# Microencapsulation of fruit juices by spray-drying: red prickly pear as study case

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## Abstract

Spray-drying has been widely applied for microencapsulation of biological material. Fruit juices are a source of biomolecules as carbohydrates, proteins, as well as of nutraceutical compounds with interesting antioxidant properties. Red prickly pear juice has been used as an example for fruit juices microencapsulation studies because of its high antioxidant red pigment content which was used to evaluate spray drying performance. Spray drying conditions were optimized using glucose syrup as microencapsulating drying aid in order to obtain a non-sticky powder with high pigment content. A high color strength (4.0) fruit juice powder was obtained of low water content (4 %) and high bulk density (0.6 g/ml). Powder particles average size were 2-4 μm. Spray drying conditions used and microencapsulation agent selected were compared with those used for spray drying different fruit juices in advance. Spray drying of fruit juices as the red prickly pear juice studied, allows obtaining free flowing microencapsulated juice powders useful for the food industry.

## Introduction

Spray-drying is the most common and cheapest technique to produce microencapsulated food materials. Equipment is readily available and production costs are lower than most other methods. Compared to freeze-drying, the cost of spray-drying method is 30–50 times cheaper. The process has usually proved not only economic but also efficient. During this drying process, the evaporation of solvent, that is most often water, is rapid and the entrapment of the interest compound occurs quasi-instantaneously. Spray-drying microencapsulation process must rather be considered as an art than a science because of the many factors to optimize and the complexity of the heat and mass transfer phenomena that take place during the microcapsule formation (Gharsallaoui et al., 2007). Foods to be spray dried can be subjectively classified into two broad groups: non-sticky and sticky. In general, non-sticky materials can be dried using a simple dryer design and the final products remain free flowing. Natural sugar and acid-rich foods such as fruit and vegetable juices, and honey are sticky materials difficult to dry under normal spray drying conditions (Adhikari et al., 2004).

The spray drying of fruit juices is limited by the stickiness as the major reason. The sticky behaviour of sugar and acid-rich materials is attributed to low molecular weight sugars such as fructose, glucose, sucrose and organic acids such as citric, malic and tartaric acid which constitute more than 90% of the soluble solids in fruit juices and purees. These compounds have low glass transition temperatures and, during drying, juice may either remains as a syrup or stick on the drier chamber wall. This might lead to low product yields and operational problems (Bhandari et al., 1993). For preventing of stickiness a drying aid have to be used. The most widely used to obtain fruit juice powders are partially hydrolyzed starch products: maltodextrins, with dextrose equivalent (DE) of less than 20, and dried glucose syrup with DE higher than 20. Examples of fruit juices spray dried with maltodextrins and dried glucose syrups are watermelon (9 DE) (Quek et al., 2007), pineapple (10 DE) (Abadio et al., 2004), mango (20 DE) (Cano-Chauca et al., 2005), or acerola (25 DE) (Righetto et al, 2005).

*Opuntia spp*. fruits known as prickly pears or cactus pears are consumed fresh, or incorporated in jams, juices, etc. Prickly pear has a pleasant fresh fruit flavour and aroma. It contains glucose, fructose, fibres, betalains as main pigments, carotenes, high levels of calcium, magnesium and vitamin C, and interesting functional compounds like quercetin (Piga, 2004). *Opuntia spp*. fruit extracts have shown important anti-inflammatory, hypoglycemic, physiological antioxidant, cancer chemoprevention, and neuroprotective effects (Kim et al. 2006, Zou et al., 2005).

Previous studies of our research group focused in the pigment characterization of fruits of different *Opuntia* species showed that *Opuntia stricta* fruits have high level of betalains (80 mg/100 g fresh fruit) with betanin and isobetanin as the main colorant components. These pigments levels were even higher than that shown by red beets (*Beta vulgaris* L.), which are used to obtain the food colorant additive beetroot red, betanin, in the European Union E-162 (Castellar et al., 2003). A concentrated red-purple betacyanin food colorant from *Opuntia stricta* juice was obtained and it presents a vivid red-purple color which is clearly distinguishable from the colors shown by other commercialized natural red food colorants (Castellar et al., 2006).

The aim of this work was the study the microencapsulation of fruit juices using the juice of *Opuntia stricta* prickly pears as study case.

## Material and methods

### *Opuntia stricta* fruit juices

Mature prickly pear fruits of *Opuntia stricta Haw.* were harvested in Murcia (south-east of Spain), and homogenised with an Ultraturrax Ika Labortechnik T25 basic (Staufen, Germany). Homogenised fruits were centrifuged for 10 min at 15000 x g, in a Z383K Hermle centrifuge (Wehingen, Germany) refrigerated at 10 ºC, and supernatants were used as fruit juice.

