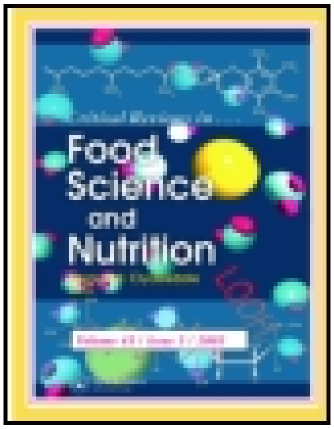

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Emerging Techniques for Assisting and Accelerating Food Freezing Processes—A Review of Recent Research Progresses

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Emerging techniques for assisting and accelerating food freezing processes — a review of recent research progresses

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Abstract

Freezing plays an important role in food preservation and the emergence of rapid freezing technologies can be highly beneficial to the food industry. This paper reviews some novel food freezing technologies, including high pressure freezing (HPF), ultrasound-assisted freezing (UAF), electrically disturbed freezing (EF) and magnetically disturbed freezing (MF), microwave assisted freezing (MWF), and osmo-dehydro-freezing (ODF). HPF and UAF can initiate ice nucleation rapidly, leading to uniform distribution of ice crystals and the control of their size and shape. Specifically, the former is focused on increasing the degree of supercooling, whereas the latter aims to decrease it. Direct current electric freezing (DC-EF) and alternating current electric freezing (AC-EF), exhibit different effects on ice nucleation. DC-EF can promote ice nucleation and AC-EF has the opposite effect. Furthermore, ODF has been successfully used for freezing various vegetables and fruit. MWF cannot control the nucleation temperature, but

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can decrease supercooling degree, thus decreasing the size of ice crystals. The heat and mass transfer processes during ODF have been investigated experimentally and modeled mathematically. More studies should be carried out to understand the effects of these technologies on food freezing process.

Keywords: High pressure freezing, ultrasound-assisted freezing, electrically and magnetically disturbed freezing, osmo-dehydro-freezing

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1. Introduction

With the increasing demand for fresh foods, freezing is always an effective method to preserve food quality and extend its shelf life. Many researches demonstrate that freezing rate is a key factor to maintain the quality of frozen foods. Generally speaking, rapid cooling rate can not only save time to some extent, more importantly, it can also lead to better food quality than slow cooling rate. There are some disadvantages of slow freezing and low degree of supercooling, as in such cases, the speed of ice nucleation is slower than the speed of ice growing, thus resulting in the formation of big ice crystals in the final frozen foods and their exclusive distribution in the extracellular region. On the contrary, fast freezing can generate small and uniform ice crystals in foods, with their distribution being mainly in the intracellular region. As a result, the microstructure of foods can be protected, and the quality of foods can be preserved. Therefore, new freezing methods have been developed to improve the food quality.

Currently, in the food industry, commercial frozen foods are obtained by the conventional freezing methods, such as air blast freezing, cryogenic freezing, direct-contact freezing, and immersion freezing (Delgado *et al*., 2009; Orlowska *et al*., 2009). However, due to the low thermal conductivity of foods ranging from 0.5 to 1.5 $W/m^2 \cdot K$, the freezing rate of these methods is considerably low (Sun & Li, 2003; Singh & Heldman, 2013). The quality of the frozen food is strongly related to freezing rate. Slow freezing rate will normally produce larger ice crystals with irregular and uneven distribution of ice crystals, resulting in serious deformation of muscle tissue.

With the main objective of enhancing the freezing rate and improving frozen food quality, novel freezing methods are being investigated and developed, including high pressure freezing, ultrasound assisted freezing, electro-magnetic disturbance freezing, and dehydration freezing. Although Li and Sun (2002a) had published a review on rapid freezing methods over a decade ago, as in the meantime freezing field is an extremely quick development domain, some of the newest methods have emerged. Therefore, this review aims to summarizing recent research advances in developing novel freezing methods. The mechanisms and influencing factors of the methods of ice crystal formation and heat transfer are discussed, and their potential applications and future trends are also proposed.

