Microencapsulation of fish oil: comparison of three production methods

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INTRODUCTION

As vitamin, flavour and pigments, sensitive oil like fish oils has increasingly important in food industries, particularly because the nutritional benefits. Although no doubt of its nutritional values, adequate daily intake of fish oil in practical is difficult to achieve. Fish consumption is relatively low in many countries and effort to incorporate fish oil directly into food formulations always challenging mainly because the "fishy" flavours revealed from final food products (Kelly, 2000). Fishy flavours are caused by the susceptibility of fish oils to oxidation which limiting their use in foods application. Furthermore, the primary products of lipid oxidation, hydroperoxides, are also considered to be toxic (Oarada, 1988).

Therefore, attempts to prevent fish oil oxidation in order to allow omega-3 fatty acids to fulfil their functions and can be benefited to human health, is not trouble-free. Microencapsulation, in many cases can be used to overcome these problems. However, enveloping fish oil to produce microcapsules which are stable during storage without detection of fishy smell and taste from final products is a great challenge in food processing.

Intensive research has been done to microencapsulate fish oil. A number of wall materials including sodium and calcium caseinate, soy protein, whey protein, gelatine, dextrin (with a wide range of DE), sucrose, lactose, starches, modified starches, gum acacia, modified cellulose (MC and HPMC), as well as highly branched cyclodextrin (HBCD) have been applied to protect fish oils against oxidation. It is obvious that determining the best technology including: the process, the recipe, optimisation of auxiliary system, choosing the right matrix and encapsulation components is the main constraints to achieve the goals. From those constraints, choosing the right materials for the wall and deciding which process should be used are the most crucial steps.

This research was aimed to produce fish oil microcapsules from combination of several wall materials, namely: maltodextrin, soybean soluble polysaccharide (SSPS), modified starch, and hydroxypropyl betacyclodextrin (HPBD). The experiment was designed to obtain the best combination of wall materials produced by spray granulation (SG), spray drying (SD) and freeze drying (FD) processes. The results were examined and compared based on the oxidative stability, the microencapsulation efficiency and microcapsules microstructure. The rate of oxidation was particularly monitored in room temperature and storage for 8 weeks.

MATERIAL AND METHODS

Materials. In this study, fish oil 33/22 ultra refined (kindly provided by Cognis Deutschland, Illertissen, Germany) was used as core material. The wall material was an aqueous solution of SSPS (Soyafibe-S-EN100, Fuji Oil, Osaka, Japan) in combination with maltodextrin (Granadex M 20,

Biesterfeld Spezialchemie, Hamburg) and/or modified starch, hydroxypropyl betacyclodextrin (provided by Kleptose, HPB, Roquette, Frankfurt, Germany). All general chemicals used in this study were of analytical grade. Distilled water was used for the preparation of all solution.

Preparation of Emulsions. This study tested four combinations of wall materials (formulas). Wall materials were dissolved in distilled water using Ultra turrax (T45, Janke & Kunkel). The mixture then immersed in a cold water bath with ice and cooled for 10-20 min until temperature 10-15^{o}C was reached. Fish oil, 25% (w/w) was added and mixing continued at medium speed for 2-3 min.

Spray granulation. The homogenized emulsions were spray-granulated using Spouted Bed, ProCell 5 LabSytem (Glatt Ingenieurtechnik GmbH, Weimar, Germany). The air temperature was in a range of 55^{o}-75^{o}C, spraying pressure of 2.5 bar and spraying rate of ± 10 g/min. Granules were collected and keep at 3^{o} C while waiting for storage and analytical tests The spray granulation process was conducted in IPC Process Centre, Dresden, Germany.

Spray Drying. A pilot-plant spray dryer (Nubilosa AJM 014) was used to convert emulsion into encapsulated powder. The inlet temperature was 180°C and outlet temperature was 85°C± 5°C. The collected jars is changed every 2 minutes to avoid prolonged exposure to heat and fish oil powder obtained is transferred into cold glass jars and immersed as quickly as possible to ice water bath.

Freeze Drying. The emulsion was placed into aluminium plates and frozen at -70°C for 4 hours. A Christ Alpha 2-4 LSC freeze dryer was used to freeze dried the emulsion. During the drying process, the ice condenser was set at lower than -50°C and the pressure was around 0.120 mbar. Cold and dried emulsion was collected and ground to obtain fine and fluffy powder.

Granules and powder were stored for 8 weeks in room temp. The stability was monitored by measurement of Peroxide Value (PV) and headspace propanal by GC. Microencapsulation efficiency was determined by analyzing total oil content by enzymatic digestion method (as described by Curtis et al., 2008) and surface oil content (washing method using iso-hexane). Scanning Electron Microscopy was performed using Philips XL 20 Scanning Electron Microscope with Magnification of 25x and/or 100x. Particle size analysis was done using Sympatec LF laser diffractometer (Sympatec, Clausthal-Zellerfeld, Germany). The evaluated particle size distribution is volume based. The experiment performed based on a factorial design and the results represent the means of two replicates. The statistical analysis was done by SPSS version 17.0.

