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Stability of microencapsulated strawberry flavour by spray drying, freeze drying and fluid bed

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Abstract

The main goal of this paper was to study the stability of a microencapsulated strawberry flavour using different encapsulating agents and drying techniques: spray drying, freeze drying and fluid bed. According to the quantification of volatile compounds, the blend MDs/Hi-Cap (9/1) at a fixed concentration of CDs (1.7%) was the most appropriate for microencapsulating the strawberry flavour. The best drying yield was observed in the case of freeze drying. In the case of the moisture content, spray drying samples presented the lowest values, followed by freeze drying and fluid bed. The study of stability at different temperatures and times revealed that the presence of CDs in the blend enabled a higher presence of volatile compounds in the powder than in its absence. Microphotographs showed smooth spherical particles in the case of spray drying, whereas the structure of the powder was amorphous, like glass, with freeze drying and irregular in the case of fluid bed.

KEYWORDS: Microencapsulation; Spray drying; Freeze drying; Fluid bed; Stability; Strawberry flavour.

1. Introduction

The microencapsulation of flavours is a process of great importance in the flavouring and food industries. This technique encapsulates flavours in liquid form in a carrier matrix to obtain a dry flavoured powder, which is easy to handle because of its solid state. The capsules can be made of sugars, gums, proteins, polysaccharides, lipids and synthetic polymers [1]. The advantages of this technology lie not only in the protection it provides against degradative reactions and the loss of flavour, but also in the controlled release of flavours during food processing, storage and consumption. Before drying, one step is necessary to transform the feed liquid into a powder: emulsification of the flavours into small emulsion droplets within a carrier solution using a homogenizer [2].

Some of the most useful encapsulating agents are maltodextrins (MDs), arabic gum (AG), modified starches (Hi-Cap), xanthan (X) and cyclodextrins (CDs).

MDs are a hydrolyzed starch commonly used as wall material in the microencapsulation of food ingredients [3]. Their use offers advantages such as relatively low cost, neutral aroma and taste, low viscosity at high solid concentrations and good protection against oxidation. However, the biggest problem of this wall material is its low emulsifying capacity, so that MDs are best used in combination with other surface active biopolymers [4-5].

Starch, the food reserve polysaccharide of plants, is a commonly used food hydrocolloid. A number of starches available today are physically and/or chemically modified (Hi-Cap 100, Capsul). Hi-Cap 100 is a good film-former and presents low viscosity for high solid concentration. It presents good retention of volatile compounds [6]. The combination of MDs and Hi-Cap is suitable for obtaining emulsions with properties suitable for microencapsulating volatile compounds.

Cyclodextrins (CDs) are cyclic oligosaccharides that may be manufactured enzymatically from starch. α-, β- and γ-CDs comprise 6, 7 and 8 D-glucose units, respectively, connected through α-1→4 linkages. Each CD is shaped more or less like a thick-walled bucket, with a hydrophobic cavity and hydrophilic exterior. This unique structure enables CDs to form an inclusion complex, entrapping the whole, or part, of a guest molecule inside its cavity, principally by means of weak forces, such as van der Waals forces, dipole-dipole interactions and hydrogen bonding [7].

Different processes are available to prepare microcapsules, although no single process exists that captures the whole variety of capsule permutations and core-shell combinations [8]. Drying by atomization or spray drying refers to the removal of moisture from fluid material (solution, dispersion or paste) by breaking it into small droplets in the presence of hot air to obtain a dry powder. In the spray drying process, the liquid feed is pumped into the drying chamber through an atomizing system [9]. The technique has been used for many years in diverse industrial processes to obtain dehydrated materials in the form of fine powders and is the most commonly used method in the food industry due to its advantages [10].

Freeze drying is one of the most useful processes for drying thermosensitive substances that are unstable in aqueous solutions. The powders obtained after freeze drying are generally characterised by their low bulk density, high porosity as well as good aroma and taste retention [11].

Fluid bed is widely applied in industry for coating solid particles such as pellets, granules or powders. Wurster fluidized beds are the most commonly used form of equipment for the film coating of small particles [12]. This configuration is characterized by a bottom-spray nozzle and a Wurster tube, located centrally above a perforated distributor plate. This special design means that the particles are forced to follow a circulating flow trajectory.

Different flavours are used in the food industry, for example, the strawberry flavour is used in the confectionery industry in the production of chewing gum at a rate of 10 g/kg. The stability of this microencapsulated flavour is important to prolong the taste in the final product.

The aims of this study were to microencapsulate strawberry flavour by spray drying, freeze drying and fluid bed, to study the drying yield and moisture content of the resulting powders, to analyze the stability of the volatile compounds in the powder at different temperatures and storage times and to characterize the microcapsules formed using MD, CDs and modified starch as wall materials.

2. Materials and methods

2.1. Materials and reagents

Strawberry flavour was supplied by Creaciones Aromáticas Industriales (Carinsa, Barcelona, Spain). MDs (DE 19) were supplied by Tereos Syral (France), modified starchHI-CAP™ 100 by National Starch (USA), β- and γ-cyclodextrins by Wacker (Germany). Arabic gum and Xanthan were from Sigma-Aldrich (Germany). Hexane (>97.0 %) was supplied by Sigma-Aldrich (Germany).

2.2. Emulsion formation

MDs (0.6 g/mL), HI-CAP™ 100 (0.06 g/mL) and CDs (1.7%) were completely dissolved in distilled water, using a rotor-stator homogenizer (5 min; 500 rpm) (Ultra-turrax, IKA, Germany). After 24 h, the strawberry flavour was added to the wall material solution and was mixed using the same homogenizer.