2. High pressure freezing (HPF)

Although high pressure has been used as a promising non-thermal technology for food processing for almost 20 years, its application for food freezing is relatively new. Due to its complexity and superiority, a series of studies about HPF have been conducted (Chevalier-Lucia *et al*., 2003; Fuchigami *et al*., 2006; Malinowska-Pañczyk *et al*., 2014; Vaudagna *et al*., 2012). As at high pressures the freezing point of liquid water can be reduced to below 0 ^{o}C to a large extent a high supercooling degree can be obtained to facilitate the formation of small ice crystals once pressure is released. Meanwhile, the formation of ice crystals is instantaneous and homogeneous throughout the whole volume of the sample (Fernández *et al*., 2007; Realini *et al*., 2011). As shown in Fig. 1, the water goes through different phases to form various types of ice (I-VI) at different pressures and temperatures. The ice density varies with the type of ice. It is well-known that the volume of water would increase during freezing at atmospheric pressure due to the formation of ice I. Ice I is the only type of ice, the density of which (0.92 g/cm^3) is lower than that of water. During freezing at pressures ranging from 0 to 209.9 MPa, water is usually

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changed to ice I. Ice II to ice VI can be formed if the pressure is increased continuously. As a result, they do not expand in volume during freezing and the problem of mechanical damage to food tissues can be alleviated. It is noteworthy that ice IV does not exit and it would be turned to ice II and ice III immediately. Moreover, ice VI with the density of 1.31 g/cm^3, may be formed at room temperature if the pressure reaches 800 MPa or higher. In this case, food materials can be frozen without any forms of cooling.

There are three different strategies for freezing under high pressure, including pressure assisted freezing (PAF, ABCD in Fig. 1), pressure shift freezing (PSF, ABEFG in Fig. 1) and pressure induced freezing (PIF, ABEFHI in Fig. 1). For PAF, the freezing process is performed under a constant pressure. In the case of PSF, ices are formed due to the sudden release of pressure. Furthermore, for PIF, the phase change starts with the increase of pressure and the freezing process continues at a constant pressure (Knorr, 1998). The strategies of PAF and PSF are usually applied for high pressure freezing, whereas PIF has been poorly studied. Generally, both PAF and PSF perform better than PIF for food freezing and it is easier to operate PAF and PSF. PIF demands much higher pressure (Fig. 1), especially if temperature probe is installed to measure the change of temperature, it is technically difficult for the equipment to achieve such a high pressure. Compared to PAF, PSF is regarded as a better choice during the high pressure freezing process, mainly due to high degree of supercooling reached after expansion and uniform distribution of ice crystals throughout the sample (Fernández *et al.*, 2006). As shown in Fig. 1, although high supercooling level can be achieved during PAF process, for example, the supercooling degrees of potato at 140 MPa and pork at 700 MPa can reach 7 ^{o}C and 10 ^{o}C, respectively (Knorr *et al.*, 1998; Molina-García *et al.*, 2004), with PSF, higher supercooling degree can be obtained, for example, the supercooling degree of shrimp at 200 MPa can reach 20 ^{o}C (Su *et al*., 2014). However, it is worth noting that the phase change time is the main difference between PSF and PAF, and the comparison between them can only be made under the condition that the amount of latent heat released and the temperature difference (ΔT) between the initial freezing point of the sample and the cooling medium are identical (Fernández *et al*., 2006). The authors (Fernández et al., 2006) indicated that the phase change time in PSF was significantly lower than that in PAF at the same pressure in gelatin samples. Generally, the shorter the phase change time is, the more and finer ice crystals can be formed.

The advantage of HPF depends on the quantity of ice crystals formed after expansion (Otero *et al*., 2009), and this advantage reflects more significantly in the PSF process. Crystallization throughout the whole sample can take place instantly if the pressure can be released extremely rapidly (Fig. 2). Due to the high rate of the process, a large number of ice nuclei are formed. The initial number of ice nuclei determines the size, shape, and distribution of final ice crystals within the sample. The ice crystals are closely related to the texture damage in frozen samples (Zhu *et al*., 2003). Otero and Sanz (2000) proved that a large number of ice crystals were instantaneously formed in agar gel (agar "Bios C" from Biolife) containing 99% water samples in the PSF process. Fernández *et al*. (2006) found that a large number of tiny ice crystals were formed immediately after expansion and they were distributed homogeneously throughout the gelatin samples during the PSF process, and the microstructure of the samples was maintained.

