Easy flowing emulsion (o/w) based spray-dried powder produced using dietary fiber as a wall material

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Easy flowing emulsion (o/w) based spray-dried powder produced using dietary fiber as a wall material

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Abstract

The production of emulsion (o/w) based microstructured food powder through spray drying is a common practice in the food industry due to better shelf-life and easy transportation of the structured material. In general, the emulsion based powder flow behavior is poor due to lipid phase diffusion into the surface. The microstructure transform during spray-drying and the reconstitution of the emulsion powder are also a challenge by preserving the desired physiochemical properties such as emulsion size, stability, the control release kinetics of actives etc. The main objective of this study is to encapsulate the lipid phase using a wall material composed of protein (whey protein) and apple fiber. The stable submicron emulsions (o/w) were prepared using a rotor-stator at room temperature. Different fiber concentrations and different spray drying conditions were tested by varying the air to liquid mass ratio (ALR). The easy flowing of the emulsion powder was achieved when a relatively small amount (max. 5%) of fiber was used; however, the flowing performance declines with higher fiber content. The excellent reconstitution of the emulsion was also found by dissolving the particles at room temperature.

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Introduction

Microencapsulation technique is the envelopment of functional ingredients or actives for long preservation or control release kinetics. It is well-recognized in the food processing field, because it can be used to entrap functional components, with the objective of enhancing the value of the food products and release the added nutrients in a way in which they can be easily digested [1].

One of the methods used for microencapsulation is spray drying. This well-known technique that allows to create stable microstructures, preventing them from depletion in a liquid (multiphase) state [2], and it has been used to encapsulate different nutrients from phytosterols [3] to probiotic microorganisms like Lactobacillus plantarum ATCC 8014 [4]. The technology is functioning from a laboratory scale to an industrial production, and the different scaling up approaches have been successfully achieved [5].

Apple fiber is a good edible and sustainable (undervalued byproduct-driven) raw material with positive health effect. Apple fiber has already been studied for some formulations to produce diet supplement or animal feeding [6], and the dietary fiber is commercially available in the market. Moreover, ensuring the right dietary fiber intake could bring a reduction of the risk of obesity, diabetes and heart diseases [7]. Mouthfeel and unknown interaction with other ingredients limits its use in food application.

In the present study, the apple fiber was used as a wall material, together with whey protein isolate and a digestion-resistant maltodextrin, Fibersol-2 (both of which have good emulsifying properties) to encapsulate the core material (a model oil phase, i.e. commercial sunflower oil), without the addition of artificial emulsifiers. Fibersol-2 has been studied showing the capability to decrease hunger and increasing satiety hormones in humans when ingested with a meal [8]. In general, the emulsion (o/w) based powder has relatively low flowability nature due to oil phase migration into the surface, which cause additional effect of agglomeration, segregation, and reduced solubility.

The objective of the present study is to produce emulsion based powder for encapsulation application having (i) dietary fibers as wall material, (ii) easy flowability, and (iii) excellent reconstitution capability.

The new challenge faced in this study is to spray a liquid containing some insoluble components, which are relatively bigger with respect to the final particle size. Different recipes were tested, by varying the relative quantity of fibers, to obtain the most processable emulsion, and different process conditions for the spray drying process were studied, to obtain the optimal microstructure and flowability.

Materials and methods

Material

The materials were obtained from the following sources: sunflower oil from Morrisons (UK), WPI from Arla Food Ingredients, Vibi J (DK), Fibersol-2 from Prince International, Emmeloord (NL), Apple fiber AF 400-30 from Rettenmeier UK Ltd. (UK). Tap water was used for the preparation of emulsion (o/w), distilled water for the analysis.

Emulsions preparation

Three solutions of WPI, Fibersol-2 and apple fiber were prepared in water with different mass ratios (6:3:1, 3.65:1.67:1, 3:1:1). The solutions were allowed to hydrate overnight in the fridge at 4°C. The encapsulated material, sunflower oil was added to the solutions in a mass ratio to the aqueous phase to 1:9. The total concentration of the dissolved solids was 45 % of the weight of the final emulsion. Tab. 1 displays all the details about the compositions for the three emulsions.