2.3. Spray drying process

Spray drying process was performed in a laboratory scale spray dryer (Mini Spray Dryer B-290, Buchi, Germany). The emulsions were fed into the main chamber by peristaltic pump, controlling the feed flow rate by means of the pump rotation speed. The

compressed air flow rate was 5 bars, the feed flow rate was 2.5 mL/min, inlet and outlet air temperature were 180 and 90 ± 2 ºC, respectively.

2.4. Freeze drying process

Freeze drying was performed in a laboratory scale freeze dyer (Christ Alpha, Germany). The emulsions were prepared and then frozen at -80 ºC for 24 h with subsequent lyophilization to obtain powdery consistency.

2.5. Fluid bed process

The fluid bed process was performed in a laboratory scale Wurster fluidizer (VCF-LAB Micro Flo-Coater, Freund Vector, USA). The air flow in the fluid bed chamber was set at 15 m^3/h to enable the homogeneous dispersion of pellets. The inlet temperature was 45 ºC. The nozzle pressure was 10 psi. The pump speed was 10 rpm and the filter pulse was used every 15 seconds.

2.6. Drying yield

Drying yield was calculated by the following equation (1):

$$Yield\ (\%) = \frac{P}{T} * 100 \quad (1)$$

where P is the amount of powder (g) obtained after spray drying, freeze drying and fluid bed and T is the total solid content (g) used for the preparation of the emulsion.

2.7. Powder analysis

2.7.1. Quantification of volatile compounds

Homogeneous solid samples of powder (1g) were completely dissolved in distilled water (2 mL) with a magnetic stirrer for 5 min. After that, hexane (2 mL) was added and stirred for 5 min. The phase with hexane contained the volatile compounds and the quantities of these compounds were expressed as µg of volatiles/g of powder. The aqueous phase was discarded due to the absence of volatile compounds.

The strawberry flavour standard was serially diluted to prepare calibration straight lines in order to analyze the encapsulation efficiency. All the samples were analyzed in triplicate.

2.7.2. Gas chromatography-mass spectrometry (GC-MS) conditions

The GC-MS analyses were conducted on a Shimadzu GCMS-QP2010 (Japan) instrument using a fused silica Supelco SLB^{TM}-5MS capillary column (30 m×0.25 mm i.d.×0.25 µm film thickness) from Sigma-Aldrich (Germany). Helium was used as the carrier gas at a constant flow of 48 mL/min. The injection was split 50:1 and the injector temperature was 250 ºC. The oven temperature programme was as follows: initial temperature 60 ºC, ramped to 160 ºC at 4ºC/min, then ramped to 232 ºC at 6 ºC/min, finally ramped to 248 ºC at 8 ºC/min and held for 5 min. Total run time was 45 min. The mass selective detector was operated in an electron impact ionization mode at 70 eV, in a scan range of m/z 40–350. The interface temperature was 260 ºC. Three compounds were selected for flavour volatile analysis: ethyl acetoacetate, benzyl alcohol and ethyl 2- (2, 4-dimethyl-1, 3-dioxolan-2-yl) acetate, commonly known as fraistone. The results of the volatile analyses are provided as chromatographic peak area counts. Ethyl lactate (60µL), used as internal standard, was added before hexane to check the extraction of volatile compounds. All experiments were performed in triplicate.

2.7.3. Moisture content

The moisture content was determined by drying 30 g of each powder at 103 ºC in a hot air oven for 72 h (Memmert, Germany). The measurement was carried out in triplicate for each experiment and the average value is reported.

2.7.4. Stability

Microcapsules were stored at 4 and 25 ºC without light incidence for 5 months. The volatile compounds of the microcapsules were measured at months 0, 1, 2 and 5.

2.7.5. Particle size distribution

The particle size distribution was measured using a laser light diffraction instrument, Mastersizer 3000 (Malvern Instruments, Malvern, UK). The particle size was expressed as the mean volumetric size $D_{[4:3]}$ (De Brouckere mean diameter), which is the mean diameter of a sphere with the same volume and is generally used to characterize a particle. The characteristic parameters D_{10}, D_{50} and D_{90} were also calculated. Spread of particles was measured as the scatter, which is defined as [13] (2):

$$\text{Span} = \frac{D_{90}-D_{10}}{D_{50}} \quad (2)$$

2.7.6. Scanning electron microscopy (SEM)

Scanning electron microscope (MERLIN VP COMPACT, Zeiss, Germany) was used to evaluate the morphology of the dried powders. Samples were fixed to SEM stubs by means of double sided adhesive carbon tape, and the surface was coated with a thin layer of gold and examined at 15kV.

2.7.7. Statistical analysis

Each sample was assayed in triplicate, and the mean ± standard deviation was plotted. Student *t*-tests were performed to determine significant differences between the means. Mean differences were considered significant at the $p < 0.05$ level. Analyses were made using SPSS 20.0 software program.

3. Results and discussion

Previous experiments in our research group showed that MDs, Hi-Cap and CDs are suitable encapsulating agents for strawberry flavour, using a 9/1 ratio for MDs/Hi-Cap at a fixed β or γ-CDs concentration so consequently, this type of emulsion was prepared for the three different drying methods, using 1.7% β- or γ-CDs.

3.1. Drying yield

The highest drying yield was obtained for the freeze drying technique, varying from 87 to 88% (Table 1). Yield losses obtained in this process (12-13%) were very low compared with spray drying (38%) or fluid bed techniques (68%). The results obtained for freeze drying yield were slightly higher than those reported in other papers in which the same technique was used: in the case of microencapsulation of *Yarrowia lipolytica* lipase the yield obtained was 77% [14] and 70% in the case of flax oil microencapsulated with zein and freeze dried [15].

