DESIGN OF POWDERS WITH CONTROLLED RELEASE PROPERTIES USING THE CPF-TECHNOLOGY

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INTRODUCTION

A variety of substances is solid or highly viscous under normal conditions, but can easily be liquefied under the influence of CO_2-pressure. This range includes a number of food grade additives like fats, waxes and different kinds of emulsifiers. A concept for the production of powders with controlled-release properties using the CPF-process [1] is based on the possibility of liquefying solid mixtures in the high-pressure unit of a CPF plant and combining them with a powdery carrier. Before spraying, valuable ingredients like vitamins, food dyes or flavours can be blended with these additives, e.g. by means of an autoclave. The resulting product is a powder containing ingredients which are encapsulated in a solid or highly viscous matrix.

The same principle can be used for the encapsulation of flavours. Most solvents which are in industrial use for the formulation of flavours, like triacetine, propylenglycol or edible oil, can be solidified by mixing them homogeneously with 10 to 40 weight % of a food grade and pressure liquefiable solid. Usually, the result is a flavour-additive-mixture with a melting point close to the melting point of the involved additive.

II - MATERIALS AND METHODS

CPF-Technology [2] (Concentrated Powder Form) is a high pressure spraying process which allows to obtain powders with liquid concentrations up to 80 weight %. Therefore a gas, normally carbon dioxide, is dissolved under high pressure (80 to 250 bar) in the liquid. Then the gas-saturated solution is rapidly expanded in a nozzle. The gas is set free and thus very fine droplets of the liquid are formed. During the expansion a powdery carrier is added cocurrently to the sprayed liquid. The expanding gas causes an intensive mixing of the fluid droplets with the solid carrier. The liquid is adsorbed on the solid surface or in case of porous carriers, the fluid pours into the cavities and soaked particles are formed. The CPF-process produces free-flowing powdery products in combination with a high liquid load of the

particles. The technology was tested by using different liquids (essential oils, extracts, oleoresins) and carriers (starches, silic acids, celluloses, maltodextrines). The experimental results showed, that the CPF-process can be applied to low and high viscous liquids and to solid carriers with a broad range of particle sizes and bulk densities (50 – 1400 kg/m³).