One emulsion per recipe was prepared using a Silverson rotor stator (model LF5NA, Silverson Machined Ltd., Chesham Bucks, UK) at 9000 rpm for 10 min. Homogenization was performed at ambient temperature (20 °C).

	EM 1	EM 2	EM 3
Water	55	55	55
WPI	21	21	21
Apple fiber	3.5	5.25	7
Fibersol–2	10.5	8.75	7
Sunflower oil	10	10	10

Tab. 1 Compositions (% w/w) of the three emulsions.

Spray drying of the emulsions

The emulsion to powders were produced using the 4M8-Trix spray dryer (ProCept, BE). The feed and the hot air entered the spray dryer co-currently. The chamber had a total height of 2 m, with an inner diameter of 0.14 m. An external mixing nozzle was used for spraying the liquid with internal liquid tip diameter of 1.2 mm. The inlet temperature used was 170 °C, the peristaltic pump rotational speed was set for a constant flow rate, the cyclone air pressure was at 0.75 bar. Different nozzle air flowrates (1.5, 3.0, 4.5, 6.0 l/min) were investigated. The dried powder was stored in a sealed container at ambient temperature for one week before proceeding with further analysis.

Droplets size and size distribution of emulsions and powders

The average emulsions droplet size and distribution were assessed using laser diffraction particle size analyzer (Master Sizer 3000, Malvern Instrument Ltd, UK). The measurements were performed by considering a refractive index of 1.4735 and an absorption index of 0.01, spherical particle. The emulsions were diluted with distilled water to avoid multiple scattering effects. The duration of the measurement was 1 min, while stirring at 2400 rpm. All measurements were repeated three times and averaged to give the final result. For the powders, the same instrument was used, with the dry module. The conditions used were the following ones: obscuration range: 0.1-15 %, feed rate 25 %, air pressure 3.6 bar, hopper height 4 mm, refractive index 1.4000, density 1.3 kg/cm^3, absorption index 0.001, non-spherical particle.

Rheology

Emulsion rheology was evaluated through an Anton Paar rheometer (MRC 301), UK. Both the dynamic viscosity and the complex viscosity measurement were performed using parallel plate geometry. The range of shear tested was 0.1 to 100 1/s, while for the complex viscosity the frequency sweep went from 0.1 to 100 Hz, with 0.5 % strain applied (within linear viscoelastic region). The measurement was performed one time per formulation after reproducibility check for one sample for three times.

Moisture content

Moisture content of the produced powders was determined using a Mettler Toledo though TGA analysis following the T program as described here: 40 to 80 °C in 3 min, 80 °C for 30 minutes, 80 to 160 °C in 4 min. The difference in weight after the first two stages gives the quantity of evaporated water.

Surface morphology of powders

Surface morphology of the spray dried powders was observed by a scanning electronic microscope (FEI NOVA 200 NanoSEM). Samples were coated with 20 to 30 nm of Au under vacuum conditions before observation at different magnifications, from 500x to 20000x.

Flowability

Powder flowability was determined using a Powder flow tester (Ametek GB LTD T/A Brookfield, UK). The powders were filled into an annular trough PFT - 405, and the corresponding vane lid PFT - 515 304 S.S. was installed. Powders were levelled using a curved profiled shaping blade, and their weight was recorded before testing. The conditions used for the standard test were: torsional speed 1 rev/h, axial speed 1 mm/s.

Results and discussion

Droplet size and size distribution of emulsions

In Fig. 1, the drop size distribution of emulsion droplets (EM 1) including apple fiber, and apple fiber alone are shown, where it is seen that the emulsion droplet size is much smaller than fiber size. Since the emulsion droplet size and distribution are important factors on the emulsion stability, the submicron emulsion were produced. Table 2 shows the produced emulsion size and corresponding span, which has been affected by the fiber size.

