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Optimization of the microencapsulation of synthetic strawberry flavour with different blends of encapsulating agents using spray drying

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ABSTRACT

Microencapsulation, increasingly considered a good alternative for retaining flavours and protecting them from degradation, is destined to have a great impact in the food industry. In the present study, the strawberry flavour was spray dried using different encapsulating agents such as maltodextrins (MDs), modified starch (Hi-Cap), arabic gum (AG), xanthan gum (X) and β-cyclodextrin (β-CD). Mixtures with different concentrations of these carriers were prepared and the stability, apparent viscosity and density were studied. The foam phase varied between 2 and 22%, the apparent viscosity ranged from 0.045 to 1.13 Pa·s and the density was approximately 1280 kg/m^3. Those mixtures considered the most suitable were spray dried using 180 °C as inlet temperature, and analyzed by gas chromatography–mass spectrometry (GC–MS). The drying yield varied between 45 and 57%, excluding the blend MDs/X/β-CDs. The blend MDs/Hi-Cap/β-CDs presented the best results in terms of the encapsulation efficiency of volatile compounds. In the case of the moisture content the values obtained ranged from 1.3 to 3.3%. SEM analysis showed that MDs/Hi-Cap/β-CDs and MDs/AG/β-CDs led to the formation of spherical, more homogeneous, and smoother particles. The results indicated that the blend MDs/Hi-Cap/β-CDs exhibited the best properties for encapsulating the flavour, followed by MDs/AG/β-CDs and MDs/X/β-CDs.

1. Introduction

The most important criteria for selecting encapsulation materials are the functionality that they should provide to the final product, the concentration of the encapsulates, the type of release, stability and cost. The materials used for the protective shell of the encapsulates must be food grade, biodegradable and able to form a barrier between the internal phase and its surroundings [1].

The most useful encapsulating agents are maltodextrins (MDs) [2], arabic gum (AG) [3] and modified starches (Hi-Cap) [4]. Other less common agents are xanthan gum (X) and cyclodextrins (CDs) [5]. MDs are formed by partially hydrolysed cornflour with acids or enzymes and are supplied as dextrose equivalents (DE), which is a measure of the degree of starch polymer hydrolysis. In selecting the wall materials for encapsulation, MDs are a good compromise between cost and effectiveness; they are also bland in flavour, have low viscosity at a high solid ratio and are available in different average molecular weights. Moreover, they have been shown to improve the stability of bioactive components and increase the yield and physicochemical properties of spray dried flavours [6]. However, the biggest problem with MDs is their low emulsifying capacity, so that it is desirable to use them in combination with other surface active biopolymers, such as AG [7], modified starches [8] or proteins [9].

AG is the gum that is most often used as a flavour-encapsulating material. Its solubility, low viscosity, emulsification characteristics and its good retention of volatile compounds make it a very versatile carrier for most encapsulation methods [10].

Starch and starch-based ingredients, such as modified starches, are widely used in the food industry to retain and protect volatile compounds. The ability of amylose to interact with certain ligands, particularly aroma compounds, has been known for a long time. Some of the formed structures are called complexes and can be described as a combination of ligand and ligand-induced helicated amylose [11].

X is an extracellular polysaccharide produced by *Xanthomonas campestris*. It is chemically composed of glucose, mannose and glucuronic, pyruvic and acetic acids. It is used in many products as a thickening or stabilizing agent and has also been used as an encapsulating agent for aromatic compounds [12].

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CDs are enzymatically modified starch molecules resulting from the action of cyclodextrin glucosyltransferase on starch. A typical application of CDs is protecting unstable and high added value speciality flavour chemicals. α-, β- and γ-CDs have previously been used to encapsulate different flavour compounds [13].

There are number of techniques available for the encapsulation of food compounds. Since encapsulating compounds are very often in a liquid form, many technologies are based on drying. Different techniques like spray drying, freeze drying and fluid-bed coating are available to encapsulate active agents [14]. Spray drying has been used for decades in many industrial processes to obtain dehydrated materials in the form of fine powders and is the most widely used encapsulation method in the food industry [15]. There are several reports on the microencapsulation of food flavours using different wall materials [13]. The encapsulation of strawberry flavour-related compounds has also been described in the literature [16].

