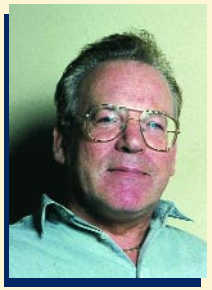

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