PGSS - The innovative production of fluid-filled microcapsules for the food industry

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Abstract

The PGSS (Particles from Gas Saturated Solutions) process is suitable for the micronisation of several substances and various polymers. With a couple of investigations it was shown that fine powders with different morphologies can be manufactured. Aim of this investigation was the production of powdery food products using the PGSS process. In the context of this work 165 experiments with the model systems palm fat/water, castor fat/water and PEG 6000/rapeseed oil were used to determine the interdependencies between process parameters of the PGSS process and particle and powder properties on the other hand. The aim was to investigate important influences on product specific characteristics. Afterwards, the knowledge obtained with the model systems was applied for the production of fluid-filled micro powderous composites for the food industry. Here, special attention was put on the influence of the process parameters on particle size and bulk density. The evaluation of the three model systems showed that the PGSS process is suitable for the production of powderous composites with variable characteristics. The particles can be adjusted by the process parameters in bulk density, particle size distribution and morphology.

Keywords: PGSS, Particles from Gas Saturated Solutions, microcapsules, encapsulation

1. Introduction

Many solid products used in the food industry are preferably applied in the form of fine-dispersed powders. Classical methods such as crystallization, (spray) drying, grinding, and sieving offer certain well-known possibilities to generate powders. However, if very small particle sizes are required, or non-brittle substances have to be reduced to the micro scale, limits of traditional processes are reached. In such cases, a solution can be found in modern high-pressure processes.

One of these processes is called PGSS (Particles from Gas Saturated Solutions). This process runs at low temperatures with pressurized carbon dioxide and is suitable for products that are sensitive to heat or oxidation. Inert packaging can easily be integrated. The use of organic solvents, emulsifying agents or other additives can be avoided. For these reasons the PGSS process operates at especially gentle conditions and is environmentally compliant. The possibilities of the PGSS process have successfully been demonstrated over the last years for numerous solids and liquids [1-11]. Recently, it has been demonstrated, that the PGSS technology is suitable for generating fluid-filled particles for food applications.

2. Experimental setup

The PGSS process permits a careful processing in an inert gas atmosphere at low temperatures, moderate pressures and is suitable for producing powders and composites of solids, very viscous melts and even liquid substances [12]. With this technique powders with different particle morphology and –size distribution can be obtained.

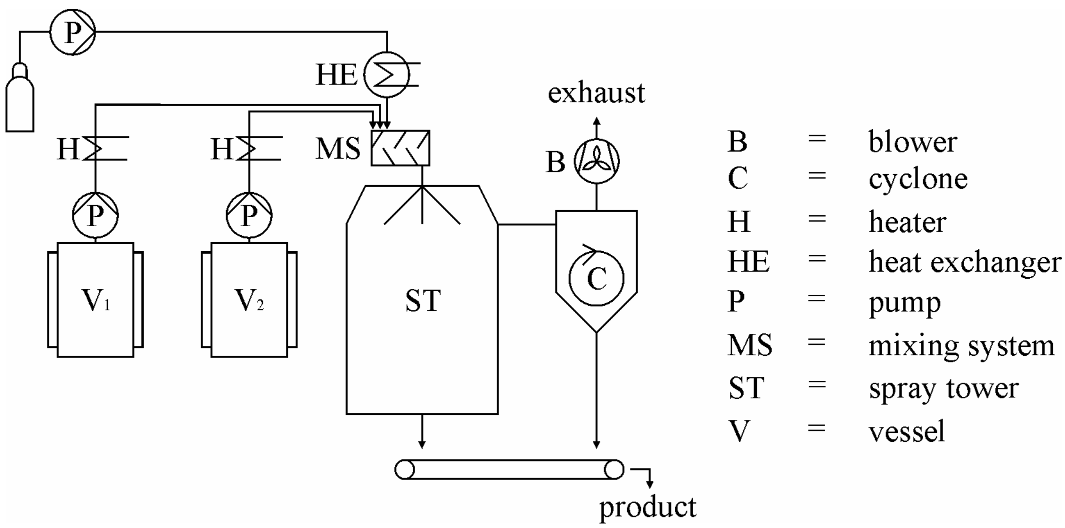


Figure 1: PGSS process for encapsulation purpose

The shell material (e.g. chocolate, palm or castor fat) is melted in vessel 1 (V_1) and the core material (e.g. liquid extracts or flavors) is filled into vessel 2 (V_2). Both materials are heated to the designated temperature, pressurized with pumps (P) to the required process parameters and dosed to a mixing system (MS). In the mixing system (e.g. static mixer) the two substances are homogenized and a supercritical fluid, predominantly CO_2, is admixed under sufficient dissipation of energy to form micro droplets of liquid in the melted shell material. Subsequently, the mixture is expanded to ambient pressure through a nozzle into a spray tower (ST) forming fine droplets. The simultaneously expanded gas removes heat from the droplets very rapidly (Joule-Thomson phenomenon), so that the shell material solidifies and forms a firm shell around the micronised droplets of the liquid, thus generating a powderous composite. The PGSS process uses the solidification of the continuous phase of emulsions for composite generation. Open as well as closed composites can be generated this way.