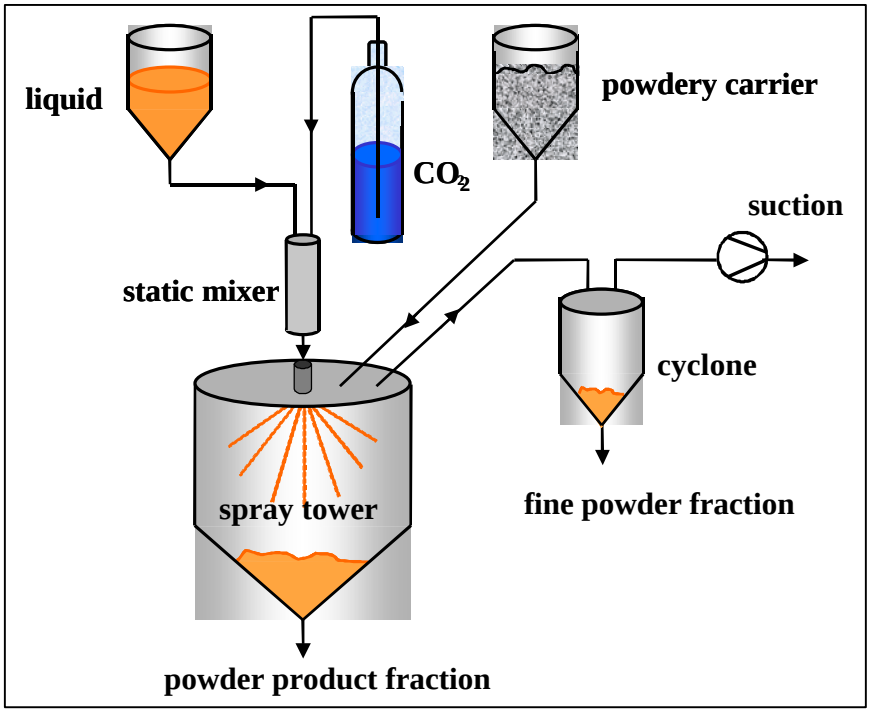

fig. 1 functional principle of the CPF-process

Release properties of powdery products were determined by measuring the concentration of encapsulated riboflavine (vitamin B2) which was released into a stirred aqueous phase during the experiment. Analyses were performed by means of a photometer at a wavelength of 450 nm. To observe the temperature controlled release of the ingredient, two kinds of tests were carried out. First was stirring at room temperature for up to 90 min. The extinction at 450 nm was measured every ten minutes. The values of released riboflavin were then compared to the values obtained when the solution was stirred under constantly increasing temperature from room temperature up to 80 °C. In this case, riboflavin values were determined every 10 °C and the current run time of the experiment was recorded. All tests were carried out in double. The results were accepted when the deviation of the measured values (extinction and time to the next 10 °C) did not exceed 10 %.

III – RESULTS AND DISCUSSION

The properties of powder formed release systems were investigated on the basis of the release of vitamin B2 (riboflavine) into a stirred aqueous phase. It was assumed, that other kinds of encapsulated ingredients (including flavours) show a similar release behaviour.

In the development of powders with specific release properties, the following targets were aimed at:

- stability against mechanical abrasion
- stability against release of ingredients while stirring in an aqueous phase at room temperature
- temperature controlled release of ingredients into an aqueous phase, in consequence of application of specific additives or additive-solvent-mixtures with defined melting points
- time controlled release of ingredients into an aqueous phase in consequence of application of additives or additive-solvent-mixtures with delayed solubility in an aqueous phase

For the following reasons the CPF process was particularly suitable for the targeted powders:

- Due to the functional principle of CPF, the processing of lipophilic blends is uncomplicated.
- Highly viscous blends, or blends which are solid at room temperature can be liquefied under pressure and/or moderate heating and subsequently handled and sprayed like a liquid.
- Formulation of ingredients is unproblematic. Flavours, for instance, can be solidified by mixing them with 10-40 % of a certain additive. The basic prerequisite for an efficient release system is a homogenious mixture of dissolved flavour and additive.
- Due to the atomised spray which is generated behind the nozzle, the pores of the powdery carriers are filled completely by the process. As a result, CPF allows comparatively high loads of the carriers.
- The functional principle of CPF allows the production of release-concept powders in a one-step process, without a subsequent coating.
- Compared to a classical coating process, only small amounts of additives are required.

In the following diagrams **the influence of an increasing percentage of additive in the sprayed blend** on the observable release behaviour is shown (additive: hardened fat; solvent: edible oil; carrier: maltodextrine). Similar results could be observed in all cases of blends although the percentage of additive which is required to ensure a sufficient encapsulation (**fig. 5**) varied from 10 to 40 % (currently: 26 additives tested).

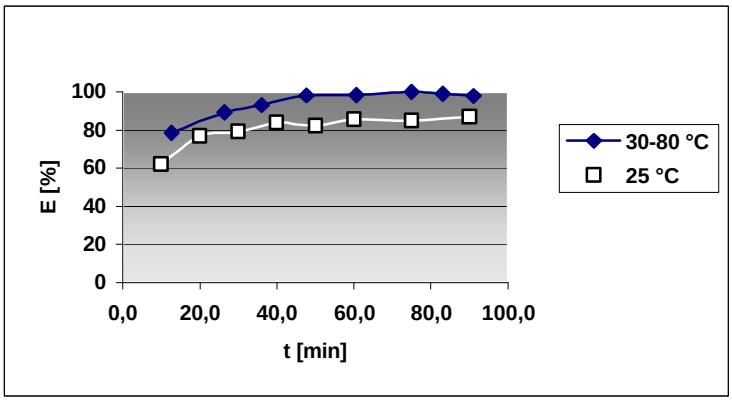

fig. 2 The sprayed blend contains 5 % additive – nearly no observable difference between the curves with constant stirring at 25 °C compared to stirring at a temperature gradient (measuring points recorded every 10 °C).