The goal of this study was the microencapsulation of strawberry flavour by means of spray drying, which is considered the most feasible and economical method, and using a wide variety of encapsulating agents, being CDs the most original product for the industry. For this reason, the obtained powders were characterized in terms of moisture content, total volatile compounds, water activity and the structural characteristics by SEM analysis. In addition, the encapsulation efficiency was determined in each ratio analyzed.

2. Materials and methods

2.1. Materials and reagents

The synthetic strawberry flavour was supplied by Creaciones Aromáticas Industriales (Carinsa, Barcelona, Spain). MDs (DE 19) were supplied by Tereos Syral (France), modified starch HI-CAP™ 100 by National Starch (USA), β-cyclodextrins by Wacker (Germany). Arabic gum and Xanthan were purchased from Sigma-Aldrich (Germany). Hexane (>97.0%) was supplied by Sigma-Aldrich (Germany), helium was supplied by Praxair (Madrid, Spain) and ethyl lactate by Sigma-Aldrich (Germany).

2.2. Mixture formation

MDs, CDs, Hi-Cap, AG and X 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 mixture and mixed using the same homogenizer. The final mixture was characterized in terms of viscosity, stability and density. Mixtures were prepared with different ratios of MDs/Hi-Cap, MDs/AG and MDs/X at a fixed concentration of β-CD (1.7% weight/weight (w/w)). The initial concentration of each encapsulating agent was 0.66 g/mL in the ratio 10/0 and the rest of the proportions were based on this. The MDs/Hi-Cap and MDs/AG ratios were the same (10/0, 9.5/0.5, 9/1, 8/2, 7.5/2.5, 5/5, 2.5/7.5 and 0/10), but the high viscosity of the MDs/X mixture prevented us from using the same ratios (10/0, 9.99/0.01, 9.98/0.02, 9.975/0.025, 9.95/0.05, 9.925/0.075, 9.9/0.1 and 9.85/0.15). The different proportions of encapsulating agents were prepared following the recommendations of CARINSA and adding CDs for commercial interest.

2.3. Mixture stability

Immediately after the preparation of each mixture, aliquots (25 mL) were transferred to graduated 25 mL cylinders, sealed and stored at room temperature, and the volume of the upper phase was measured after 24 h. The stability was measured as % of separation, as expressed by Eq. (1) [17]:

$$\text{Separation}\ (\%) = \frac{H1}{H0} \times 100 \tag{1}$$

where H0 (cm) represents the initial height of the mixture and H1 (cm) is the height of the upper phase.

2.4. Mixture viscosity

The apparent viscosity was measured by rotational viscometer (Fungi Lab, Spain), running the spindle at 60 rpm, and the apparent viscosities were recorded at of 25 ± 2 °C [17].

2.5. Mixture density

The initial density of each mixture was determined in triplicate with a pycnometer (Isolab, Germany). at 25 °C [17].

2.6. Spray drying process

Spray drying was performed in a laboratory scale spray dryer (Mini Spray Dryer B-290, Buchi, Germany). Before drying, the strawberry flavour was added to the mixture at a concentration of 15% w/w of the total solid content. The mixtures were fed into the main chamber through a peristaltic pump and the feed flow rate was controlled by the pump rotation speed. The compressed air flow rate was 5 bars, the feed flow rate was 2.5 mL/min, and the inlet and outlet air temperatures were 180 and 90 ± 2 °C, respectively.

2.7. Drying yield

Drying yield was calculated by the following Eq. (2) [17]:

$$\text{Yield}(\%) = \frac{P}{T} \times 100 \tag{2}$$

where P is the amount of powder (g) obtained by spray drying and T is the total solid content (g) used in preparing the mixture.

2.8. Encapsulation efficiency

Encapsulation efficiency (EE) indicates the efficiency of the process to microencapsulate the different volatile compounds of strawberry flavour and was calculated as the ratio between the quantities of volatile compounds present in the final powder and the quantity of these compounds present in the initial mixture. The EE was calculated by the following Eq. (3) [18]:

$$\text{EE}\ (\%) = \frac{\text{Volatile compounds in the final powder}\ (\mu g/g)}{\text{Volatile compounds in the initial mixture}\ (\mu g/g)} \times 100 \tag{3}$$

2.9. Moisture content

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

2.10. Water activity (a_w)

The water activity was measured using a water activity meter (Aqualab, Series 3TE). Triplicate samples were analyzed and the mean was recorded.