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Influence of agglomeration on physical characteristics and oxidative stability of spray-dried oil powder with milk protein and trehalose wall material

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ABSTRACT

Sunflower oil has been microencapsulated by spray drying method in the matrix with trehalose and whey protein isolate (WPI) or sodium caseinate (NaCas). The obtained preparations have been subjected to wet fluidized-bed agglomeration with the use of trehalose and polyvinylpyrrolidone (PVP). Agglomeration of fine powders, irrespective of the wetting liquid, enabled obtaining a free-flowing product easily reconstitutable in water. Encapsulation efficiency of oil reached up to 96% and 99% in powder with WPI and NaCas respectively, whereas it decreased to 90–96% after the agglomeration, on account of insufficient agglomerate strength. The lower level of oil oxidation was observed for the agglomerate preparations, especially the agglomerate obtained with the use of the trehalose solution as a wetting liquid. The impact of agglomeration was reflected in a significant decrease of the level of adsorbed water, being sufficient to initiate the transition of amorphous trehalose to the crystalline state.

1. Introduction

A variety of food ingredients have been encapsulated as core materials by using different encapsulating wall materials and encapsulation methods for various purposes: protection from deterioration, preservation of functional characteristics, extended shelf storage stability, controlled release, targeted delivery, and alleviation of formulation and processing problems (Gouin, 2004; Vega and Roos, 2006; Gharsallaloui et al., 2007; Janiszewska and Witrowa-Rajchert, 2007; Vignolles et al., 2007; Jayasundera et al., 2009). Microencapsulated oil powder can be dry-blended with other dry ingredients, reconstituted as a stable emulsion in an aqueous solution or added directly to wet food products (Lee and Ying, 2008). A range of foods are reported to be able to be formulated with microencapsulated oils, including beverages, dressings and sauces, baked goods, dairy products, and powdered products.

The technique of spray-drying is still the main method of encapsulation in the food industry (Gharsallaloui et al., 2007). The selection of a suitable wall material is critical for successful microencapsulation (Gouin, 2004; Kagami et al., 2003; Vega and Roos, 2006; Palzer, 2011). Wall materials especially suitable for oil encapsulation have to be able to form and stabilize emulsions during homogenization, and to form a continuous compact wall matrix acting as an oxygen barrier between oil droplets in microcapsules when the emulsion is subsequently processed for drying (Shu and Rosenberg, 1995; Vignolles et al., 2007).

Milk proteins in conjunction with some low molecular weight carbohydrates have been reported to be an excellent encapsulating agents for oils and fats (Vega and Roos, 2006; Vignolles et al., 2007; Jayasundera et al., 2009). Recently, it has been shown that oil-in-water emulsions stabilized predominantly by caseins are more stable to spray-drying than those stabilized predominantly by whey proteins, which was attributed to denaturation of globular whey proteins (Sliwinski et al., 2003). Sodium caseinate has been reported to be the most effective emulsion stabilizer for fats (Hogan et al., 2001b). The primary function of proteins is to emulsify a system rather than to encapsulate it (Vega and Roos, 2006). Small molecules of carbohydrates, such as maltodextrins, corn syrup solids, glucose, and lactose, have been used to improve oxygen barrier properties of the wall matrix, to enhance drying property of the wall matrix, and to improve physical properties of spray-dried oil powders (Shu and Rosenberg, 1995; Jayasundera et al., 2009). The main property of these components is related to their formation of amorphous state in the dried wall matrix of microcapsules during spray drying (Bhandari and Hartel, 2005). In this context, physicochemical properties of trehalose appear to be very promising concerning its use for microencapsulation (Drusch et al., 2006; Alvarez Cerimedo et al., 2008). It possesses a uniquely high glass transition temperature (T_g) that ranges from 79 °C to 113 °C and is attributed to polymorphism in the crystallization pattern

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(Willart et al., 2002). Being a non-reducing sugar, this saccharide does not enter into the Maillard reaction with amino compounds such as amino acids or proteins (Sussich et al., 2001). It is particularly effective as a protein-stabilizing agent when combined with phosphate ions (Teramoto et al., 2008).

The amorphous state of the encapsulating carbohydrate material is a potential factor affecting the stability of oil. In the glassy state, the amorphous matrix is considered to be relatively stable. Problems associated with the use of low molecular weight carbohydrates in microencapsulation are caking and structural collapse as well as crystallization of the amorphous carbohydrate matrix upon storage (Roos, 1993; Buera et al., 2005). Apart from the negative impact on handling properties, both caking or collapse and crystallization may lead to a release of the encapsulated substance from the matrix (Drusch et al., 2006). Therefore, carbohydrates ought to be prevented from the development of crystalline form and storage conditions ought to be chosen carefully (Alvarez Cerimedo et al., 2008).

The occurrence of carbohydrate crystallization is dependent on molecular weight and conformational structure of carbohydrates, their glass transition temperatures, and relative humidity (Miao and Roos, 2005; Schebor et al., 2010).

The rate of crystallization is determined by both the absorption of humidity into the powder bed as well as the nucleation and growth of crystalline regions within the material (Hunter et al., 2010; Palzer, 2010). In this context, it would be valuable to explore how the kinetics of vapor sorption in powder relates to the crystallization of amorphous trehalose.