RESULTS & DISCUSSION

Formula	Wall materials (w/w %)			
	SSPS	Malto-dextrin	Modified Starch	HP β-CB
1	12,5	62,5	-	-
2	10	65	-	-
3	10	-	65	-
4	10	50	-	15

Table 1. Composition of wall materials

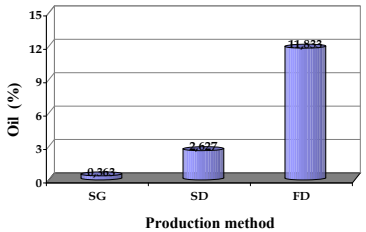


Fig 1. Surface oil content of microcapsules

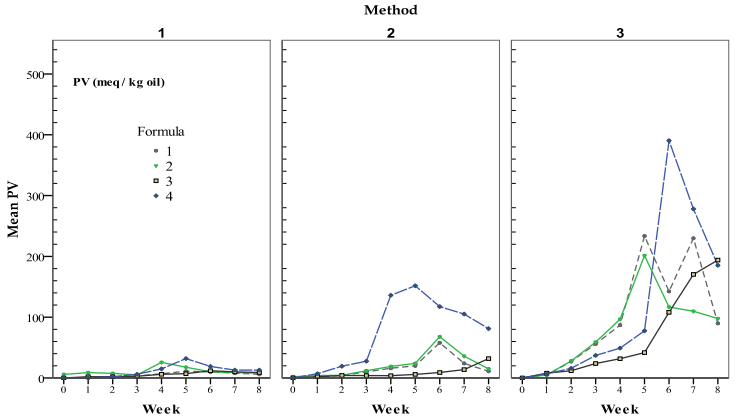


Figure 2. Oxidative stability of fish oil microcapsules in room temp., method used: (1) spray granulation, (2) spray drying, (3) freeze drying

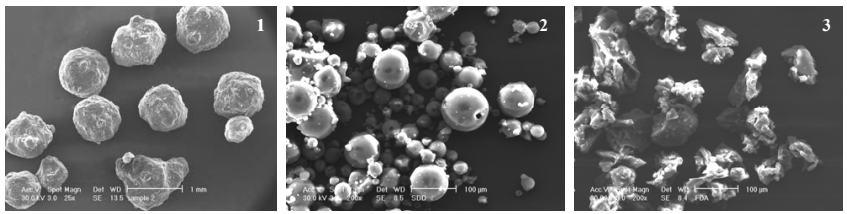
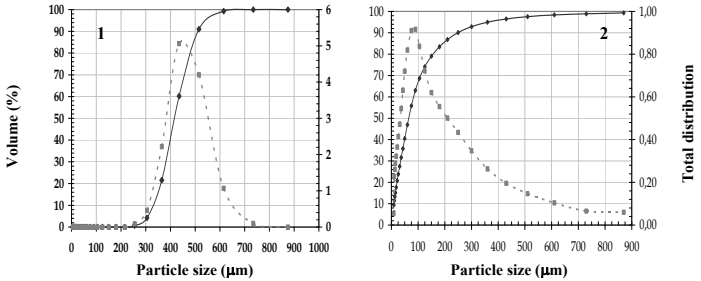


Figure 3. SEM micrographs of fish oil microcapsules from: (1) spray granulation, (2) spray drying, (3) freeze drying



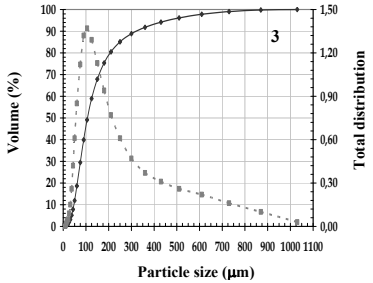


Figure 4. Particle size distribution of microcapsules: (1) spray granulation, (2) spray drying, (3) freeze drying

As can be seen from Fig.1, the surface oil content of SG powder was very low (0.363%) and therefore the microencapsulation efficiency was also high. Fish oil microcapsules from SG were relatively stable against oxidation over 8 weeks storage compared with those from SD and FD (Fig.2). PV of SG powder was less than 35 meq/kg oil and max of 12 meq/kg oil in formula 1 & 3. However, PV increased sharply after 3 weeks in SD powder using combination of SSPS, maltodextrin and HPBD. FD powder obviously formed more hydroperoxides in all formulas.

The high concentration of hydroperoxide as well as propanal (data and figures are not shown) in SD and FD microcapsules was correlated with the particle size and powder microstructure. SG powder has almost spherical shape with solid microstructure inside the granule which was different with typical SD powder and irregular shape of FD powder (Fig.3). The particle size of SG powder was in a range of 400–600 µm while SD powder was 80-120 µm and FD powder of 50-200 µm (Fig.4).

CONCLUSION

- Combination of SSPS and modified starch protected fish oil better than others three formulas.
- SG was the best production method to microencapsulate fish oil compared to SD and FD. It produced granules which high oxidative stability and minimum surface oil content. Low drying temperature and compact solid-shape of particles determined the stability.

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