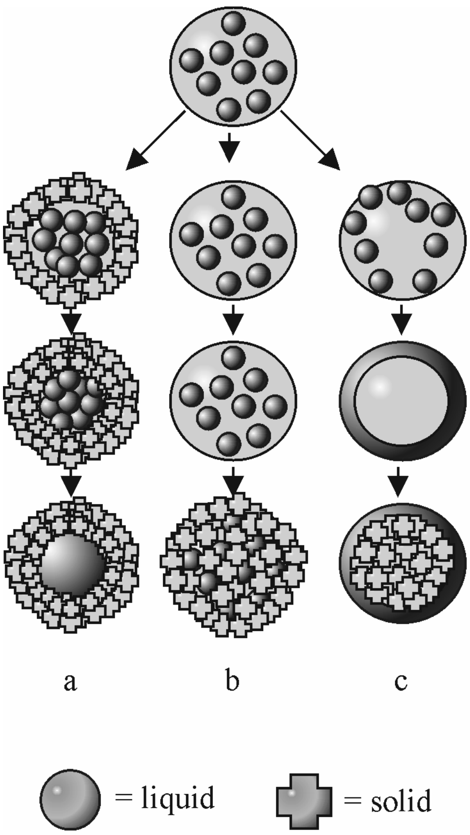


Figure 2: Solidification of a dispersion

In figure 2 a dispersion of the core material in the continuous shell phase is shown at the moment right after the dispersion has left the nozzle. The formation of small droplets in the nozzle is supported by the simultaneously expanded gas. The gas

(CO_2) is cooled during expansion via the Joule-Thomson phenomenon and efficiently removes heat from the droplets. A core-shell morphology (see figure 2a) is accessible if the solidification of the surface of the shell material is faster than the phase separation, while the solidification of the inner shell material is slower than the phase separation. In this case coalescence of the dispersed droplets of the dispersed core material may occur inside the already solidified shell. If the solidification of the outer and inner shell material occurs faster than the phase separation between shell and dispersed liquid droplets, closed composites, as shown in figure 2 b, are formed. The composites obtained consist of solid shell material in which the core material is encapsulated (matrix encapsulation). If solidification is slower than the phase separation open composites are generated (see figure 2 c), where the liquid may reach the solid surface or even cover the particle. So by changing the solidification and/or the velocity of separation different composite morphologies are generated.

3. Materials

In this investigation three material combinations were chosen as model substances. Palm fat (melting point: 58-60 °C; viscosity: $19.5 \cdot 10^{-3}$ Pa s at 75 °C) and castor fat (melting point: 88-92 °C; viscosity: $25.3 \cdot 10^{-3}$ Pa s at 100 °C) as well as polyethylene glycol 6000 (melting point: 56-63 °C; viscosity: $600 \cdot 10^{-3}$ Pa s at 80 °C) were used as shell materials. The two hard fats (lipophilic) as well as the hydrophilic polymer demonstrate the possibilities of composite production with different covering materials. Palm and castor fat are used in the food industry in large quantities. Among other applications PEG 6000 is frequently used in the cosmetic and pharmaceutical industries and as model substance for the PGSS process. The material properties under high pressure as well as the production of powders from pure substances have already been examined in numerous works [13-17]. In both hard fats water was encapsulated and rapeseed oil was bound in the polymer. These material combinations show the ability of the process both to form powderous w/o (water in oil) and o/w (oil in water) emulsions.

4. Experimental results

In the context of this work 165 experiments with the model systems palm fat/water, castor fat/water and PEG 6000/rapeseed oil were used to determine the inter-dependencies between process parameters of the PGSS process and particle and powder properties on the other hand. The evaluation of the three model systems showed that the PGSS process is suitable for the production of powderous composites with variable characteristics.

4.1. Particle size distribution

A clear influence of the pre-expansion pressure and the temperature in the spray tower on the particle size distribution of the castor fat/water powders was found. With increasing temperatures in the spray tower, the solidification process of the particles takes longer than at lower temperatures. At high temperatures in the spray tower agglomerated particles were observed. The effect of the pre-expansion pressure is more complex. A high pre-expansion pressure leads to a strong Joule-Thomson phenomenon. Droplets of the melted shell material and the liquid dispersed inside are cooled down faster at higher than at lower pressures. So at higher pressure smaller particles are formed and less or no agglomeration occurs.