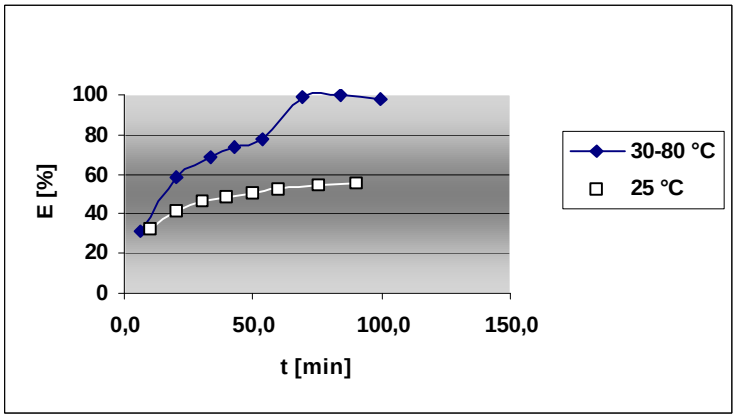

fig. 3 The sprayed blend contains 10% additive – release delayed while stirring at 25 °C, the temperature gradient curve shows a bend around the melting point of the blend.

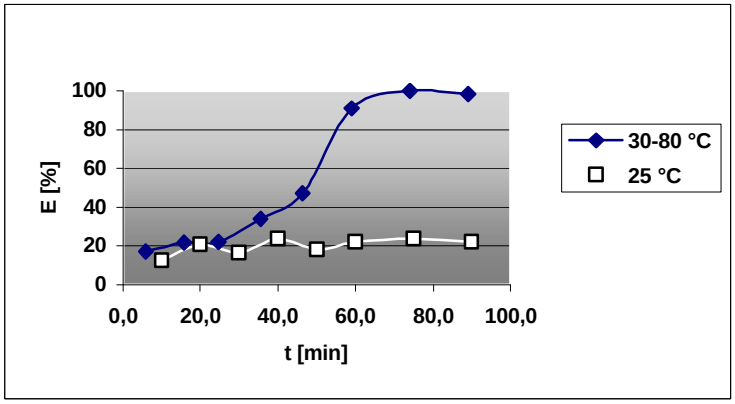

fig. 4 The sprayed blend contains 20% additive – release while stirring at 25 °C remains constant at 20%; temperature gradient curve shows a sharp bend close to the melting point of the blend.

Regarding **fig. 4,** it is obvious that the powdery product is not 100 % sealed. Depending on the additive which was used, 10 to 20 % of the encapsulated vitamin was released under constant stirring at room temperature during the first 10 minutes. Thereafter, the measured concentration of riboflavin remained constant until the end of the test (90 min). This observation is assumed to be based on fractional abrasion from the particles on the one hand, and the possibility of incomplete encapsulated riboflavin crystals which are quickly dissolved when the powder comes in contact with water, on the other hand. The contribution of the last-mentioned process can be minimized when more finely ground crystalline material is used for encapsulation.

Regarding **fig. 2 to 4,** it can also be seen that a temperature controlled release effect at a chosen temperature can not be generated by means of a user-defined percentage of a given additive with a solvent. In fact, when suitable additives are homogeneously blended with food grade solvents in a percentage of 10 to 40 % (depending on the nature of the additive) solid blends are obtained with a melting point close to the melting point of the additive itself. In this constitution, the blend results in powdery products showing a release behaviour similar to **fig. 4**. Lower percentage of the additive usually leads to results as displayed in **fig. 3** or **fig. 2**.

As a consequence of this observation, temperature controlled release at a certain temperature can only be achieved by the application of suitable additives with a melting point within the desired range. **fig. 5** shows examples for release curves at different temperatures using different food grade emulsifiers, fats or waxes.