In Table 2, it is seen that the mean emulsion droplet size is unaffected by the fiber even doubling the mass. After one day of storage at 4 °C, the emulsions present some signs of instability, presumably due to the Ostwald ripening (the major responsible of the big particles growth due to containing lower Laplace pressure compared to the smaller ones). This suggests that the spray drying operation should be performed immediately after the emulsion preparation.

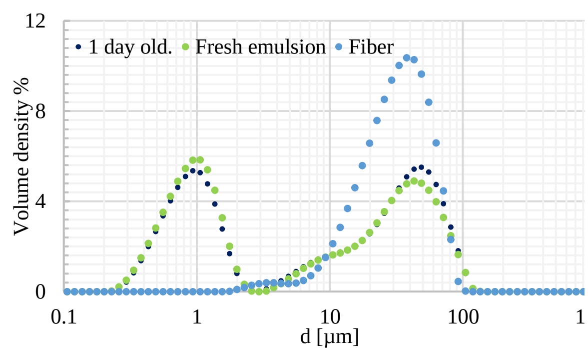

Figure 1. Particle size distribution of an emulsion sample (EM1) and the apple fiber used to prepare the emulsion.

	Fresh emulsion			After 1 day		
	d_{43}	d_{32}	Span	d_{43}	d_{32}	Span
EM 1	1.43	1.28	1.38	1.41	1.26	1.38
EM 2	1.39	1.23	1.40	1.31	1.17	1.33
EM 3	1.59	1.37	1.55	1.58	1.36	1.54

Tab 2. Emulsion droplet size including fiber and the corresponding polydispersity (Span).

Rheology

Fig. 2 shows that the emulsions are shear thinning fluid and having very high zero shear viscosity. The quantity of fibers directly affects the viscosity of the emulsion, and the viscosity increases with increasing fiber quantity. It was also observed that above 7 % of fiber concentration, the viscosity was too high for passing through the nozzle due to clogging and solidification at nozzle tip during spray drying.

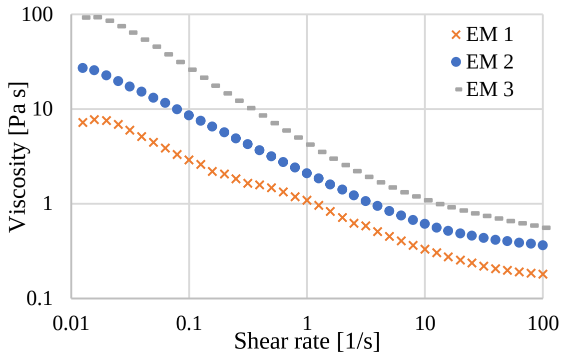

Figure 2. The viscosity of three emulsions as function of shear rate.

Spray drying powder size and size distribution

Fig. 3 shows the relative mean particle diameter (d_{50}) to nozzle diameter (d_{nozzle}) as a function of Reynolds number (*Re*), which was calculated using Eq. 1.

$$Re = \frac{\rho \left(v_{air} - v_{liq}\right) d_{eq}}{\mu_{air}} \qquad (1)$$

where, ρ is the density of air, v_{air} and v_{liq} are the velocity of air and liquid at nozzle tip respectively, d_{eq} is equivalent diameter of air area, and μ_{air} is the viscosity of the air. The air density and viscosity have been considered as the one at 25 °C, since the air provided to the nozzle tip is not heated.