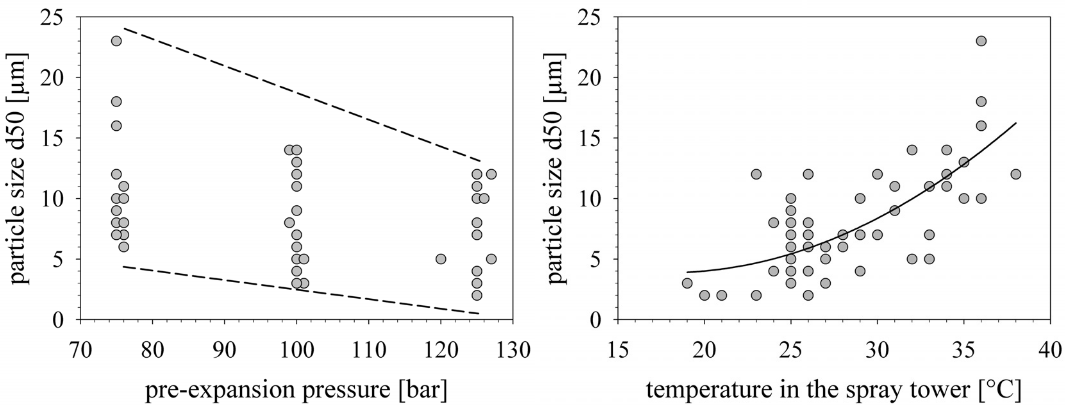


Figure 3: Particle size d50 versus pre-expansion pressure (left side) and temperature in the spray tower (right side), system: castor fat/water

4.2. Bulk density

The spray experiments were performed at three temperature levels (65, 90 and 125 °C) which can be recognized well in figure 4. Within these ranges the bulk density varies to different extents.

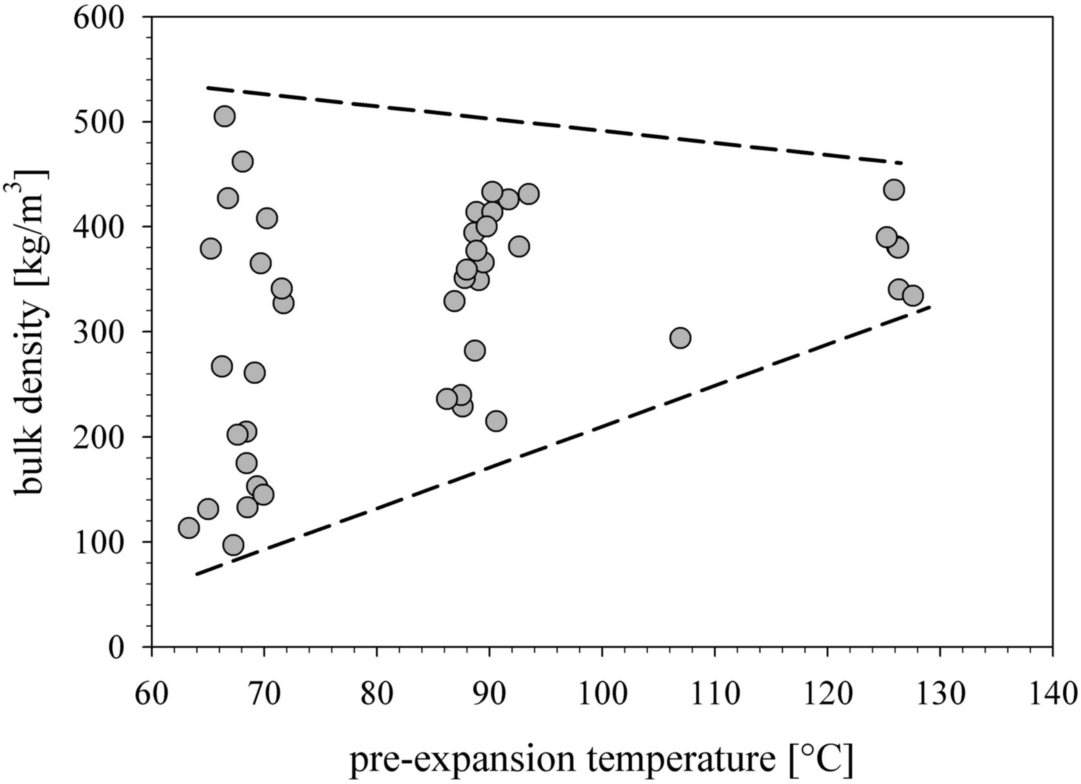
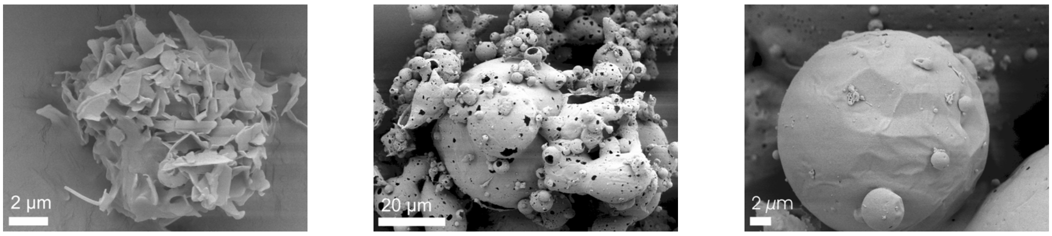