In order to delay the adverse phenomenon of sugars crystallization, use can be made of polyvinylpyrrolidone (PVP) addition. It is a water-soluble polymer amphiphilic in character, with good coating and adhesive properties (Hassoun et al., 2009). In a research by Mahlin et al. (2006), in the presence of PVP, the crystallization of sugars was initiated at a higher relative humidity (RH) of the environment, compared to crude sugars.

However, particles produced by simple spray drying can be smaller than 50 μm in diameter, which leads to poor flowability and slow reconstitution or lump formation during rehydration. Thus, usually a further agglomeration step is required to increase the particle size and to modify the particle structure in order to improve the final quality of powder (Hogekamp and Schubert, 1999; Szulc and Lenart, 2007). The mode of agglomerates formation in a given process determines their properties (Pietsch, 2003; Domian, 2005). Methods that afford favorable conditions for developing products with good instant properties include fluidized-bed and vapor–stream agglomeration (Hogekamp and Schubert, 1999). Properties of agglomerates formed in the fluidized bed are influenced by: properties of primal particles like size and character of their structure, type of binder and its concentration, process parameters including: air temperature, air flow rate, method of spraying the wetting liquid, and amount of the wetting solution added (Fuchs et al., 2006; Gao et al., 2002).

The aim of this study was to establish the effect of wet fluidized-bed agglomeration on the structure and selected characteristics of microencapsulated sunflower oil powder produced by spray-drying of an emulsion stabilized with sodium caseinate or whey protein isolate with the addition of trehalose. The scope of the study included the analysis of the effect of agglomeration in the aspect of the wetting liquid used in the process (an aqueous solution of trehalose and solutions of low- and high-molecular-weight polyvinylpyrrolidone) on such properties of the powder as: structure and size of particles, free fat content, bulk density, particle density, flowability, wettability and dispersibility in water, hygroscopicity and susceptibility to caking, as well as oxidative stability of the microencapsulated oil in stored preparations.

2. Materials and methods

2.1. Materials

The basic raw materials used in the study included: sunflower oil (Marlibo, Poland); sodium caseinate – NaCas (Polsero, Poland), protein content 90% (in dry matter), water content 6%; whey protein isolate – WPI (Carbery, Ireland), protein content 91.6% (in dry matter), water content 4.3%; trehalose (Hayashibara, Japan), trehalose content min. 98%; phosphate Carnal 2110 being a combination of di- and tri-phosphates of sodium and potassium (Brenntag, Poland), P_2O_5 content 50–52%, Na_2O_5 content 12–16%, K_2O content 32–37.5%, pH of 1% solution 8.6–9.0; low-molecular-weight polyvinylpyrrolidone (PVP) with the molecular weight of 29,000 Da (Sigma–Aldrich, Poland); high-molecular-weight polyvinylpyrrolidone with the molecular weight of 55,000 Da (Sigma–Aldrich, Poland); silica Aerosil 200 (Evonik, Germany).

The analytical material included preparations of microencapsulated sunflower oil in the form of powder and their agglomerates, as presented in Table 1. The W and C powders, containing 55% of sunflower oil, 34% of trehalose and 10% of a protein component, were obtained as a result of spray-drying of o/w emulsions stabilized with, respectively, whey protein isolate (W) and sodium caseinate (C) (Table 1). In terms of fat content, the composition of the obtained powders correspond powdered high fat-filled ingredients made of vegetable oil (palm oil, coconut oil) on a dairy base, in which 50–60% is normal. Preparations CT, CTPL, CPL and CPH in the form of agglomerates were produced as a result of agglomeration of C powder using aqueous solutions of respectively: trehalose, trehalose and low-molecular-weight PVP, low-molecular-weight PVP, and high-molecular-weight PVP, as wetting liquids. Adding the amount of wetting liquid as shown in Table 1, were append to the 100 g of preparation: 8 g of trehalose to the CT agglomerate, 8 g of trehalose and 2 g of low-molecular-weight PVP to the CTPL agglomerate, 3 g of low-molecular-weight PVP to the CPL agglomerate and 3 g of high-molecular-weight PVP to the CPH agglomerate, respectively.

2.2. Emulsion preparation, spray drying and agglomeration

2.2.1. Preparation of emulsions

o/w emulsions were prepared according to the recipe: water phase 78% (including 4% WPI or NaCas, 13.6% trehalose, 0.4% phosphates, and 60% water) and oil phase 22%. According to the above proportion of components, the content of oil in powders obtained after the spray-drying of emulsions reached 55%. The wall material was dissolved in distilled water under magnetic agitation, 1 day before emulsification. The pH of protein solutions was adjusted to pH 7.0 using a phosphate. Pre-emulsions were obtained by a rotor–stator system (Ultra-turrax IKA T18 Basic, Wilmington, USA). Emulsions were prepared by a final two-step homogenization at 600/150 bar with two passes in a high pressure homogenizer (Panda 2 K; Niro Soavi, Italy).

2.2.2. Spray-drying

The spray-drying of the emulsions was performed with a laboratory scale spray dryer (1–7 kg/h water evaporative capacity, Mobile Minor, Niro A/S, Denmark) equipped with a rotating disk for atomization. An air inlet temperature of 150 °C and an outlet air temperature of 60 °C were selected and disk rotation was at approximately 20,000 rpm.

2.2.3. Agglomeration

Wet-fluidized bed agglomeration was conducted in a STREA 1 agglomerator (Niro-Aeromatic AG, Germany). Before the process,