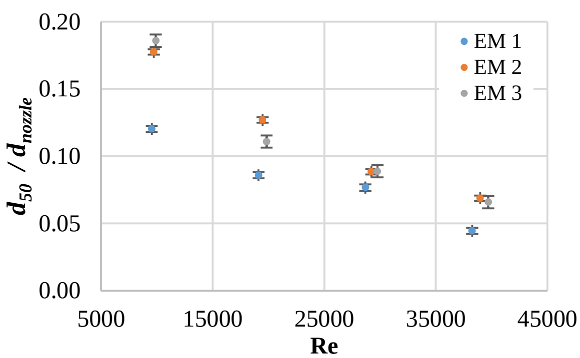

Figure 3. Ratio of the spray dried particle to nozzle inner diameter (d_{50}/d_{nozzle}) of the three formulations as a function of the Reynolds number of air at nozzle tip.

As expected (shown in Fig. 3), the spray drying particle size decreases with increasing *Re*. The quantity of fiber has an influence on the liquid velocity and viscosity and so the *Re* number. The emulsion with higher content of fibers seems to produce relatively higher particle size. However, a very small deviation has been found for the two larger quantities of apple fiber contained spray dried particles. This may be due to the limitation of flowability of high fiber containing emulsion in an extension flow in the nozzle. In practice, the liquid velocity decreases while the fiber content increases due increasing viscosity, so the actual *Re* is higher due to higher relative velocity (shown in Eq. 1). The mean particle size obtained using those emulsions are between 50 to 250 µm.

Surface morphology of the powders

The morphology of the produced powders has been assessed through scanning electronic microscope. One powder granule per emulsion recipe is shown in Fig. 5 (air nozzle corresponding condition: 3 l/min). The samples were broken on purpose, to show the wall microstructure and the successful inclusion of fibers, which can be identified as smaller spheres with a smooth surface.

The external shape is almost spherical, with occasional shrinkage zones, most probably due to different drying rates. The micro-pores inside the wall indicate how the oil was well included and evenly distributed. It cannot be excluded that some air was also included inside the pores, but the major part of it was present at the center of the sphere. The distinction between the voids creates let by the oil and the air cannot be made since all the oil present was gone due to the vacuum conditions inside the chamber, but the average dimension of the pores reflects the one of the oil droplets in the particle size distribution (centered between 500 and 950 nm in all cases).

The thickness of the walls is quite regular, and its width is approximatively 10 % of the total particle diameter.

Moisture content

The moisture contents for the three measurements, calculated with Eq. 2 those have been reported in Tab 3. Again, it can be noticed that the quantity of apple fiber did not influence the moisture content in the powder, and remains almost constant at a given drying condition. The reference powders for the analysis are the ones produced with 3 l/min of nozzle air.

$$MC\,\% = \frac{initial\ weigh - final\ weigh_{at\ 100\,°C}}{initial\ weigh} \cdot 100 \qquad (2)$$

Powder EM 1	Powder EM 2	Powder EM 3
3.7954 %	3.4920 %	3.6937 %

Tab. 3: Average moisture content of different emulsion based powders.

Flowability of powder

In Fig 5, it was found that the flow characteristic curve for the three samples produced with the same air nozzle condition (3 l/min). All the three types of powder are easy flowing, so they can potentially be produced on a large scale and be stored in a silo without creating arching and obstructions at the outlet. There is no correlation between flowability and the quantity of fibers within the analyzed range, however, the EM3, failed on the last locus. This may be an indication that a higher quantity of fiber can lead to a lower flowability nature.