Figure 4: Bulk density versus pre-expansion temperature, system: PEG 6000/ rapeseed oil

With consideration of the complex influence of all other process parameters, it was possible to produce powders with a bulk density between 97 and 505 kg/m^3 at low temperatures before the nozzle. The variability of the bulk density is approx. 400 kg/m^3. This range decreases in the temperature range from 90°C on 250 kg/m^3 and lies in a range from 150 kg/m^{3}with a pre-expansion temperature of 125°C.

4.3. Morphology

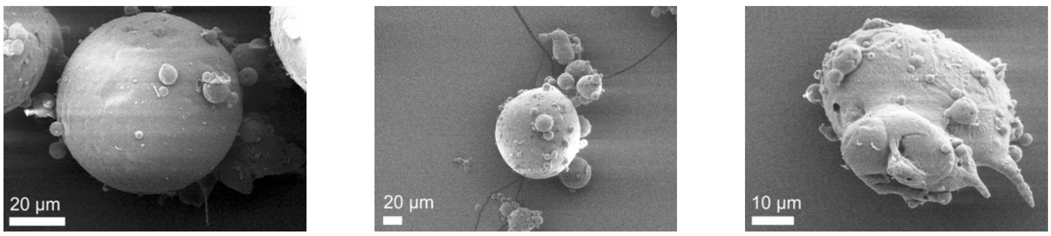
In order to investigate different morphologies photographs from a scanning electron microscope (SEM) were used. The particle geometry varies from needles to spheres. Three representative pictures of pure shell material particles are shown in figure 5.



needles foams spheres

Figure 5: SEM pictures of fat particles

For composite formation a lot of different combinations of materials were used. For example cherry liqueur was encapsulated in chocolate, soy sauce and an aqueous solution of Rooibos extract in palm fat and rum and honey in castor fat. Figure 6 shows some examples of castor fat/honey composites with a content of liquid honey up to 59 wt.-%.



20 wt.-% honey 47 wt.-% honey 59 wt.-% honey

Figure 6: SEM pictures of castor fat/honey composites

From left to right the amount of honey increases from 20 over 47 to 59 wt.-%. All particles have a spherical morphology. The surface of the spherical composites is completely closed and uniform.

5. Summary

The PGSS process runs at low temperatures with pressurized carbon dioxide. No organic solvents, emulsifying agents or other additives are necessary for the process. New food products can be generated by powderization and protection by encapsulation can be achieved. Encapsulation thus offers new possibilities for the release control of active substances. Even low melting shell materials can be frozen by fast and strong cooling (Joule-Thomson phenomenon). The low temperature retains components with high volatility (e.g. water and fragrances) effectively.

Since 1994, when the PGSS-process and acronym was presented first, its potential has successfully been investigated for numerous solids such as polyethylene glycols, powder coatings, monoglycerides, citric acid, vitamins, antioxidants, solid fats and green tea. Recently, it has been demonstrated, that the process is also suitable for the generation of solid-liquid composites. A series of compounds have been generated, e.g. palm fat powders that contain liquid soy sauce or castor fat powders that contain over 50 wt.-% of micronised water droplets. Not only fats were used as shell material, but also substances like natural or synthetic polymers and waxes.

As an innovated example from the food sector melted chocolate was transformed into fine powder and liquid aromas could successfully be encapsulated in globules of chocolate. Only a few micrometers in diameter, the chocolate globules hold tiny drops of aroma or even high-proof liquors inside and can be stored just like normal bars of chocolate. Now the first industrial products that contain the chocolate powder are on the market and numerous ways of application can be imagined, for instance combining the powder with fresh fruit or ice cream. Meanwhile the PGSS process has reached industrial standard and the first industrial plants have been put into operation.

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