In the case of spray drying, to avoid high losses, the experiment was carried out with a high inlet temperature and slow pump rate. The spray drying efficiency was very similar for MD/Hi-Cap (66%), MD/Hi-Cap/β-CD (62%) and MD/Hi-Cap/γ-CD (60%) (Table 1). During spray drying, spray gas flow has little effect on the product properties and the pump rate determines the feed solution rate. If the pump rate is fast, the water will not vaporize fully in a short time and the spray dried powder will not be sufficiently dry; similarly, if the inlet temperature is low, a quantity of water will exist in the product and wet powders readily stick to the chamber wall (generally, only those powders collected in the container are regarded as effective), which will decrease the drying yield [16].

The fluid bed technique gave the lowest drying yield, which was similar in both samples (with or without β-CDs) at around 30% (Table 1), indicating that fluid bed is the worst encapsulating technique in terms of drying yield compared with spray or freeze drying.

3.2. Powder analysis

Before spray drying, the strawberry flavour was added to the emulsion at a rate of 15% of the total solid content. Strawberry flavour contains 20 volatile compounds (Figure 1). Ethyl acetoacetate (t_R = 6.2 min), which is a minor compound in the flavour and two of the major compounds, benzyl alcohol (t_R = 8.8 min) and fraistone (t_R = 13.2 min), were selected for analysis.

3.2.1. Moisture content

The characteristics of dried microparticles depend on the physical and chemical properties of the feed, dryer design, and operating conditions, while the shelf life of the dried microcapsules will depend on the moisture content and preparation/storage temperature [15].

According to Klinkesorn [17], the maximum moisture specification for most dried powder in the food industry is 3-4%. The results of the moisture content of the powder obtained by using spray drying, freeze drying and fluid bed techniques were consistent with the specifications for the industry (Table 2). Nevertheless, it is important to note that spray drying produced microparticles with the lowest moisture content (1.9 to 2.3%) compared with freeze drying (3.9 to 4.1%) and fluid bed (3.8 to 4.6%) (Table 2). The results achieved with spray drying were similar to those obtained by Frascareli [18] for the microencapsulation of coffee oil, where the moisture content varied from 0.8 to 3.2%.

3.2.2. Effect of temperature and storage time on the quantity of volatile compounds.

The powders obtained with the different drying techniques were stored at different temperatures (4 and 25 ºC) and the quantity of volatile compounds of the strawberry flavour was measured at different times (0, 1, 2 and 5 months) to evaluate the effect of storage temperature and time on the stability of volatile compounds in each powder. The results reflect the protective effect of the encapsulating agents on the volatile compounds analyzed.

3.2.2.1 Ethyl acetoacetate stability

Figure 2 shows the effect of storage time (up to 5 months) and temperature (4 and 25 ºC) on the stability of ethyl acetoacetate with different blends of encapsulating agents and drying techniques (spray drying, freeze drying and fluid bed).

The spray drying technique has been successfully used for the microencapsulation of different compounds in the food industry since the late 1950s to provide flavours with protection against degradation/oxidation and also to convert liquids to powders. Today,

spray drying is the most widely used technique for microencapsulation in the food industry [19]. Regarding the spray drying process, the blend MDs/Hi-Cap/β-CD (○) at 4 ºC (Figure 2A) provided a higher retention of ethyl acetoacetate after 5 months than MDs/Hi-Cap/γ-CD (□) and MDs/Hi-Cap (●). With β-CD in the mixture, approximately 70% of ethyl acetoacetate remained after 5 months of storage, whereas in the other blends this value was 50% in both cases.

At 25 ºC, the protective effect of the three blends over ethyl acetoacetate was very similar. However, as can be observed in Figure 2A and B, the increase in the temperature from 4 to 25 ºC caused a decrease in the remaining ethyl acetoacetate in the powder obtained by using spray drying process. The blend MDs/Hi-Cap/β-CD achieved the best results as in the case of 4 ºC, and approximately 45% of ethyl acetoacetate remained in the powder after 5 months. This value was very similar to that obtained for MDs/Hi-Cap and lower than the 40% obtained in the presence of γ-CD. It is important to remark that at both temperatures, the presence of γ-CD did not improve the protective effect of volatile compounds compared with β-CD. For this reason, freeze drying and fluid bed techniques were carried out only with the blends MDs/Hi-Cap (●) and MDs/Hi-Cap/β-CD (○).

Freeze drying or lyophilization is one of the most useful techniques for drying thermo sensitive compounds that are not stable in aqueous solution. Except for the long dehydration period required, freeze drying is a simple technique, which is particularly suitable for the encapsulation of aromatic compounds. At both storage temperatures (4 and 25 ºC) similar results were obtained (Figures 2C and 2D). At 4 ºC (Figure 2C), MDs/Hi-Cap/β-CD (○) retained 55% of ethyl acetoacetate after 5 months. This value was higher than 35% retained in the same period in the absence of CDs (●). Compared with the results obtained in the case of spray drying at 4 ºC, freeze drying did not improve the retention capacity of this volatile compound.

The curves obtained at 25 ºC (Figure 2D) were similar to those observed at 4 ºC, the presence of CDs in the mix leading to more ethyl acetoacetate being retained after 5 months; however both values were lower than those obtained for spray drying at 25 ºC in the presence or absence of β-CDs.

