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Food grade microcapsules produced by membrane emulsification

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Microcapsules or microspheres are widely used in many different applications in food, cosmetic and pharmaceutical industries [1,2] to encapsulate, protect and deliver bioactive compounds. The food industry is more and more focused in developing healthier foods by re-designing the production processes and enriching the foods with biocompounds. Fish oil, a natural source of poly-unsaturated fatty acids (PUFA) with physiological and health benefits, can be employed by the food industry to enrich foods in PUFA. However, the use of fish oil has some limitations, mainly associated to its oxidative susceptibility and the fishy odour and flavour [3,4]. To overcome these drawbacks and extent the shelf life of the product, microencapsulation techniques are used. Oil encapsulation is carried out in two steps: (i) preparation of an oil-in-water (O/W) emulsion and (ii) drying. The emulsification step plays an important role in the efficiency of the microencapsulation process, since the droplet size correlates with the biocompound encapsulation efficiency. The present work has studied the feasibility of combining membrane emulsification (ME) (lower energy requirements and more suitable to shear-sensitive ingredients than traditional emulsification techniques), with spray drying to produce fish oil microcapsules.

The O/W emulsions have been prepared by premix ME using 10 or 20% menhaden fish oil stabilized with food grade emulsifiers (Tween 20, whey protein, WPI, whey protein hydrolizate, WPH, or sodium caseinate, CAS). A coarse O/W emulsion was prepared by mechanical stirring (4 minutes, 20000 rpm) which was then passed several times (cycles) through a 0.8 μm membrane (Nylon or nitrocellulose mixed esters). The effect of the emulsifier type and concentration and the type of membrane in the flux and droplet size distribution was studied. As seen in previous works [5] the flux increases after each emulsification cycle for Tween 20, while it decreases when the emulsion was stabilized with a protein. This effect is mainly attributed to membrane fouling caused by the protein. Regarding the droplet size of the emulsions, the use of Tween 20 always resulted in the smallest values of the droplet size (close to the pore size of the membrane), while for proteins the droplet size was between 2 to 4 times the pore size of the membrane employed, depending on the emulsifier type and concentration.

Fish oil microcapsules were obtained by (spray) drying the O/W emulsions. The proteins used to stabilize the emulsions (WPI, WPH, CAS) combined or not with a polysaccharide (maltodextrin, MD) were used to build the microcapsules wall. Since the oil encapsulation efficiency is highly correlated with the droplet size of the O/W emulsion and with the oil-wall material ratio, three different oil–wall ratios (1:1, 1:2 and 1:3) were employed. All the produced microcapsules were analyzed to obtain, among others, the particle size, the surface oil content, the total oil content and the oil encapsulation efficiency (OEE). The outer and inner morphology of the microcapsules was characterized by SEM.

The ME results show a high correlation between the increase of the emulsifier concentration and the decrease of the droplet size of the emulsion (Figure 1a and Table 1). However, higher emulsifier (protein) concentrations result in lower values of the flux during ME (Figure 1b),

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attributed to an increase of membrane fouling. Regarding the oil encapsulation efficiency (OEE) the highest values correspond to microcapsules produced with the highest ratios of oil-wall material (1:3),reaching values close to 70% for O/W emulsions prepared with 20% fish oil,10% CAS and 50% MD (Table 1).

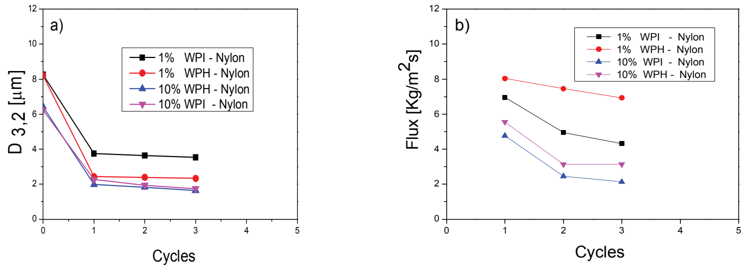

Figure 1. Droplet size diameter (a) and permeate fluxes (b) versus the number of cycles of O/W emulsions obtained by premix ME at 700 kPa with a Nylon membrane and different concentrations of whey protein (WPI) and whey protein hydrolizate (WPH).

Table 1. Formulation of the emulsions produced by premix ME with a nylon membrane and oil encapsulation efficiency of microcapsules produced by spray-drying.

Fish oil (%)	Emulsifier	Final droplet size of emulsion (□m)	Wall Material	Oil :Wall ratio	Oil Encapsulation Efficiency (OEE)
10%	1% WPI	4.93	30% MD	1 :3	27.62
	10% WPI	1.77	-----	1:1	29.44
	1% WPH	4.32	30% MD	1:3	31.3
	10% WPH	1.64	-----	1:1	34.1
	2% Tween	1.34	30% MD	1:3	40.56
	10% WPH	1.44	20% MD		55.95
20%	10% CAS	1.59	10% MD	1:1	39.19
		1.62	30% MD	1:2	61.18

		1.69	50% MD	1.3	71.18

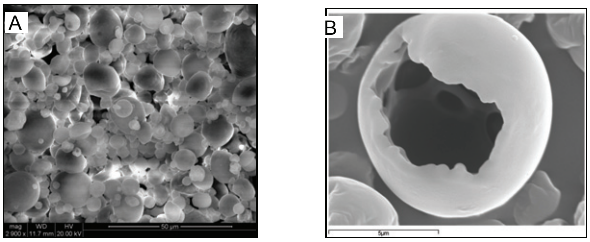

Figure 2. SEM internal (a) and external (b) images of microcapsules obtained drying O/W emulsions produced by premix ME: 20% fish oil, 10 % CAS and 50% MD.

The inner and outer morphology of the microcapsules was characterized by electronic microscopy (SEM). Typically an increase in the oil-wall ratio produces capsules with a thicker wall, more round in shape and without defects on the surface, while for the lowest oil-wall ratios the capsules do not have spherical shape, most of them are deflated and they present cracks on the surface and many vacuoles in the inside. Figure 2 shows, as an example, images of microcapsules prepared with 20% fish oil emulsion stabilized with 10% sodium caseinate and with 50% MD. The results of this work demonstrate that membrane emulsification combined with spray drying can be used to produce food grade fish oil microcapsules with high oil encapsulation efficiencies.

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