The thermal properties of the ice-water hybrid of frozen products after rapid depressurization, which can affect the final freezing behavior, are also influenced by the ratio of ice to water (Chizhov & Nagornov, 1991). Furthermore, the simulation of the freezing process,

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as well as the duration of the whole freezing process, is also influenced profoundly by the original amount of ice nuclei (Otero & Sanz, 2003). The pressure and temperature conditions before expansion, the supercooling degree, the amount of ice nuclei are the main factors affecting ice crystals. Higher amounts of instantaneous frozen water would be produced when increasing pressure at a given temperature before expansion (Otero & Sanz, 2006). Homogenous distribution of tiny ice crystals would be obtained under a large supercooling degree and a large initial amount of ice nuclei as noted by Otero & Sanz (2006). Furthermore, after expansion, the pressure medium plays a remarkable role in the heat removal from the sample, and the original ratio of pressure medium/sample is also an important factor. Both of them influence the growth stage of ice crystals.

In general, two methods have been utilized to determine the amount of ice crystals, i.e., mathematical modelling and measurement of heat balance. The former developed by Sanz *et al*. (1997), considered the relationship between some parameters, including pressure, temperature, and specific volume in the liquid water and ice I regions, and in the boundary between the regions, and then predicted the ice percentage. In their study (Sanz *et al*., 1997), 36% of ice in PAF, 1% agar gel samples was predicted at 200 MPa with the supercooling degree of 20 ^{o}C reached. Otero and Sanz (2000) also predicted an instantaneous formation of up to 29.1% ice in agar gel samples (99% water) using the same heat balance model. The latter, with a calorimetry, has recently been effectively used to investigate thermodynamic properties of foods and phase-change phenomena. Chevalier *et al*. (2001) first used an isothermal calorimeter to measure the latent heat of ice-water compound after the remove of pressure instantaneously during quasi-adiabatic PSF process, and then calculated ice ratio that ranged from 0.117 to 0.402 (kg ice/kg ice-water mixture) at 115 MPa/-10 °C and 210 MPa/-21 °C, respectively. In addition, a high-pressure (HP) calorimetry has been developed to analyze the latent heat and the percentage of ice crystals of food materials under HP conditions by Zhu *et al*. (2005), who used the HP calorimeter to evaluate the percentage of ice crystals formed by sudden pressure release during PSF, and then the ratio was determined by the calorimetric peak measured and heat balance. The calorimetric signal appeared to be stable with a baseline balance very close to zero, and the quantity of heat transferred between sample and reference cells was estimated by integrating the calorimetric peak subtracted from the baseline. Finally, equations about pure water (Eq.(1)) and pork meat (Eq.(2)) ice crystals ratio were derived, respectively, which indicated that the ratio of ice crystals is only related to the pressure-related parameters of pressure medium, rather than to various thermal properties of food samples. It was found that about 28% and 20% of ice crystals formed at 200 MPa/-20 °C in pure water and pork tissue, respectively. Moreover, corresponding regression relationships between the observed ice crystal ratio and pressure ($R^2 \geq 0.95$) were obtained.

$$R_{ice-water} = 100\left(1 - \frac{Q - Q_P}{mL} + \frac{C_{Pice}(T_f - T)}{L}\right) \qquad (1)$$

$$R_{ice-water} = 100\left(1 - Z_T - \frac{Q - Q_P}{mL} + \frac{\overline{C}_{Pfs}(T_f - T)}{L}\right) \qquad (2)$$

where $R_{ice\text{-}water}$ is the ice crystal ratio; Q is the heat differential value between water sample and reference cells; Q_P is the differential value of adiabatic compression/decompression heat owing to the volume differential value of pressure medium between sample and reference cells; C_{Pice} is the specific heat of pure ice; T_f is the freezing temperature of water (°C); T is the calorimeter temperature of the calibration tests performed (°C); Z_T is the fraction of unfrozen water in frozen sample; m is the sample mass (g); L is the latent heat between water and ice at atmospheric pressure; $\bar{C}_{Pfs}$ is the mean specific heat of frozen pork sample at the relevant temperature.