Originally developed as a pharmaceutical technique, fluid bed coating is now increasingly applied in the food industry. This technique is well known for its good solid mixing properties, leading to a lower tendency to agglomeration, as well as its good heat and mass transfer rates and a uniform temperature distribution [20]. The Wurster process is recognized as the best of the fluid bed processes for coating individual particles [21]. At 4 ºC (Figure 2E) the blend MDs/Hi-Cap/β-CD (○) obtained better results (35% of ethyl acetoacetate retained after 5 months) than the blend without CDs (●) (approximately 5%). At 25 ºC (Figure 2F), the losses of ethyl acetoacetate increased in the presence of CDs and only 15% of this aromatic compound remained in the powder after 5 months of storage time in the blend MDs/Hi-Cap/β-CD (○). After 1 month of storage, in the absence of CDs, the losses of ethyl acetoacetate were approximately 90% at both temperatures, 4 and 25 ºC.

Comparing these results for ethyl acetoacetate with those obtained for spray and freeze drying, it can be concluded that fluid bed is not suitable for microencapsulating strawberry flavour due to the losses of ethyl acetoacetate after 5 months of storage. In addition, it is important to note the protective effect of CDs over ethyl acetoacetate in the three techniques used.

3.2.2.2 Benzyl alcohol stability

Figure 3 showed the effect of storage time (up to 5 months) and temperatures (4 or 25 ºC) on the stability of benzyl alcohol using different blends of encapsulating agents and drying techniques (spray drying, freeze drying and fluid bed).

In the case of the spray drying process (Figures 3A and 3B) similar results were obtained for the three different blends analyzed. At 4 ºC (Figure 3A), the presence of β-CD (○) led to a higher retention of benzyl alcohol after 5 months compared with the blends MDs/Hi-Cap/γ-CD (□) and MDs/Hi-Cap (●). At 25 ºC (Figure 3B) the results were similar and the presence of β- (○) or γ-CD (□) led to better retention rates than in the absence of CDs, although differences were slight. Of note is the high stability of benzyl alcohol because more than 85% was present in the powder after 5 months of storage.

As regard the freeze drying process (Figures 3C and 3D), it can be observed that the presence of CDs protected the volatile compound after 5 months of storage at 4 or 25 ºC better than the absence of cyclic sugars. Figure 3C showed that approximately of 85% of benzyl alcohol remained in the powder after 5 months, whereas this value was lower in the case of the blend MDs/Hi-Cap (●) (75%). At 25 ºC (Figure 3D) similar values were obtained. Comparing the spray and freeze drying processes, it can be concluded that spray drying increases the quantity of benzyl alcohol retained compared with freeze drying.

Finally, the stability of benzyl alcohol with the fluid bed process is shown in Figures 3E and 3F. At 4 ºC the blend MDs/Hi-Cap/β-CD (○) showed similar values to those observed in the case of spray and freeze drying (85%) while the absence of CDs led to only 25% of the initial volatile compound remaining in the powder after 5 months. When the temperature was increased to 25 ºC, the losses in benzyl alcohol were higher with the fluid bed process (Figure 3F). Despite the fact that the quantity of benzyl alcohol decreased when the temperature increased, the presence of CDs in the mixture improved the protection of this compound. Again, it should be remarked the protective effect of β-CDs over benzyl alcohol in all drying procedures employed.

3.2.2.3 Fraistone stability

Figure 4 showed the effect of storage time (up to 5 months) and temperature (4 or 25 ºC) on the stability of fraistone with different blends of encapsulating agents and drying techniques (spray drying, freeze drying and fluid bed).

The stability of fraistone in the powder obtained by spray drying is depicted in Figures 4A and 4B. At 4 ºC (Figure 4A), the blend MDs/Hi-Cap (●) protected fraistone better than the presence of β- or γ-CDs (○ or □). However, at 25 ºC, the blend MDs/Hi-Cap/β-CD (○) showed the best results (90% in the presence of β-CDs and 80% in its absence (●)). The presence of γ-CD (□) did not improve protection so this kind of CD was discarded for use with freeze drying and fluid bed.

The results obtained for freeze drying are shown in Figures 4C and 4D. As can be seen, both figures were similar. At 4 ºC (Figure 4C), 60% of the fraistone remained after 5 months in the presence of CDs and approximately 45% in their absence. Similar behaviour was observed at 25 ºC (Figure 4D). As mentioned above, fraistone shows better thermostability because an increase in the temperature did not affect the quantity of this compound after 5 months.

Regarding fluid bed process, the results for fraistone stability are shown in Figures 4E and 4F. At 4 ºC fraistone decreased substantially in the case of MDs/Hi-Cap/β-CD (○) and only 45% remained in the powder after 5 months of storage, whereas less than 10% remained after 1 month of storage in the absence of CDs, indicating the importance of CDs for protecting this volatile compound. The results obtained at 25 ºC were similar to those obtained at 4 ºC and the losses in the presence or absence of CDs were very similar too.

3.2.3. Particle size distribution

Figure 5 shows the particle size distribution of the powders obtained with different encapsulating techniques. The encapsulation process was the factor that most affected the particle size if it is taken into account that the same proportion of encapsulating agents

was used to prepare the emulsions that were to be dried using spray drying, freeze drying and fluid bed.

The lowest particle size was obtained when spray drying was used. The parameter $D_{[4:3]}$ was 8.76 µm, a value similar to other compounds encapsulated using spray drying, e.g., coffee oil, when the mean particle diameter varied from 7.88 to 13 µm [18] or the microencapsulation of lipids in powder using spray drying, when the diameter was seen to vary between 8.1 and 17.9 µm [22].

The value $D_{[4:3]}$ obtained in the case of freeze drying was 637 µm. The larger particle size of the freeze-dried sample is due to the low process temperature, and the lack of strength to break the frozen drops or to alter the surface during drying [23]. In agreement with our results, microencapsulation of a phenolic extract with encapsulating compounds by means of freeze drying generated a similar diameter (684 µm) using guar gum and polydextrose [24]. Finally, fluid bed showed an intermediate value between spray and freeze dying, $D_{[4:3]}$ being 462 µm.

