# Nano particle encapsulation of blueberry by Inulin and β-cyclodextrin

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*Abstract*— **In this study, blueberry essential oil as a core material was nanoencapsulated with Inulin and β-cyclodextrin at a ratio of 1:5. Oil in water nano-emulsions were used by ultrasonic liquid processors and then transformed to encapsulated powder in spray dryer. Results show that amount of β-cyclodextrin had a tremendous effect on emulsion droplet size, encapsulation efficiency and morphology of capsules.**

***Keywords-Blueberry; nano-emulsion; ultrasound; spray dryer; Inulin; β-cyclodextrin.***

## I. Introduction

Encapsulation is a rapidly expanding technology with a lot of potential in different areas including pharmaceutical and food industries. It is a possible by which small particles of core materials are packaged within a wall material to form microcapsules (1). Application of encapsulation has become widely accepted in the flavor industry, because most flavors are volatile and chemically unstable in the presence of oxygen, light, water and heat (2,3,4).

Blueberries are one of the few fruit crops that are native to North America and, next to strawberries, are the second most important berry in the U.S (5). Among berry fruits, blueberries (Vaccinium corymbosum L.) are considered to be a good source of phenolic compounds and praised for their high antioxidant activity scores (6).

Inulin is a carbohydrate built up from β (2,1)-linked fructosyl residues mostly ending with a glucose residue and it is present as storage carbohydrate in a large number of plants (7,8).

Cyclodextrins are cyclic oligosaccharides composed of glucose units (9,10). The best-characterized forms are α, β and ɣ-Cyclodextrin consisting of six, seven and eight D-glucose units, respectively. Cyclodextrins have found numerous applications in food industry. They are used for the removal and masking of undesirable components and controlled release of desired food constituents (11). Cyclodextrins are used in food formulations for flavor protection or flavor delivery. Most natural and artificial flavors are volatile oils or liquids, and complexation with cyclodextrins provides a promising alternative to the conventional encapsulation technologies for flavor protection. β-Cyclodextrin as a molecular encapsulant allows the flavor quality and quantity to be preserved to a greater extent and longer period compared to other encapsulants and provides longevity to the food item (12).

It has been well documented that emulsion droplet size (EDS) plays an important role in the retention of volatiles and surface oil content of encapsulated powders during spray drying (13,14,15,16). It has been proved that the lower the emulsion size, the higher is the encapsulation efficiency. Accordingly, many emulsion properties such as stability, rheology, and color, depend on the EDS and size distributions (17,18). Based on EDS, emulsions can be divided into micro - (10–100 nm), mini (nano) - (100–1000 nm) and macro-emulsions (0.5–100 μm) (1). Nano-(submicron) emulsions are kinetically stable systems that can be transparent (EDS < 200 nm) or ‘‘milky” (EDS - 500 nm) (19,20), and because of their very small EDS and high kinetic stability, they have been applied in various industrial fields, for example, personal care and cosmetics, health care, pharmaceuticals, and agrochemicals (21,22).

Spray drying is the most common methods used for microencapsulation because it is economical. It is also one of the oldest encapsulation methods used originally in the 1930’s to encapsulate flavours using gum acacia (23).

The objectives of this study were production of blueberry essential oil nano-emulsions by ultrasound and to spray dry them in order to investigate the properties of encapsulated powder. It should mention that more studies were done for encapsulation of core materials by β-cyclodextrin using inclusion complex. But we used a new method for β-cyclodextrin in this study.

## II. Materials and methods

### A. Materials

In this study, blueberry essential oil (Fermotec, Holland) was used as the core material. The wall materials were Inulin (Sigma-Aldrich, USA) and β-cyclodextrin (Sigma-Aldrich, USA). N-Hexane, Isopropanol and Tween 80 were purchased from Merck Company (Germany). Distilled water was used for the preparation of all solutions. All general chemicals used in this study were of analytical grade.

### B. Preparation of emulsions

20% of wall materials were dissolved in distilled water by magnetic stirring at 60 ˚C. They were kept overnight in ambient temperature in order to warrant a full saturation of wall materials. Blueberry essential oil in the ratio of 1:5 (core: wall) and 1% of Tween80 were added to emulsions. After that, they were stirred by magnetic stirring for pre-emulsion preparation.

### C. Ultrasonication

An Ultrasonic Liquid Processor (Model S-4000-010, USA) was used in this study for transforming pre-emulsion to nano-emulsion that equipped with an ultrasound probe with 4.8 mm in diameter. It was operated at 24 KHz for 130s.

### D. Emulsion droplet size analysis

The size distribution of emulsion droplets was determined by Stabisizer (Model PMX200C, Germany). Nano-emulsions were diluted by distilled water at a ratio of 1:40.

### E. Spray draying

The emulsions were immediately dried by pilot-plant spray drier (Model B-191, Buchi, Switzerland). The spray drier was operated at 120 ˚C inlet temperature and 65 ˚C outlet temperatures. Ultimately, dried powders were collected and stored at 4 ˚C for further analysis.

