# Encapsulation of antioxidant compounds extracted from unused chokeberries by extrusion, co-crystallization, and freeze-drying

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Berries are among the richest sources of polyphenolics, which have been the focus of numerous studies with regard to their putative impact on human health. Black chokeberry (*Aronia melanocarpa*) belongs to the latter group of berries. Many berry species may be consumed fresh, but the fruits of some berry plants are not suitable for eating because their flavor is less favorable or even unacceptable by the consumers. Consequently, all harvests of such berries have to be processed into other products. Chokeberries are used for making fruit wine; they are also dried for herbal teas, used for flavoring and coloring beverages or yoghurts, and added to juice blends for color and for strengthening antioxidant properties. However, biorefining of unused chokeberry into higher added value ingredients is an important task.

Therefore, considerable effort has been devoted to the extraction of phenolic compounds from *A. melanocarpa* in order to use the extracts as dietary supplements or as food colorants. A literature search revealed that the number of publications on the key words "antioxidant" and "chokeberry" has strongly increased in the last five years emphasizing the growing interest in the topic. However, the effectiveness of polyphenols depends on preserving the stability, bioactivity, and bioavailability of the active ingredients. There are unsaturated bonds in the molecular structure of polyphenols, which makes them susceptible to oxidants, light, heat, pH, water, and enzymatic activities. They oxidize very quickly, leading to the progressive appearance of a brown color and/or unwanted odors with a considerable loss in activity. In addition, many phenolic compounds show limited water solubility and have an unpleasant taste, which must be masked before their incorporation in foodstuffs or oral medicines. Microencapsulation is one of the techniques used for enhancing the shelf life and stability of phenolics.

Among encapsulation technologies, freeze-drying is one of the most widely used for food industry due to its suitability for drying of heat sensitive compounds, since it conserves almost intact the initial functional properties of these components. However, the drying technique and the material used as coating usually affect the retention capacity of compounds within the matrix. Therefore, it is of great importance to properly select both, the coating material and the encapsulation technique in order to maximize the incorporation and retention of the functional compounds within the encapsulation matrix. Extrusion also proves to be a simple, precise, efficient, and economical method for obtaining particles with encapsulated bioactives. The method is based on formation of small particles by the use of forces, which disrupt the liquid filament at the tip of a needle (Manojlovic´ *et al*, 2008). It has great potential for controlled preparation of microbeads with appropriate organoleptic and pharmacological characteristics and uniform size. Among numerous carriers for microencapsulation, sodium alginate has attracted great attention, because of its excellent biocompatibility, possibility to delay the release of actives compounds, as well as its biodegradability and non-toxicity (Stojanovic´ *et al*, 2012). Encapsulation via co-crystallization is a relatively new method that offers an economical and flexible alternative since the procedure is relatively simple. During recent years, a small number of studies have reported on the encapsulation process by co-crystallization, in which the crystal structure of sucrose is modified from a perfect crystal to a conglomerate. This structure provides a porous configuration which can accept the addition of a second ingredient. In general, co-crystallization improves solubility, wettability, homogeneity, dispersibility, hydration, anticaking, stability, and flowability of the encapsulated materials.

To the best of our knowledge, literature data for encapsulation of chokeberry polyphenolic compounds are limited. In a very few studies, chokeberry polyphenols were encapsulated via inclusion complexation (Howard *et al*, 2013) or stabilized via different drying methods. Thus, the objective of this work is to optimize a new method for unused chokeberries application in food industries based on the extraction of phenolics compounds and their subsequent encapsulation by extrusion or co-crystallization or freeze-drying.

According to a previous comparative study on different extraction techniques to recover polyphenols from chokeberries, microwave assisted extraction exhibited the highest yield in total phenolic content. In this work, the extraction conditions used were based on a previous study on optimization of phenolic compounds extraction. Extrusion was applied for encapsulation of chokeberry extract in calcium-alginate gel beads. Alginic acid sodium salt was dissolved in the prepared extract. Chokeberry extract containing calcium chloride was used as a collecting solution. For the co-crystallization process, sucrose and water were used each time to prepare syrup. Sugar and water were mixed and then heated and stirred to make syrup. The temperature of the syrup was monitored continuously and once it reached 128 °C, the syrup was removed from the heating and a known quantity of extract was rapidly added. The mixture was vigorously agitated for a short time and was then placed in a water bath at room temperature until the dry agglomerates were obtained and the temperature of the co-crystals decreased to below 60 °C. For the freeze-drying experiments, mixtures of maltodextrin (13.5 DE):skim milk powder (50:50) and maltodextrin (13.5 DE):whey protein concentrate (50:50) were used as wall materials.

Central composite designs were applied to determine the effects of various parameters on encapsulation efficiency and yield (Table 1). Characterization of powder properties (moisture content, bulk density, solubility, hygroscopicity, particle size) was accomplished to determine if successful development of the product was achieved.

**Table 1.** Investigated parameters for all encapsulation methods studied.

| Parameter | Range |
|---|---|
| Extrusion | |
| Calcium chloride concentration (%, w/v) | 1.0 – 2.5 |
| Alginic acid sodium salt concentration (%, w/v) | 2.0 – 4.0 |
| Extract concentration (%, w/v) | 0.5 – 20.0 |
| Co-crystallization | |
| Solids concentration in the extract (Brix) | 35 – 70 |
| Solids in the extract/sucrose (g/g) | 0.1 – 0.7 |
| Freeze-drying | |
| Time at -40°C (days) | 1 – 3 |
| Wall to core material ratio (g/g) | 2.3–9.0 |

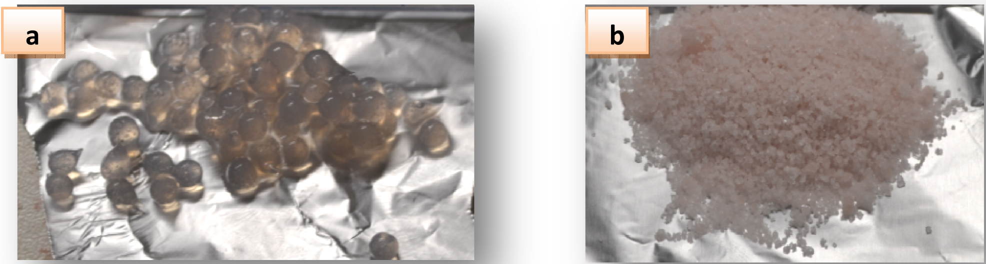


**Fig. 1.** Chokeberry extracts encapsulated by extrusion (a) and co-crystallization (b).

**References**

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