The span values are related to particle size distribution, with lower values indicating a more homogeneous distribution, which is a desirable trait [25]. Fluid bed gave the best homogeneity (1.64) according to span parameter, followed by spray drying (2.11) and finally freezes drying (2.42).

3.2.4. SEM analysis

The powders obtained by using spray drying, freeze drying or fluidized bed techniques were analyzed by scanning electron microscopy. As can be seen in Figure 6, the different drying processes resulted in powders with different particle morphologies. The smooth spherical particles observed in Figure 6A were achieved with spray drying and this is the best shape for protecting and retaining volatile compounds. An amorphous glass-like structure was formed during the freeze drying process (Figure 6B). According to Kaushik

and Roos [26] such a shape protects the entrapped molecules from exposure to heat and oxygen.

Figure 6C shows the microstructure of the particles obtained in the case of fluid bed. The amorphous and irregular structures observed are difficult to compare with other photographs due to the lack of research using fluid bed to microencapsulate food components.

4. Conclusions

The freeze drying process produced the highest drying yield of strawberry flavour, whereas the lowest moisture content was obtained with the spray drying technique. In the stability studies, spray and freeze drying led to similar results for the stability of ethyl acetoacetate, benzyl alcohol and fraistone. The presence of CDs in the mixture permitted better protection of these volatile compounds. This effect could be attributed to the structure of CDs, their internal cavity entrapping these volatiles and so protecting them from external agents that could break them down in the absence of CDs. The worst protective drying technique was fluid bed; the volatile compounds analysed decreased dramatically with both temperature and time of storage. The microstructure of the powder obtained reflected the typical spherical particles with spray drying samples, this shape is the most useful to microencapsulate volatile compounds.

The efficiency of spray and freeze drying were similar for the encapsulation of strawberry flavour; however it is important to consider the advantages and disadvantages of each process. Spray drying is cheaper than freeze drying and it is a more straightforward technology, so it should be selected as the main technique for microencapsulating strawberry flavour in the confectionery industry.

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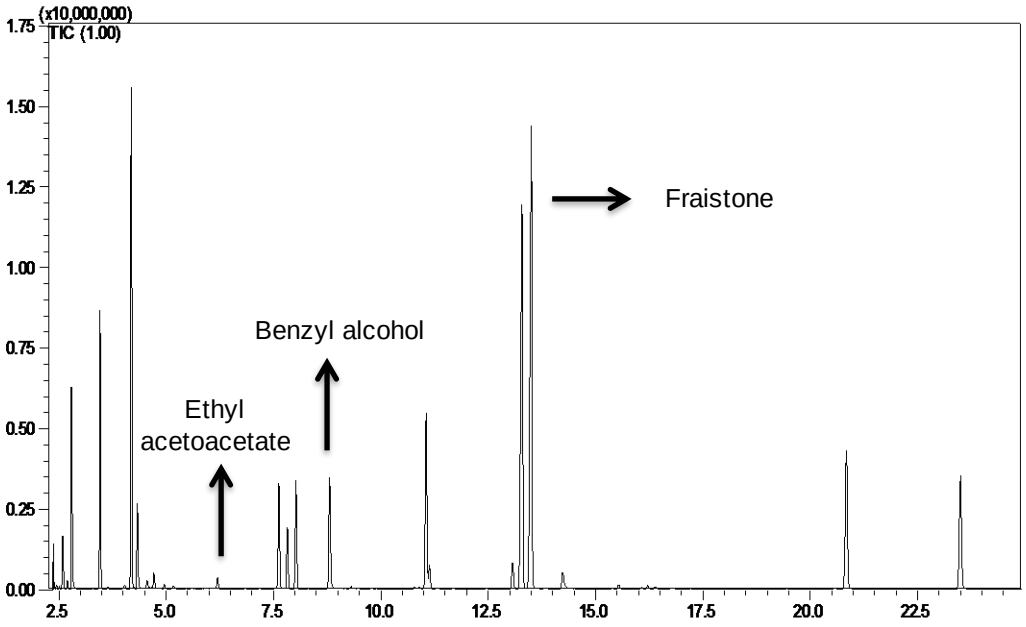

Figure 1. Chromatogram of the strawberry flavour with the three compounds analyzed.