In addition, the ice in all the above researches obtained was near the melting curve between the liquid and ice I. The strategy of increasing pressure considerably beyond the curve, where different types of ice can be formed, was not taken into consideration in the above studies. Thus, the range of temperature and pressure should be expanded properly in future studies, so as to extend the application of the model and verify its accuracy. Otero *et al*. (2009) also validated the aforementioned assumption that different types of ice could be obtained near or beyond the curve between the liquid and ice I under various conditions, which were both close to and far from the melting curve of liquid-ice I and it was found that 28.8% of ice crystals were formed at 200 MPa / -20 °C, and 25% of ice crystals were formed at 450 MPa / -10 °C. Furthermore, Schlüter and Knorr (2002) found an unexpected metastable liquid region above the curve separating ice I and ice V at 240 MPa / -28 °C during potato freezing, and recommended that the triple point of liquid / ice I / ice V was the most appropriate point for PSF freezing, thus increasing the supercooling degree by 30%. More studies are expected to explore the metastable zone during freezing of various products. Therefore future research about this metastable zone and other potential metastable zones should be considered in the experimental design to confirm these possible assumptions, including different types of ice that are formed in different regions, the increase of supercooling degree and the achieving of the best freezing point for HPF, in different products.

Although calorimetric techniques have been used to measure the latent heat, there are some disadvantages of these specific heat measuring methods. For modelling, it is well-known that both latent heat and sensible heat are involved in specific heat, which has a peak value around the freezing point. As a result, it is difficult to obtain convergence during modelling calculation and the value of latent heat obtained may be underestimated (Norton *et al*., 2009). On the other hand, Pham (2006) developed a simple and efficient approach to avoid the heavy computation, which was based on the enthalpy formulation, and was termed as the quasi-enthalpy method, as shown in Eq. (3), as enthalpy is always conserved. Norton *et al*. (2009) applied this method to simulate the HPF process. In their study (Norton *et al*., 2009), the total freezing time and the amount of ice formed upon the instantaneous removal of pressure were calculated. The predicted results were also validated experimentally.

$$H_i^{New} = H_i + c_i(T_i^{New} - T_i) \qquad (3)$$

$$C\frac{T^{New} - T}{\Delta t} = -K\overline{T} + \overline{f} \qquad (4)$$

where H_i^{New} is the new nodal specific enthalpy; H_i is the specific enthalpy; T is a vector of nodal temperatures; T_i^{New} is obtained from Eq. (4); C is the global capacitance matrix containing the specific heat; c is the specific heat ($J \cdot kg^{-1} \cdot K^{-1}$); the subscript "$i$" is the node number; the superscript "New" refers to temperatures at the end of the time step; Δt is the time step (s); K is the global conductance matrix; $\overline{T}$ is the time-averaged value of the nodal temperatures; $\overline{f}$ is the average global forcing matrix.

Recently, HPF has been utilized to freeze various food products. There is no doubt that HPF can benefit foods through preserving their original properties and improving their quality. Volkert *et al*. (2012) used HPF to treat sugar rich dairy product, and the resulting samples had superior properties in the aspects of smoothness and mouth coating. Otero *et al*. (2012) investigated the ice crystal shape and distribution in fluid foods treated by PSF and found that ice crystals were formed quasi-instantaneously and distributed homogeneously throughout the sample, with the ice crystals being of round shape without indentations. Thus, the food quality after PSF was better than that after conventional crystallization. Fuchigami and Teramoto (2003) examined the texture and structure of high-pressure-frozen gellan gum gel and found that HPF at 200-500 MPa was effective in improving the surface and structure of gels.

Moreover, HPF has been also applied in other fields. For example, Kurth *et al*. (2012) used HPF to preserve the ultra-structure of coccidian oocysts. The result demonstrated that HPF with freeze-substitution and epon-embedding pretreatment was a suitable method for analyzing the ultra-structure of coccidian oocysts. Meanwhile, Weiner *et al*. (2013) investigated the vitrification of thick samples by HPF using soft X-ray cryo-tomography and showed that the application of HPF could avoid crystallization artifacts and improve imaging conditions in samples. Furthermore, Ellinger *et al*. (2010) developed a novel approach for understanding the endocytic compartments. Specifically, the combination of HPF with peroxidase-catalyzed cytochemistry could provide excellent structural preservation. Consequently, the complex 3D structures of endocytic compartments could be discerned clearly and the subtle details could be understood better.

3. Ultrasound assisted freezing (UAF)

Ultrasound is sound waves oscillating at a frequency beyond the threshold of human hearing. Ultrasound has been widely used in different fields, including medicine, chemistry and food science and technology. In the food industry, ultrasound waves have been widely used to inactive microorganisms and enzymes in foods and enhance the quality of final products.

