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Influence of emulsion composition on the quality of spray dried orange oil

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INTRODUCTION AND OBJECTIVES

This study evaluates the influence of capsule material interactions on selected quality parameters of spray dried flavours. While the influence of various parameters like core to shell ratio, solid content, DE value and inlet and outlet temperatures has been presented in several publications (Bangs 1990, Jayusundera 2009, Madene 2006, Ré 1998, Reineccius 2001, Vega 2006), investigations on the influence of capsule material interactions in spray drying encapsulation are poorly available. On the other hand, the effects of polymer interaction are well known and actually used for example in the field of texturising of food or as a means of separation in biotechnology called membraneless osmosis (Tolstoguzov 2000, Tolstoguzov 2002, Benichou 2002, de Kruif 2001).

The phenomenon which leads to the so-called synergistic effect in mixtures of different polysaccharides can be attributed to a thermodynamic incompatibility of these polysaccharides leading to a sharp increase of the viscosity of their mixtures compared to the viscosities of the single polymers. Thermodynamic incompatible polymers build up water-in-water emulsions, with an interface which is preferably occupied by particles and hydrophobic substances (Turgeon 2003). If oily substances settle in these interfaces, the droplets might coalesce leading to a fast demixing of an emulsion (Tolstoguzov 2003).

In this study the influence of the composition of an emulsion composed of incompatible polymers as capsule material and orange oil as a model core substance is investigated by systematically varying relevant parameters. It is shown in which way the resulting spray dried powders differ in chosen parameters like surface oil or storage stability as a direct result of the emulsion formulation.

MATERIAL AND METHODS

The slurries were prepared with varying amounts of a modified OSA-starch (National Starch), methylcellulose (MC) Methocell A15 (food grade, DOW Chemicals) and Brazilian orange oil (density 0.83-0.85 g/cm^3) in distilled water (IEL, Bonn). After pre-blending with Ultra-Turrax T45, the samples were homogenized at 200 bar and spray dried within 2 hours at a inlet temperature of 180 °C and an outlet temperature of 90 °C. The samples were immediately weighed at aliquots of 5 grams in Petri dishes and sealed. Samples were stored at room temperature.

Design of experiments

The influence of incompatible biopolymers as capsule materials on selected quality parameters of spray dried orange oil was investigated by setting up a design of experiments with the following parameters using 3 levels of each design variable:

- Solid contend: 20 – 40 %,
- Starch in capsule material mixture: 90 – 100 %,
- Core to shell ratio: 1 : 3 – 1 : 6,

resulting in 20 spray dried samples including 6 center points.

Quality parameters of spray dried orange oil

The following parameters and the respective ratings were chosen to evaluate the effect of the polymer mixtures in the slurry on the spray dried orange oil (methods in brackets):

- Emulsion stability: in 1 [not stable] to 5 [stable]; (Visual evaluation).
- Powder yield: in % of solids in slurry
- Retention of orange oil: in % of oil in slurry; (Steam distillation of 10 grams redispersed sample).
- Surface oil: in % of oil in powder; (Washing of the powders with iso-hexane, followed by evaporation of the iso-hexane and weighing of the residue).
- Storage stability: in limonene content in % of initial content; (Gas-chromatographic analysis of oil, obtained by steam distillation of 5 grams of redispersed sample).

RESULTS AND DISCUSSION

The results, summarized in table 1, clearly indicate that the ratio of OSA-starch to Methylcellulose has an influence on the chosen quality parameters. The only exception is the powder yield. In this case it was not possible to find a correlation, as the determined yield for the 6 center points varied from 27 to 90 %, showing, that emulsions made from incompatible polymers result in strongly fluctuating powder yields.

In case of emulsion stability, the highest stability of more than 12 hours' duration could be observed for emulsions with 100 % starch as capsule material at every level of solid content. The higher the amounts of MC, the less stable were the emulsions. This effect could partially be counterbalanced by increasing the solid content of the mixtures. The increase in viscosity, by increasing the solid content, stabilises the emulsion because the coalescence, even in an incompatible system, is inhibited. This observation leads to the assumption that orange oil tends to preferably adsorb to the interface of the water-in-water-emulsions in this incompatible system. The result can be a more or less stable water-in-oil-in-water emulsion depending on for example the viscosity of the surrounding continuous phase.

Table 1: Parameters and their influencing factors

Parameter	Influencing factor
Emulsion stability	Solid content Starch : MC ratio
Powder yield	–
Retention	Solid content Starch : MC ratio Core : shell ratio
Surface oil	Starch : MC ratio Core : shell ratio
Storage stability	Solid content Starch : MC ratio Core : shell ratio

The retention of orange oil in the powders is influenced by all factors. While the interrelationship between solid content and core : shell ratio is well investigated (Ré 1998), influences of the material mixture is rarely discussed. In case of a known incompatible system, like in this study, it can be assumed, that the resulting structures (W/W-emulsions) lead to an accumulation of orange oil in the interfaces of the water-in-water-emulsions, which might then coalesce and destabilize the emulsion. This again leads to a well-known phenomenon, i.e. a decreasing retention with an increase in droplet size.

The surface oil content is influenced by the ratio of the polymers in the mixture and the well-known factor active to shell ratio (Vega 2006). In case of the surface oil, it can be expected, that an emulsion in which the oil droplets coalesce because of the incompatibility of the wall polymers will lead to high amounts of surface oils. In this study the quantity of surface oil was low for emulsions made with pure starch compared to any mixture of starch with MC at any level of solid content.

Following the results of this study, the storage stability can be increased, if only the starch is used as an encapsulating material. As soon as a water-in-water-emulsion is built up by two incompatible polymers, and the emulsion starts to coalesce, the storage stability sharply drops.
The other factors are interacting. At a core to shell ratio of 1 : 6 the solid content has no impact on the storage stability. The stability remains constant. In contrast to this the storage stability of formulas with high active loads (1 : 3) can be influenced by the solid content. The higher the solid content of the emulsions, the higher the storage stability of the powders.

CONCLUSIONS

From the results it can be concluded, that the emulsion composition influences almost all properties important for powder quality. While general effects are well investigated, one less apparent effect, i.e. polymer incompatibility, is sometimes neglected when creating slurries for spray drying. In this study it has been shown, that polymer incompatibility in emulsions for spray drying leads to often observed effects, like coalescence and emulsion instability, resulting in high drying losses and/or low storage stability, which are often described as results of bad emulsion stability without taking the polymer interactions in the emulsions into consideration.

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