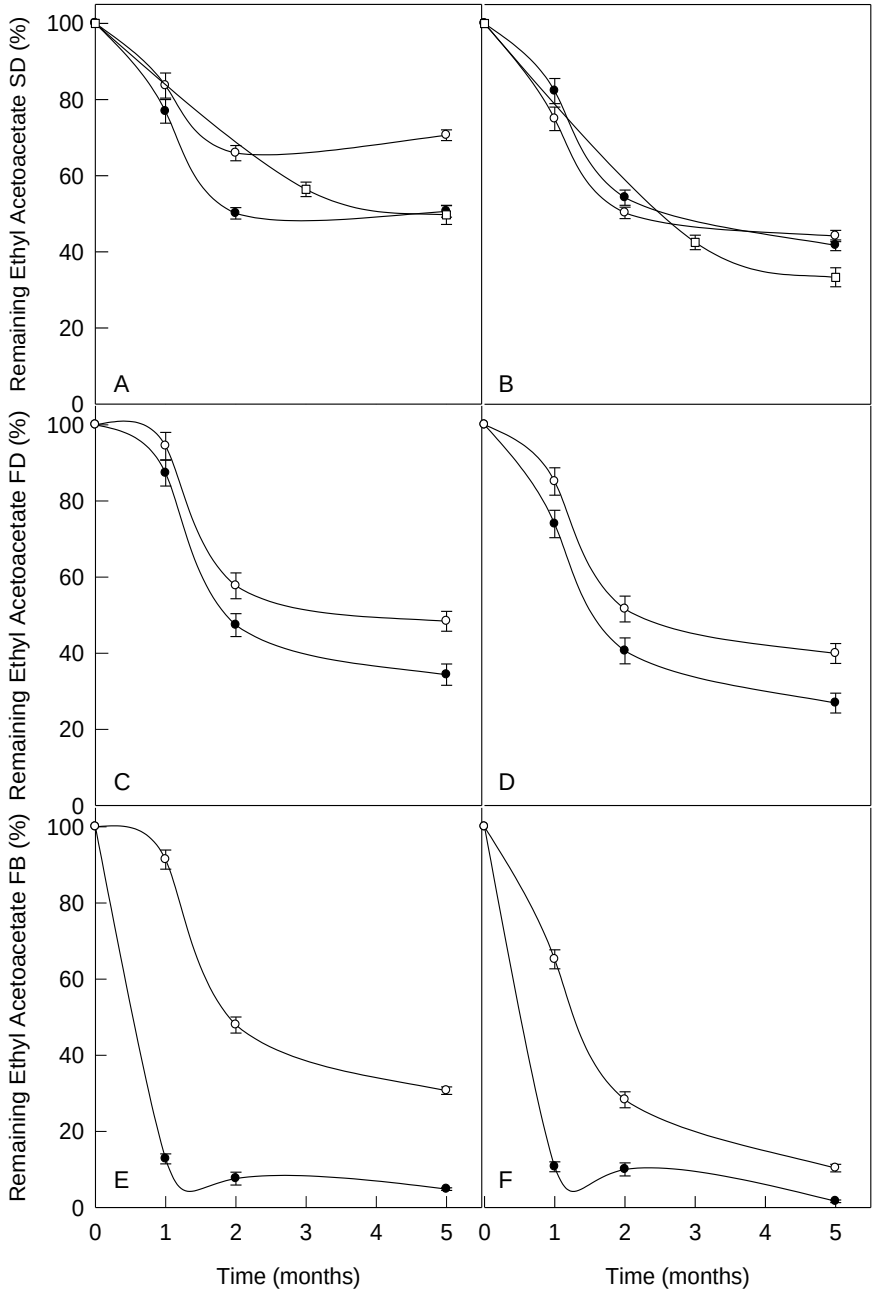

Figure 2. Effect of storage time and temperatures on the remaining ethyl acetoacetate for different blends, MDs/Hi-Cap/β-CD (○), MDs/Hi-Cap/γ-CD (□), MDs/Hi-Cap (●) and drying techniques. (A) 4 ºC Spray Drying. (B) 25 ºC Spray Drying. (C) 4 ºC Freeze Drying. (D) 25 ºC Freeze Drying. (E) 4 ºC Fluid Bed. (F) 25 ºC Fluid Bed.