Generally, ultrasound can be divided into two categories according to the applied frequency and intensity, namely high-frequency and low-intensity ultrasound (usually > 100 kHz), and low-frequency and high-intensity ultrasound (usually 20-100 kHz) (Awad *et al*., 2012). The former is mainly used for non-destructive analysis and food process control, due to its significant effect on food characters and food packages, etc. (Mason *et al*., 1996; Ross *et al*., 2004; Xu *et al*., 2014b). On the other hand, the low-frequency and high-intensity ultrasound, which is also termed as power ultrasound, can be used in different areas of food processing, such as emulsification/homogenization, sterilization/pasteurization, extraction, drying, freezing, etc. (Li *et al*., 2004; Hengl *et al*., 2014). It is noteworthy that in the case of UAF, ultrasound with both low frequency and low intensity is recommended. For example, the study of Kiani *et al*. (2013b) found that during the freezing process under ultrasound irradiation (25 kHz, 0.25 $W \cdot cm^{-2}$), the viability of *Lactobacillus Plantarum* cell could be increased. Although the application of UAF for food freezing is a relatively new topic, recent studies have proved that this technology is promising to enhance the freezing process and preserve food quality (Delgado & Sun, 2011; Delgado, *et al*., 2009; Li & Sun, 2002b; Islam *et al*., 2014; Sun & Li, 2003; Xu *et al*., 2014a; Zheng & Sun, 2006). This enhancement is achieved by initiating ice nucleation, increasing heat and mass transfer rates, and controlling the size and shape of ice crystals. Several physical phenomena generated by ultrasound in the liquid medium are responsible for the ultrasonic enhancement, which include cavitation effect, micro-streaming, fracture of big ice crystals, etc.

The cavitation effect (Fig. 3) includes the formation of microbubbles and the violent collapse of these bubbles, and is the main contributor in accelerating the freezing of foods. The transmitting of the sound waves through a dissipative fluid medium produces the cavitation. The

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negative pressure during the rarefaction in sound wave transmission can make the liquid to facture, leading to the formation of bubbles or cavities (Ashokkuman & Grieser, 1999). The collapse of cavitation bubbles can create a positive pressure (5 GPa or more) in nano-seconds (Hickling, 1965), resulting in high supercooling degrees can serve as a driving force for nucleating instantaneously (Inada *et al.*, 2001). Furthermore, these cavitation bubbles can act as nuclei for ice nucleation, as long as reaching the critical nucleus size (Mason *et al.*, 1996). Therefore UAF can promote primary nucleation, which involves crystallization process in a solution starting without pre-existing crystals, as confirmed by various experiments. For example, Chow *et al*. (2003; 2004; 2005) found that ultrasound irradiation had the ability of triggering nucleation in supercooled aqueous solutions; Ruecroft *et al*. (2005) and Saclier *et al*. (2010) reported that power ultrasound could initiate nucleation in industrial crystallization processes of organic molecules; and Xu *et al*. (2014a) discovered that ultrasound irradiation was able to induce nucleation in radish cylinders during UAF process, and a good fit to linear equation was exhibited between the nucleation temperature and the ultrasound irradiation temperature (7 s duration, 0.26 $W{\cdot}cm^{-2}$ intensity). In addition, nucleation is expected to occur immediately once the collapses of cavitation bubbles. Nevertheless, some researches have revealed that ice nucleates lingeringly, which leads to a delay between the cavitation and the occurrence of nucleation, rather than occurring immediately (Hu *et al.*, 2013a; Chow *et al.*, 2005; Zhang *et al.*, 2003). Furthermore, the pressure gradient generated by cavitation bubbles is also regarded as the driving force for ice nucleation (Dodds *et al.*, 2007; Grossier *et al.*, 2007).

Meanwhile, micro-streaming can be also regarded as another potential driving mechanism for the ice nucleation during UAF process. Micro-streaming is generated due to the movement of stable cavitation bubbles, which do not collapse suddenly after the formation, as shown in Fig. 4. Zhang *et al*. (2003) detected two separate zones with different nucleation rates during UAF, and the nucleation rate of the second zone was higher than the first zone (although the nucleation rate of the first zone was very high), which was contrary to Hickling's theory (Hickling, 1965), in which the ultrasound induced ice nucleation is only caused by the bubble collapses, namely, just one zone of nucleation is occurring during UAF process. They thus suggested that flow streams were probably the secondary cause of nucleation. Chow *et al*. (2005) also confirmed that suggestion on the basis of microscopic observations, and illustrated that stable cavitation bubbles, which do not collapse immediately, can cause flow stream to induce nucleation.

