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Influence of Thickness on Ice Crystal Formation in Strawberry during Freeze-drying

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Abstract: This study demonstrates the capability of X-ray Micro-computed Tomography (XMT) technique to characterize the internal ice crystal microstructure of fresh strawberry after freezing. The method requires freeze-drying of the sample to remove frozen water before scanning to indicate ice crystal and internal structure of the material. Results are presented for the 2-D ice crystals formed within strawberry frozen at different rates. The dendrite spacing of ice crystals has been related to the freezing conditions of the material. Moreover ice crystal parameters such as size, volume and width can be measured by using the technique. The overall results indicate that the ice crystal distribution within strawberries diverse with the axial distance of the material from its cooling surface and thickness of the samples. In the latter stage the ice crystal size is bigger when the material is far from the cooling surface while the smallest ice crystal size will be formed from the thickness of the materials.

Key words: Strawberry, ice crystal, x-ray micro computed tomography, freezing, freeze-dried

INTRODUCTION

Improving methods of food preservation is an important technique needed by all of the food industry. Achieving the right preservation technique is very important in result of the changes of nutritional and sensory qualities of food. Freezing is a conventional food preservation technique in which the temperature of food is reduced below its freezing point and a proportion of the water undergoes a change in state to form ice crystals. A quick versus slow freezing mechanism that caused towards ice crystal formation is shown in Fig. 1. The immobilization of water to ice and resulting concentration of dissolved solutes in unfrozen water lower the water activity of the food (Fellows, 2000). This method often results in substantial textural damage caused by the growth of ice crystals within the delicates structure either present naturally or created during processing (Mousavi *et al*., 2005). Recent studies visualized the ice crystal structures formed during freezing of a number of foods has been applied using X-ray micro-CT. An understanding of the relationship between the freezing conditions and the size of ice crystal formed is critical in controlling product quality and texture (Miri *et al*., 2007). The observation of the ice crystal size can be direct or indirect. Direct observation of ice crystal can be done by cryo-scanning electron microscope (Russell *et al*., 1999), cold microscopy (Donhowe *et al*., 1991) and confocal laser scanning microscopy (Evans *et al*., 1996). Indirect method such as freeze substitution (Bevilacqua *et al*., 1979; Martino and Zaritzky, 1998), freeze fixation (Miyawaki *et al*., 1992) and freeze drying techniques (Fayadi *et al*., 2001; Woinet *et al*., 1998a, b) which followed by the sectioning had been used. Freeze drying is well established process for the indirect method to observe ice crystal formation.

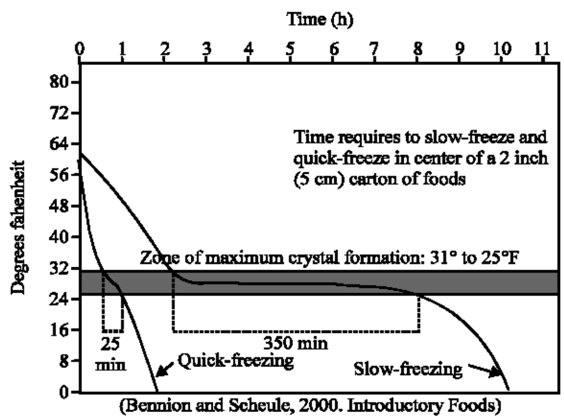

Fig. 1: Quick versus slow freezing mechanisms

However, the indirect methods assume the original morphology is maintained during the sectioning into thin enough layers to allow microscopic and quantitative

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information about ice crystal formation of these strawberries in thick samples (Mousavi *et al*., 2005). X-ray micro-CT is relatively a new technique method to be used. Previously a number of research efforts have been reported that take into account the ice crystal formation in frozen food (such as fish, meat and mycoprotein) may effect their textural, microstructural and qualitative changes. The quality of freeze dried strawberry pieces had been studied and an experiment on a thick layer of strawberry pieces in different operating condition of freeze drying had been done. The working pressure and heating plate temperature during freeze drying were the most important factors affecting the criteria of final product quality in terms of its appearance, shape, colour, texture and dehydration ratio. The optimal conditions for freeze drying process for strawberry is at 30Pa, 50°C and the time ranged for freeze drying is from 60 to 65 h (Hammami and Rene, 1997). Although, the freeze drying time, appearance and colour of the freeze dried strawberries had been investigated, there is no qualitative which has found potential applications in food science research and quality evaluation (Mendoza *et al*., 2006). An axial and lateral resolution down to a few micrometers and without sample preparation and chemical fixation of the architecture of cellular materials can be visualize and analyze through this microCT technique (Van Dalven *et al*., 2003).