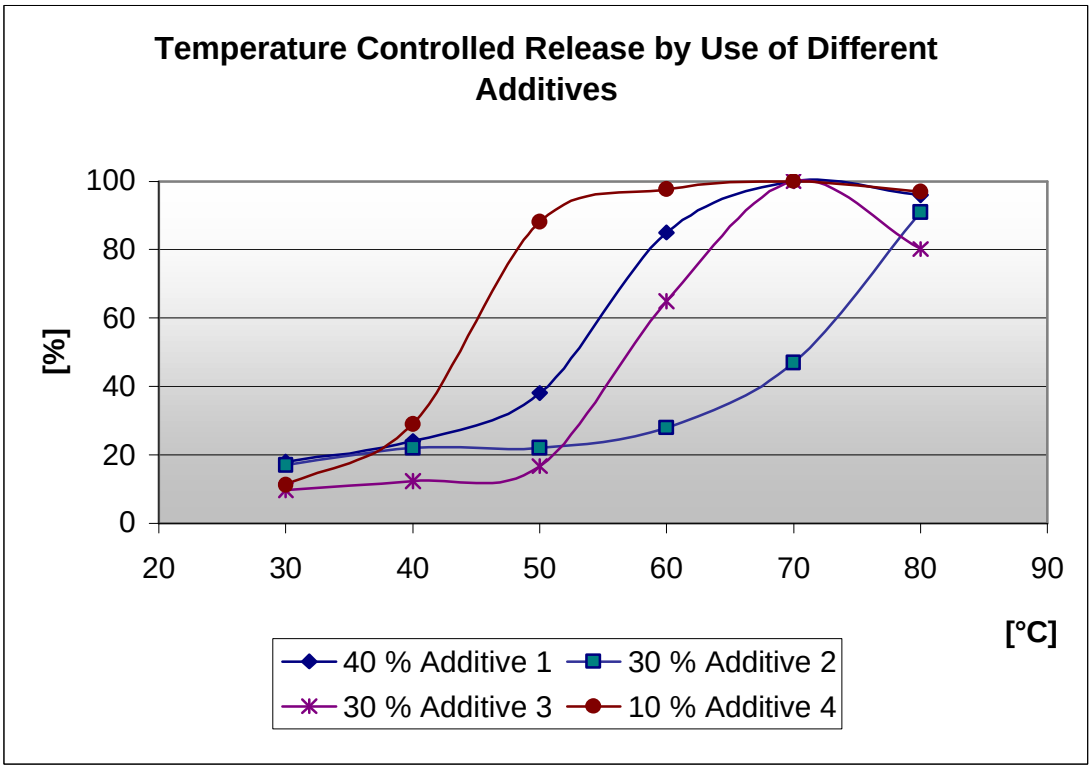

fig. 5: Examples of temperature controlled release behaviour of encapsulated riboflavin caused by different additives in edible oil as a solvent.

In contrast to different additives, powdery carriers had none or little influence on the release behaviour of the final products. When the same liquefiable blend was used to manufacture powders with different carriers like maltodextrines, starches, celluloses or silicic acids. The results in all cases were powdery products with release curves displaying no statistical significant deviation, although, the carriers show basical differences in view of properties like e.g. porosity, bulk density, liquid loading or solubility in water.

IV - CONCLUSION

The tests show that an effective encapsulation can easily be realised in a one step process, by blending a food grade solvent with a suitable food grade additive. Although the encapsulation is not 100 % seal, the process is to be viewed as useful for a great variety of different applications. In addition to that, previous tests have shown, the loss of volatile components e.g. in flavours during processing with the CPF-technology is significantly reduced when thickening additives are homogeneously mixed with the flavours [3].

As an example, a possible application area for CPF-release-powders are pulverised flavours for bakery products. The flavours can be formulated in blends of synthetic or refined edible oil with 10-40 % of a suitable liquefiable additive. The use of silicic acid as a carrier results in free flowing products, containing up to 80 % of the flavour-additive-blend. The powdery flavours are stable, sufficiently encapsulated, dust free and consist up to 55 % of the flavour formulated in edible oil. Under the influence of elevated temperatures during baking, flavours release and permeate the dough. As a result of the formulation, the loss of flavours during the baking is significantly reduced compared to the application of a liquid flavour. Silicid acid is applicable as a carrier in bakery products. The pulverisation of flavours which are formulated in other food grade solvents is also possible.

Other possible areas of application are pulverized flavours for yoghurts and instant soups or encapsulated sensitive ingredients like vitamins or unsaturated fatty acids in nutraceuticals. Future experiments will show, if the CPF-technology can also be used to encapsulate pharmaceutical active ingredients for pH-controlled release.

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