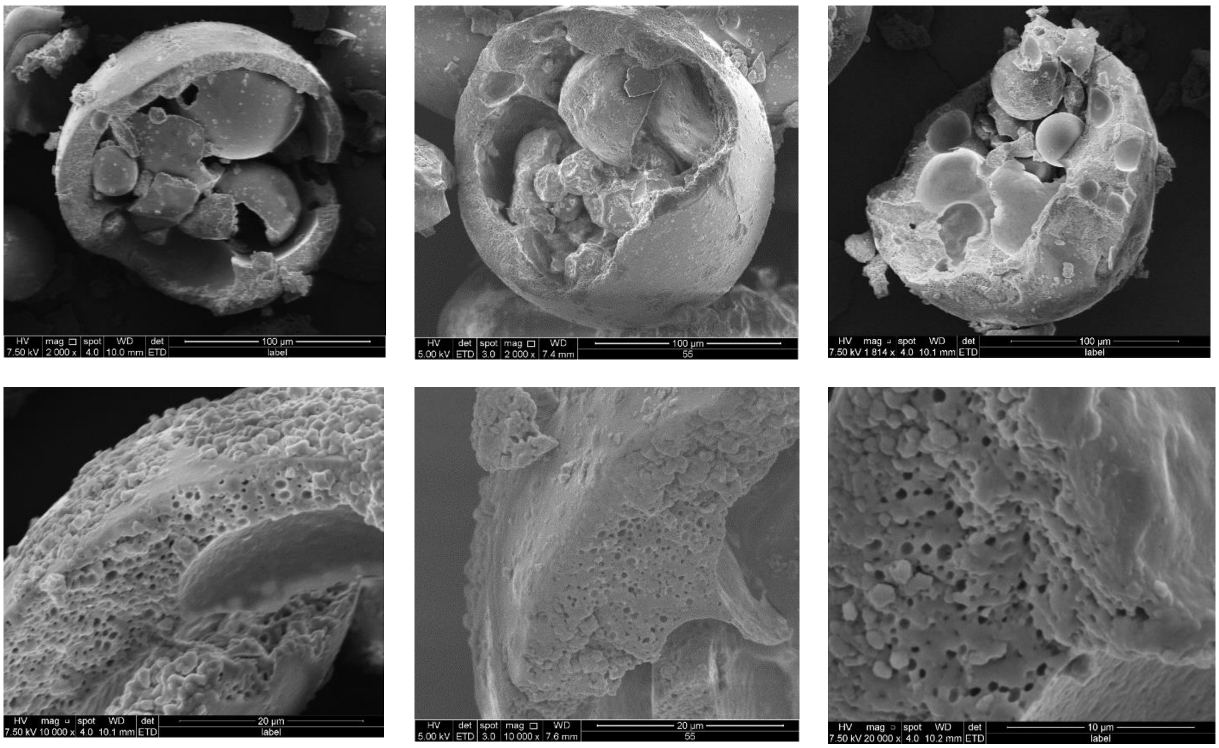

Figure 4. Microstructure of broken powder granules for the three different formulations.

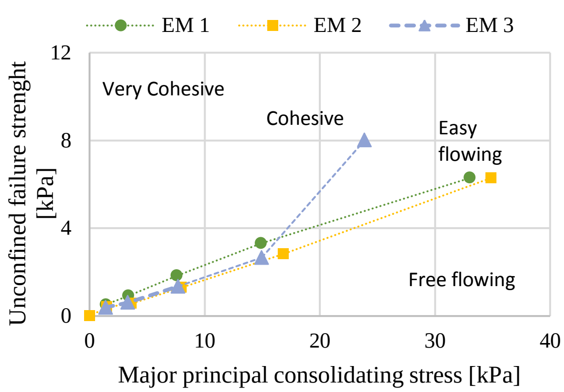

Figure 5. Flowability curves for three different spray dried powders at an air flow condition of 3 l/min.

The reconstitution of those emulsion powder into liquid emulsion has been also studied, where excellent dispersion ability has been found for all powders, however, some alteration of emulsion droplet has been found. The further investigation is required to quantify the phenomena.

Conclusions

Emulsion based powder incorporated with apple fiber exhibits an excellent flowability. The present study shows that emulsion based powder with fiber as wall material has potential for encapsulate active ingredients due to easy flowability. The easy flowing of the emulsion powder was achieved when a relatively small amount (max. 5%) of fiber was used; however, the flowing performance declines with higher fiber content. In addition, the dietary fibers have several health benefits and excellent reconstitution possibility due to porous structure. Further investigation is required to understand the benefit on the reconstitution of the emulsion using fiber.

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