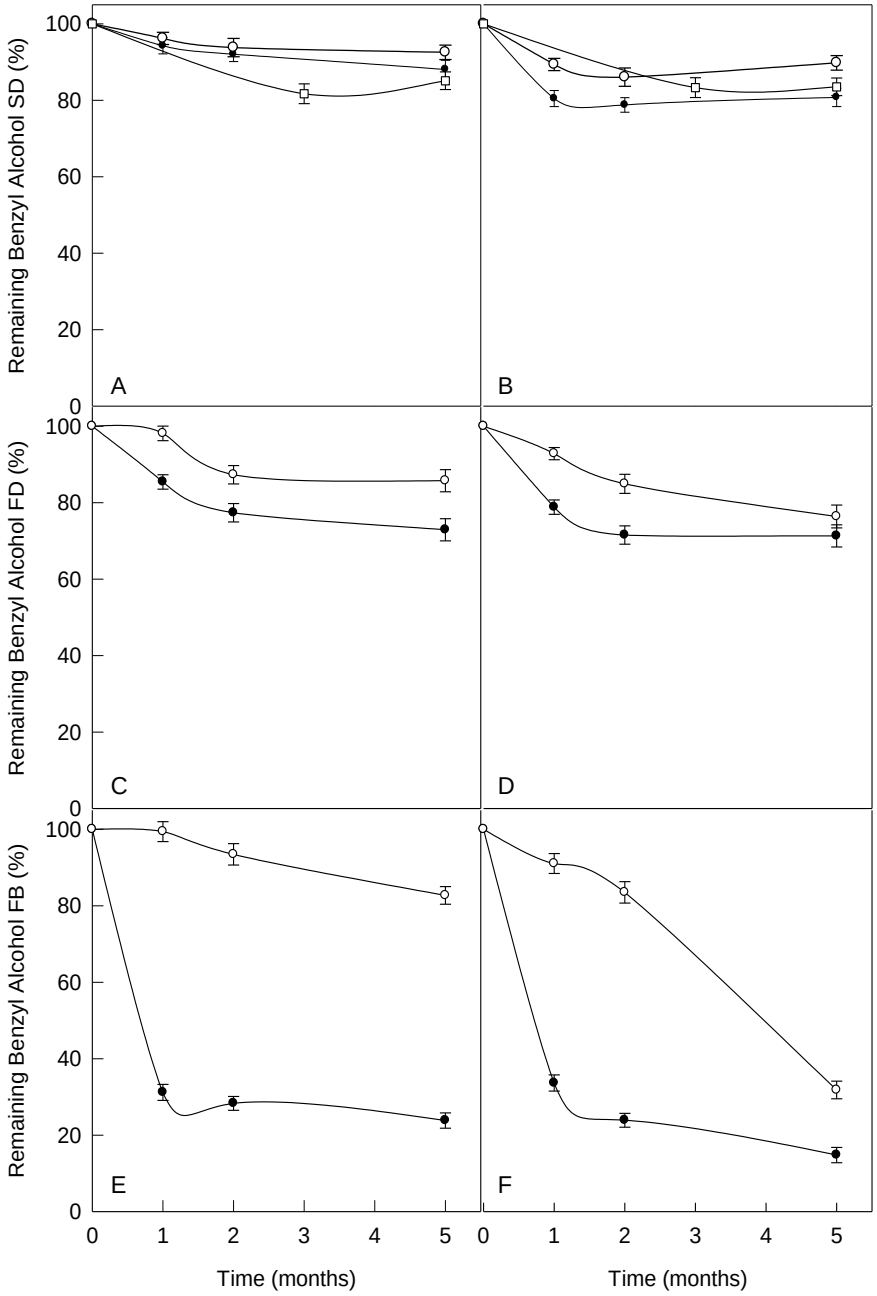

Figure 3. Effect of storage time and temperatures on the remaining benzyl alcohol for different blends MDs/Hi-Cap/β-CD (○), MDs/Hi-Cap/γ-CD (□), MDs/Hi-Cap (●) and drying techniques. (A) 4 ºC Spray Drying. (B) 25 ºC Spray Drying. (C) 4 ºC Freeze Drying. (D) 25 ºC Freeze Drying. (E) 4 ºC Fluid Bed. (F) 25 ºC Fluid Bed.

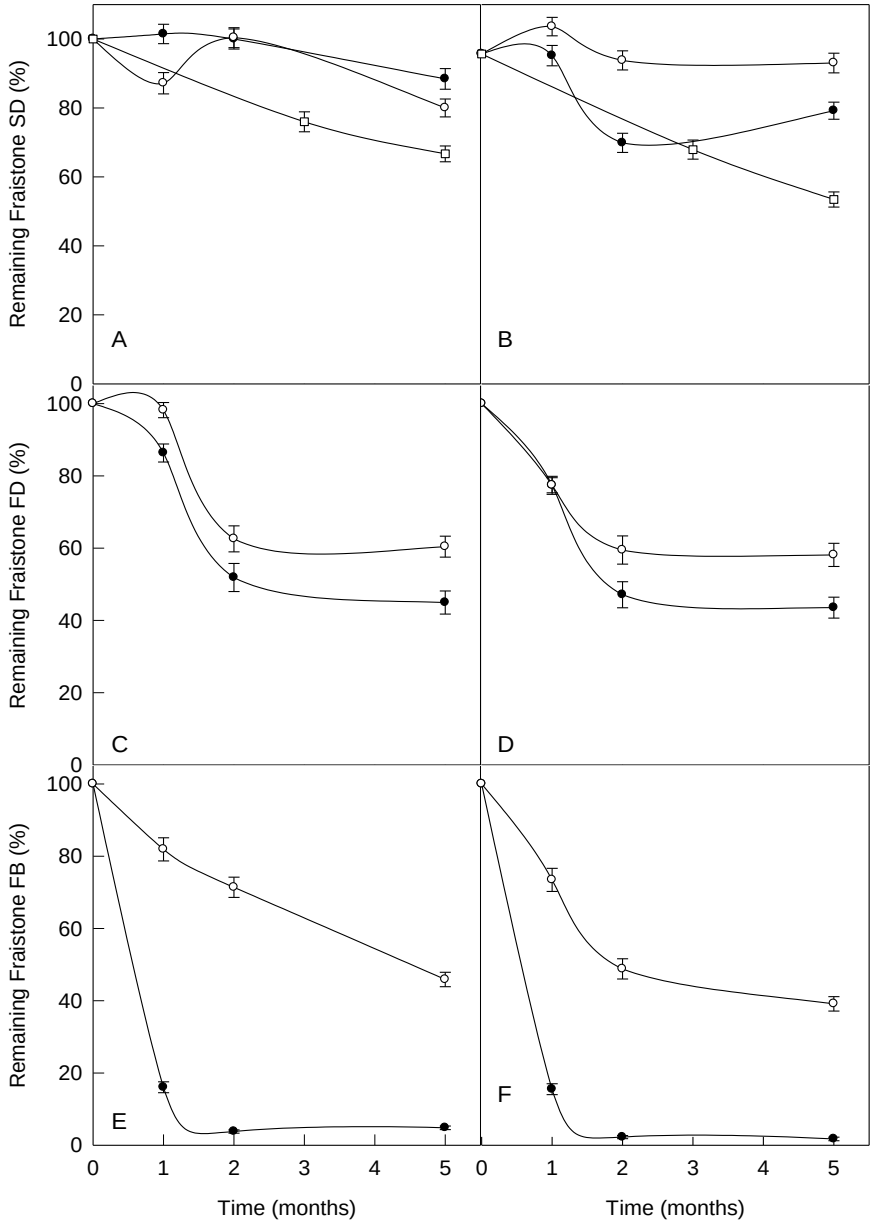

Figure 4. Effect of storage time and temperatures on the remaining fraistone for different blends MDs/Hi-Cap/β-CD (○), MDs/Hi-Cap/γ-CD (□), MDs/Hi-Cap (●) and drying techniques. (A) 4 ºC Spray Drying. (B) 25 ºC Spray Drying. (C) 4 ºC Freeze Drying. (D) 25 ºC Freeze Drying. (E) 4 ºC Fluid Bed. (F) 25 ºC Fluid Bed.

