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# Effect of freezing with temperature fluctuations on microstructure and dissolution behavior of freeze-dried high solid systems

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**Abstract**

This study demonstrates the potential to control ice crystal formation in highly concentrated system for process design of energy efficient freeze-drying technology. I, the effect of freezing with temperature fluctuations on the microstructure and dissolution of aerated and non-aerated freeze-dried concentrated coffee (50 and 60% w/w) is presented. Samples were either frozen at -40˚C or subjected to fluctuating temperatures between -40 and -20˚C prior to drying. Total freezing time was kept constant for both freezing profiles. Microstructural characteristics of the freeze-dried solids were revealed using scanning electron microscopy (SEM) and high speed camera was used to record the dissolution process. Solution with 60% solids nucleated at lower temperatures (< -20˚C) and developed smaller pore sizes (by 70%) than 50% concentration. SEM micrographs of the freeze-dried solids showed the formation of voids similar to the shape of crystal dendrites in less concentrated coffee sample. Aerated systems were characterized by growth of small dendrites (20-40 μm) within large air bubbles which diameter varied from 200-1000 μm. Meanwhile, collapsed microstructure was observed in 60% coffee but was not visible on aerated sample. Temperature fluctuations doubled the observed pore sizes in both concentrations which effectively accelerated the dissolution process. Morphology of ice crystals is also affected by the periodic temperature fluctuations with growth of hexagonal ice crystals (50-100μm) was evidence from the SEM images.


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*Keywords:* Freeze-drying; Freezing; Concentrated systems; Ice crystals morphology; Microstructure

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## 1. Introduction

In the food industry, it is common that food products are dehydrated for quality preservation, product development and provide convenience for consumer. Freeze-drying is a preferred dehydration process for its ability to retain bioactive compounds and yield highly porous dried matrices which is important for reconstituted products[1], [2]. This relatively expensive process involves freezing of a solution and sublimation of the ice under low temperature and pressure.

The ice crystal formation has pronounced effect on the quality characteristics of the final dried product. Morphology of the ice crystals formed have influential role on the interconnectivity and stability of the porous network of the final dried product as well as efficiency of the subsequent drying steps [3]–[5]. Many studies were able to correlate freezing parameters with physical properties and quality of freeze-dried foods [6]–[10]. For instance, liquid nitrogen and rapid freezing gave better microstructure preservation and instantaneous rehydration to the freeze-dried solids as a result of narrow voids formation [5], [7]. On the contrary, it has been reported that solubility of freeze-dried fruit powders decreased with increasing freezing rate due to high mass transfer resistance in small pores [8]. Also it was demonstrated that development of large crystals from slow freezing yield freeze-dried starch based food with high rehydration ratio [11]. Despite the contradictory conclusions, these findings suggest that freezing is the critical step to control attributes of the freeze-dried system.

The impact of ice crystal development on drying performance has also been investigated. Most of the works focus on reducing product resistance to shorten the drying period hence processing cost. It was reported that optimization of drying cycle can be achieved through temperature-controlled nucleation which produced ice crystals of desired sizes and shapes [12]–[14]. Among the techniques studied to control ice nucleation are use of nucleating agents such as Pseudomonas Syringae and silver iodide (AgI) [12] rapid freezing with nitrogen gas [15], annealing [4] and freezing under with ice-fog technique [13]. Previous works showed the pronounced effect of nucleation temperature on ice crystal morphology hence porous network of the freeze-dried products which determine the rate of ice being sublimated. In example, materials nucleated at high temperature using ultra-sonic vibration exhibited growth of large and directional crystal dendrites [16]. This crystal morphology effectively reduces product resistance at the sublimation interface and shortens the primary drying duration.

As the recent trend in food processing is heading towards low energy impact operation, a number of studies have been carried out to optimize and minimize the energy consumption. Operational models taking into account processing conditions and materials properties have emerged as a simple and convenient tool to facilitate process design of energy efficient freeze-drying process [17], [18]. The contributions of [19]–[22] showed that combined drying treatments can increase the energy saving percentage during freeze-drying. Energy utilized in freeze-drying of pineapple slices reduced as much as 60% when combined with osmosis and microwave drying [22]. Accordingly, the pre-treatments before freezing lower the amount of water to be involved in freeze-drying and enhance mass transfer rate which reduces the processing time.

Processing high solids system is considered in the present work to lower the energy expenditure due to the small fraction of water to be removed. However, the low water availability makes it increasingly difficult for ice crystal development since water molecules mobility is restricted in concentrated solution. Nevertheless, quality characteristics of the freeze-dried materials are greatly affected by ice crystal formation in the freezing step. Therefore, understanding and controlling ice crystal development is the key parameter to ensure quality of the final dried product and process efficiency.

In this research, the effect of temperature fluctuations during freezing and application of aeration on the final microstructure and dissolution of freeze-dried concentrated coffee solutions (50 and 60% by weight) were studied. Cooling profiles were used to determine the effect of product formulations on nucleation temperature and freezing time. Freeze-dried samples were observed using scanning electron microscopy (SEM) technique and images were used to characterize the microstructural characteristics. Dissolution behavior of the freeze-dried coffee was recorded to correlate microstructure with its rehydration capacity. This work provides input to food manufacturers on manipulating product formulations and freeze-drying conditions towards achieving process design that ensures energy efficiency as well as desired end-product properties.