Using X-ray micro-CT, the study had been done on various field such as an internal microstructure study for ice crystal visualization of mycoprotein, carrot, meat, fish, chicken, potato and cheese (Mousavi *et al*., 2005; Miri *et al*., 2007), detected internal quality changes in peaches, investigated core breakdown disorder in Conference pears based on their mass density variations during storage and quantitatively analyze and characterize apple tissue to micrometer resolution (Mendoza *et al*., 2006). X-rays are short wave radiations, which can penetrate through fruit. The level of transmission of these rays depends mainly on the mass density and mass adsorption coefficient of the material. The density of many fruits increases with maturity (Mendoza *et al*., 2006). The motivation of those studies was found in the necessity to extract realistic and statistical 2-D internal data for the ice crystal formation in frozen strawberry at micron resolution. Observing the ice crystal formation of the internal microstructure of food is important as it will help in preserved the texture quality of the frozen and freeze-dried strawberry.

MATERIALS AND METHODS

Experimental system: Strawberry fruits were collected from an orchard in Cameron Highlands, Pahang, Malaysia in October 2008. Fresh strawberries were transported to the Analytical Lab of Chemical Engineering Department of University Malaysia Pahang on the same day of recollection and were immediately cut into various thicknesses and frozen -20°C before undergone drying treatment. The average moisture content of the fresh and dried strawberries was 89.6 and 10.8%, respectively. Three batches of fruits were sampled, dried and analyzed. Drying process of strawberries was conducted according to the method modified from previous work (Hammami and Rene, 1997). For freeze drying of strawberries, 250 g sample was initially sorted at about 5, 10 and 15 mm thickness as shown in Fig. 2 and placed on a stainless steel tray. The trays were placed in a freeze dryer (BioTron model Cleanvac 12S) and the blast-freezing process was started at -40°C for 120 min. Under 0.1 Torr constant pressure, the drying process of strawberries required 72 h to reach a moisture content of 10.8%. Dried strawberries was then stored at 4°C until analyzed within a week.

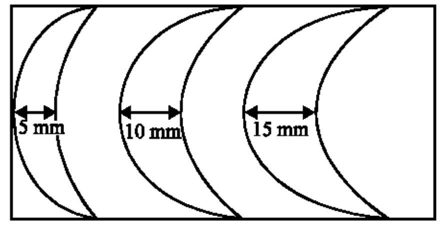

Fig. 2: Schematic drawing of strawberry slicing in different thickness

X-ray micro-computed tomography: The white painted metal sheet of Skyscan 1072 X-ray microtomograph is being used in the present study. Figure 3 is illustrated the schematic diagram of X-ray micro-computed tomography technique. It is includes a chamber where the sample is being placed throughout the analysis. The lead shield outside the chamber is kept closed in order to prevent any X-rays to escape from it. It is using the electricity powered and appropriate software attached from a computer drive. It has to be connected to the computer during operation because an exchanges data between both is needed during the analysis period. It is only respond to the command when it is being connected to each other. A flashing red light at the back of this apparatus will be lit automatically when it detects that X-rays are generated from this equipment. The sample is placed on the sample holder and it is being fixed securely into the right position before the operation can take place. The X-rays is being produced by the apparatus and directly spot onto the sample. The sample is perpendicularly rotated about its long axis and the images are directly shown as grey scale image on the computer. The photographs of the sample at

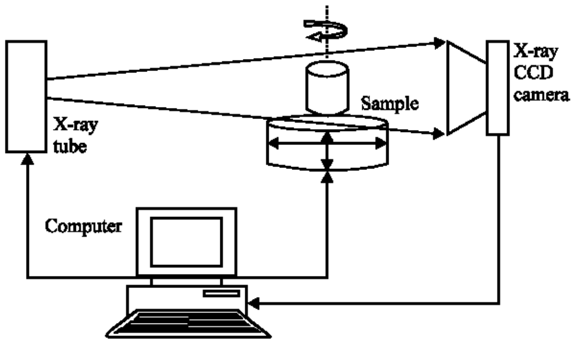

Fig. 3: Schematic diagram of X-ray micro-computed tomography technique

different positions are taken and being saved to the computer drive. The images then being reconstructed using software (Nrecon) and a plane image obtained is further used in the analysis of the sample that performed by the analysis software (CT analysis).

RESULTS AND DISCUSSION

Ice crystal quantification: Cooling rates lead to the development of a dendrite ice crystal structure and the changes is from the bottom to the top of the samples can be clearly seen by the X-ray (Mousavi *et al*., 2005). Figure 4a-c also compared clearly that the shape of ice crystals changed from sample base to its top and thus creating the different width distribution of ice crystals in the samples. The width distributions range between 0.06 to 0.39 mm. As seen, ice crystal width increased from the bottom to the top of the samples. These happen due to the top of the sample is far from the cooling surface and leading to larger ice crystals, while the bottom sample is near to the cooling surface and thus creating much smaller ice crystals throughout the samples. The rate of cooling decreasing with increasing distance from cooling surface and these leading to larger ice crystals which is possible to influence the microstructure by changing the freezing conditions (Mousavi *et al*., 2005). Freezing of different thickness of strawberry samples produced similar shapes of ice crystal as shown in Fig. 5. Similar shapes of ice crystal for each sample but the size is different neither in each axial position nor the thickness itself. Figure 6 has been noted the trend that thicker sample gave smaller ice crystal distribution measurement compared to the less thick of the samples. Figure 6 shows the width distribution of the ice crystal measurement for the different thickness of the samples that using the same freezing technique. The relationship between microstructure and ice crystals is clearer when these ice crystals structure formed in