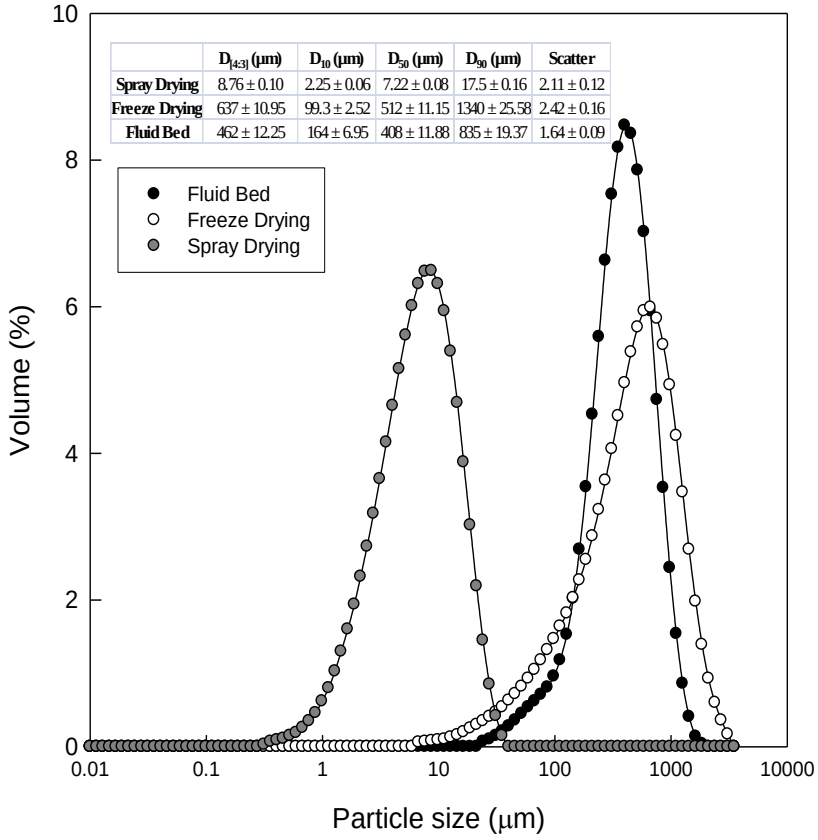

Figure 5. Particle size distribution of powders produced with spray drying, freeze drying and fluid bed. **Inset.** Values obtained for the different parameters analyzed for each encapsulating technique.

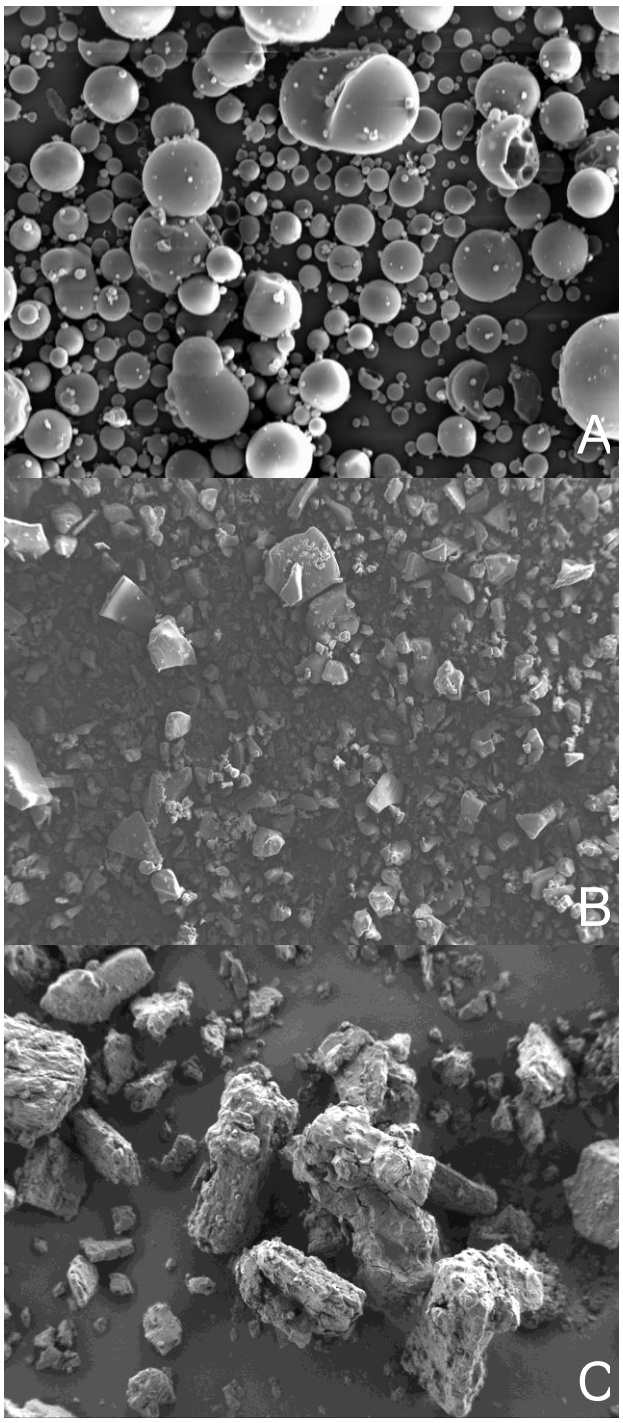

Figure 6. Scanning electron microphotograph. (A) Spray dried sample. (B) Freeze dried sample. (C) Fluidized bed sample.