### Spray drying experiments

Fruit juice was mixed with dried glucose syrup (DGS) at different ratios. Dried glucose syrup (DGS) Glucidex 29 with an average DE 28-31 was a kind gift of Roquette Laisa España, S.A (Valencia, Spain). Liquid samples were spray dried with a Büchi Mini Spray Dryer B-290 (Flawil, Switzerland). Maximal evaporative capacity was 1 l/h at inlet air temperature of 220 ºC. Different spray drying experimental conditions were assayed and after each experiment powder was collected carefully from the cyclone and the collection vessel. Powders fixed on the wall of the drying chamber or in the exhaust tube were neglected. Powders were placed in Petri dishes and stored at room temperature (25 ºC) in a desiccator filled with anhydrous silica gel. Experiments were done at least in triplicate.

### Measurements and calculations

Color strength of the juice was defined as the absorbance at 535 nm of a 1% (v/v) solution. Color strength of the powder was defined as the absorbance at 535 nm of a 1% (w/v) solution. Absorbance was determined with an 8453 UV-visible Agilent spectrophotometer (Waldbronn, Germany). Total soluble solids (ºBrix) of liquids were measured by a Zeiss Opton hand refractometer (Jana, Germany). Water content was measured by Karl-Fischer titration with a 787 KF Titrino (Metrohm, Herisau, Suiza). Drying yield was calculated as (grams of powder obtained x 100 / grams of feed solid-content). Color yield was calculated as (color strength of powder obtained x grams obtained powder x 100 / color strength of juice x juice volume in ml used). Bulk density of powders was measured by weighting 5 g of a sample into a 10 ml graduated cylinder. Cylinder was vibrated to obtain a near optimum packing, and when a steady volume was reached the bulk density was calculated as g/ml. Scanning electron microscopy images were obtained for all the spray-dried powders to examine particle morphology. The Scanning Electronic Microscopic S-3500N Hitachi HighTechnologies (Tokyo, Japan) worked with a voltage of 5 kV with the lens at 7 mm.

## Results and Discussion

The spray dryer worked in open-cycle, with co-current flow of hot air and sprayed material. In this unit, atomization is done with a two fluid nozzle, using compressed air. Spray nozzle has an orifice of 0.7 mm in diameter. A glass cylinder (50 x 15 cm) is used as drying chamber. Feed temperature was thermostatized at 20 ºC. The instrument settings used were: inlet air temperature (120-160 ºC), liquid feed rate (0.72 l/h), spray air flow-rate (0.47 $m^3$/h), and aspirator flow-rate (36 $m^3$/h, 90%). Selections of values are in a combined system influencing the product properties. Dryer chamber design and air flow-rate provide a droplet residence time of about 1 s.

Initial trials to get powders from *O. stricta* juice (12 ºbrix) at 120 ºC inlet air temperature were not successful, and the addition of a microencapsulating agent was necessary. Microencapsulation was done using dried glucose syrup with a dextrose equivalent of 28-31. Spray drying variables were selected to get high yields on powder recovery but keeping unchanged the colorant components (betacyanins) sensitive to high temperatures and oxygen. Two fruit juice concentrations were selected, 10% (v/v) and 80% (v/v), and different amounts of drying aid were assayed. As can be seen in Figure 1 the higher juice concentration assayed led to low drying yields due to stickiness problems. Drying yield was 18% with 80% (v/v) while it was 52% with 10% (v/v). This effect was observed at all microencapsulation agent concentrations assayed. The ratio of microencapsulation agent concentration versus juice concentration was changed from 0 to 3.33 in this study. Concentrations were expressed as final ºbrix. A minimum DGS/juice ratio of 1.66 was needed to get a free flowing microencapsulated powder. Pigments were stable during drying and thus microencapsulated powders of high colour strength were obtained.

**Figure 1. Powder stickiness on dryer chamber during spray drying of 10% (v/v) *O. stricta* juice (left) and 80% (v/v) *O. stricta* juice (right). Ratio DGS/juice is 1.66.**

A new set of experiments were conducted at a higher inlet air temperature of 160 ºC, obtaining better drying yields (60%) and powders with the highest colour strength (4.0). Powders had a low water content (4 %) and high bulk density (0.6 g/ml). SEM photographs showed that powder particles had round shape and due to the drying process they show crust shrinkage. The average size was between 2-4 μm.

In this study microencapsulation was done with a DGS/juice ratio of 1.66. Table 1 compares the conditions reported for different fruit juice microencapsulation studies.

| Fruit | Microencapsulating agent concentration | Juice concentration | Inlet air-temp (ºC) |
|---|---|---|---|
| Pineapple | Maltodextrin 10 DE 10 ºBrix | 12 ºBrix | 190 |
| Mango | Maltodextrin 20 DE 12 ºBrix | 12 ºBrix | 160 |
| Watermelon | Maltodextrin 9 DE 5 ºBrix | 12 ºBrix | 155 |
| Acerola | Glucose syrup 25 DE 20 ºBrix | 17 ºBrix | 120 |

**Table 1. Spray drying conditions used in literature**

## Conclusions

A high color strength (4.0) microencapsulated powder of *Opuntia stricta* fruit juice was obtained with glucose syrup 29 DE. It has low water content (4 %) and a high bulk density (0.6 g/ml). Selection of spray drying conditions was fundamental to obtain high microencapsulation yields.

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