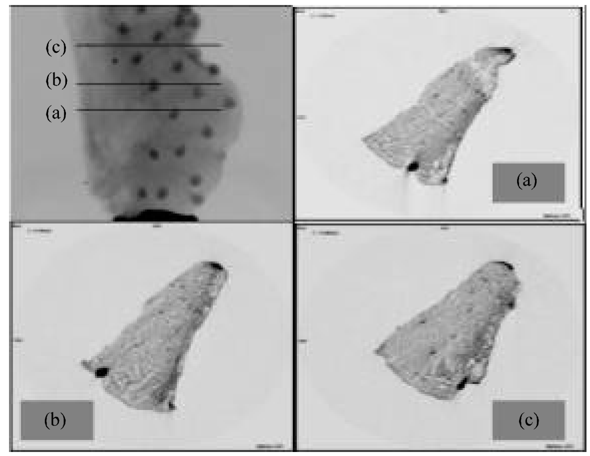

Fig. 4: Tomographic image of freeze-dried strawberry: (I) Typical side view of X-ray image; (a) 7.435 mm from cooling surface (b) 9.700 mm from cooling surface and © 12.945 mm from cooling surface

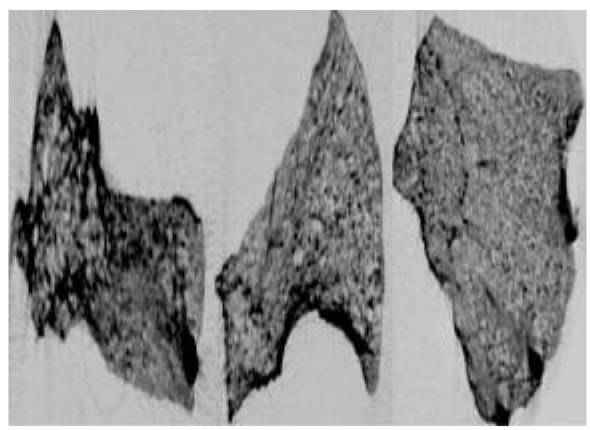

Fig. 5: Microstructure of different thickness of freeze-dried blast freezing strawberry

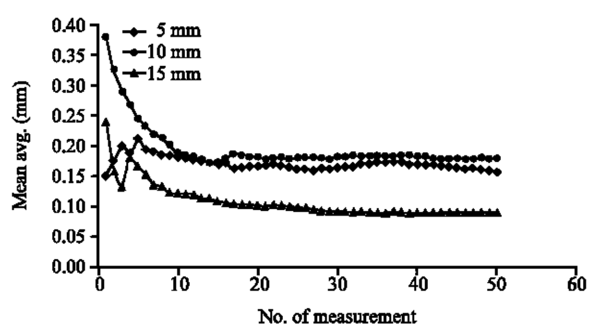

Fig. 6: Average width distribution of the ice crystal measurement for the different thickness of the freeze-dried strawberry

different axial position and thickness of the strawberry. As figured, ice crystal in the central zone of the strawberry are larger than upper and bottom of the samples. The stronger cell wall in the intermediate of the sample might cause these larger ice crystals. It was clear here that the size or thickness and direction or position of the samples mostly affect the size of ice crystal. However, the shape of the ice crystal and structure under this freezing condition is not much different because the strawberry samples have similar microstructure at either direction.

CONCLUSIONS

Ice crystals in frozen strawberries are qualitatively and quantitatively investigated using X-ray micro-CT. The X-ray technique could visualise and differentiate the effect of ice crystal growth in frozen and fresh strawberry and the freezing rate condition of the strawberries regarding the distances of the frozen strawberries from the cooling surface and thickness of the strawberries. Ice crystals in thin samples for frozen strawberries have the smallest ice crystal size and the samples that isolated from the cooling surface have the slowest freezing rate thus creating the biggest ice crystals in frozen strawberry. Larger ice crystals and broader crystals size distributions were seen at the top of the sample than the centre, while at the bottom of the strawberry samples, the ice crystals formed are much smaller. The result suggests that the freezing rate affect on microstructures of these frozen strawberries, the interrelationship between ice crystal and strawberry microstructure in each case and freeze dried is a good technique to visualise the microstructure of food samples.

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