### F. Scanning electron microscopy of encapsulated powder

Microstructural properties of the encapsulated powder were evaluated by scanning electron microscopy (Model S360 Mv2300, England). Powders were placed on SEM stubs using a two-sided adhesive tape. The samples were coated with gold using Auto sputter coater (Model E5200, Bio-RAD, England) and samples analyzed at voltage of 15 kV. The images were obtained with instrument's software installed on a PC connected to the system.

## III. Results and discussion

### A. Emulsion droplets size analysis

Results revealed that amount of wall materials had very significant difference ($p<0.01$) with emulsion droplets size (Table 1). Using β-Cyclodextrin (25%) and Inulin (75%) as wall material produced the smallest emulsion size. While Combining Inulin(25%) and β-Cyclodextrin(75%) led to increase emulsion droplets size. It could be related to change spatial structure or coalesce emulsion droplets. Also it could be related to amount of β-Cyclodextrin. Whereas increasing β-Cyclodextrin could lead to increase emulsion droplet size.

Klaypradit and Huang (2008) encapsulated fish oil with different concentration of wall materials (chitosan, maltodextrin and whey protein isolate) by ultrasonic atomizer. They found that increasing amount of chitosan, emulsion droplets size decreased. Our results were similar to their results.

Table 2.

| **treatments** | **Influence of wall materials on emulsion size and encapsulation efficiency** | |
|---|---|---|
| | ***Emulsion size (nm)*** | ***Encapsulation efficiency(%)*** |
| Blueberry+β-CD(75%)+ Inulin(25%) | $72.25 \pm 1.81^{d}$ | $98.73 \pm 0.02^{c}$ |
| Blueberry+β-CD(25%)+ Inulin(75%) | $70.63 \pm 2.55^{d}$ | $99.13 \pm 0.06^{a}$ |

$^{a-d}$ Means within the same column fallowed by different letters are very significantly ($p<0.01$) different

### B. Encapsulation Efficiency

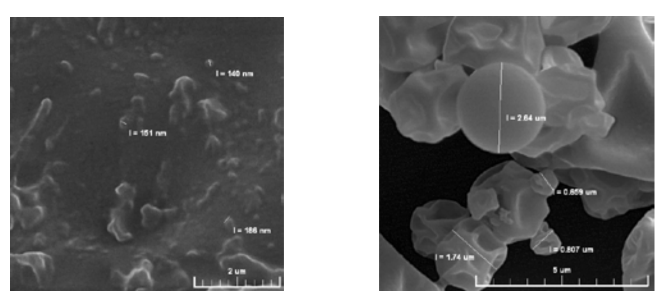
When blueberry essential oil was encapsulated by Inulin and β-Cyclodextrin, results showed that samples containing 75% of Inulin and 25% of β-Cyclodextrin had the highest encapsulation efficiency (table.1). That cause could be interaction between Inulin and β-Cyclodextrin that had the best capacity for preventing instability of emulsions and it was the most resistant sample. Also, it was found that Inulin covered blueberry essential oil better that β-Cyclodextrin.

On the other hand, there is an inverse relationship between encapsulation efficiency and emulsion droplets size. Namely, increasing emulsion droplets size leads to decrease encapsulation efficiency.

Kaushik and Roos (2007) was studied encapsulation of limonene in freeze drying by various matrices containing of gum Arabic, sucrose and gelatin. They found that increasing emulsion droplets size decreased encapsulation efficiency. In this study, powders containing encapsulated limonene by gum Arabic had the highest encapsulation efficiency.

### C. SEM photos analysis

"Figure.1" shows morphology of capsules. The results indicated that incorporating Inulin with β-Cyclodextrin in two different ratios lead to produce powders with different microstructural properties. In other words, Inulin had a profound influence on the structure and surface morphology of encapsulated powders. Photos showed that powder particles containing Inulin (25%) and β-Cyclodextrin (75%) had spherical surface without shrinkage that its reason was increasing β-Cyclodextrin in emulsions. On the other hand, this ratio produced Nano-capsules more. Increasing β-Cyclodextrin, while on the one hand reduced encapsulation efficiency, on the other it produced Nano-capsules. It is showed that Inulin had a profound effect on increasing encapsulation efficiency and forming particles with more dents and shrinkage.



A B

Figure 1. SEM photographs of blueberry essential oil encapsulated powders A: blueberry essential oil encapsulated powders containing β cyclodextrin (75%) and Inulin (25%), B: blueberry essential oil encapsulated powders containing β-cyclodextrin (25%) and Inulin (75%)

## IV. Conclusion

The finding of our study showed obviously that using ultrasonic waves with 24 KHz intensity for 130s produced emulsions with diameter of 70 – 75 nm and increasing particle size, after drying, can attribute to instability of emulsions during spray draying. Considering two different wall materials, we found that decreasing amount of Inulin led to increase encapsulation efficiency and decrease emulsion droplet size. However, other different ratios of wall material for decreasing emulsion droplet size and producing more Nano-capsules are to be studied further.

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