The above mechanisms of cavitation effect and micro-streaming are the two major principles to accelerate ice nucleation. Consequently, the number of produced nuclei is increased, leading to the control of the size and shape of ice crystals, which would be small and uniformly distributed. Moreover, ice crystals with big size that grow randomly can be fractured due to the variation of acoustic pressure, which further ensures the prevailing of tiny and uniform of ice crystals. Besides, micro-streaming was shown to be involved in the enhancement of the heat and mass transfer as well as local cavitation clouds (Kiani *et al*., 2011) and bubble collapses close to the solid–liquid interfaces, which is owing to providing violent agitation in the liquid phase (Li & Sun, 2002b; Zheng & Sun, 2005) and disrupt boundary layers respectively (Legay *et al*., 2011). Therefore the resistance of heat and mass transfer would be reduced at the ice/liquid interface, and thus freezing rate would be increased (Li & Sun, 2002b; Sun & Zheng, 2006). More importantly cavitation clouds at the surface of the sphere sample is the main effect on enhancing heat transfer (Kiani *et al*., 2012), and in general, high rate of heat and mass transfer can enhance ice crystal growth.

Many factors affect the effectiveness of UAF, such as the intensity and frequency of ultrasound, the position of the samples, cooling medium temperature and flow rate, etc. Kiani *et al*. (2012) investigated the heat transfer phenomena using a stationary copper sphere (k = 386 $W{\cdot}m^{-1}{\cdot}K^{-1}$, c = 384 $J{\cdot}kg^{-1}{\cdot}K^{-1}$, ρ = 8660 $kg{\cdot}m^{-3}$) at different positions with different intensities, and showed that the intensity of ultrasound and sphere position were directly related to cavitation bubble population and acoustic streaming patterns, and observed that strong cavitation clouds and acoustic streaming existed at the surface of the sphere, resulted in a high cooling rate. The properties of cooling medium could also significantly affect heat transfer and ice crystal formation, as Kiani *et al*. (2013a) showed higher viscosities and lower flow rates in the cooling medium due to its mixing and cavitation effects at appropriate ultrasound irradiation (890 $W{\cdot}m^{-2}$).

To date, ultrasound has been used to assist the freezing process of both solid and liquid samples. Sun & Li. (2003) examined the microstructure of potato tissues frozen by ultrasound-assisted immersion freezing at 25 kHz and 15.85 W. The results showed that not only the freezing rate was increased but the occurrence of cell destruction was also avoided. As a result, small intercellular ices were generated throughout the potato sample and its micro-structure was preserved well. Hu *et al*. (2013b) studied the impact of UAF on the quality and microstructure of frozen dough. It was found that under sonication at 25 kHz and 288 or 360 W, the freezing time was reduced significantly and a large number of tiny ice crystals were generated inside the frozen dough. Furthermore, the study of Xin *et al*. (2014) proved that the microstructure and the firmness of broccoli tissue frozen by UAF were markedly better than those of broccoli tissue frozen by traditional immersion freezing method. In the meantime, the drip loss of samples frozen by UAF was significantly reduced and the optimal UAF condition was obtained when the ultrasound power was 150 W for 30 kHz ultrasound and 175 W for 20 kHz ultrasound (Xin *et al*., 2014). Yu *et al*. (2012) applied power ultrasound through the whole nucleating stage from 0 ^{o}C in pure water and degassed water during UAF process, and found that supercooling degree was decreased and ice nucleation was initiated. Furthermore, Hu *et al*. (2013a) studied the effect of pre-existing bubbles on ice nucleation and crystallization during UAF of water and sucrose solution at 0.21 $W\cdot cm^{-2}$ and 20 kHz, and indicated that pre-existing bubbles might be transformed into cavitation bubbles, thus enabling the promotion and control of the ice nucleation process.

On the other hand, Kiani *et al*. (2011) investigated the mechanism of ultrasound (25 kHz, 0.25 $W\cdot cm^{-2}$) for freezing both liquid (sucrose solution) and solid (agar gel) samples. In their study, it was found that ultrasound behaved more effectively to initiate the ice nucleation in liquid samples, rather than in solid samples. There was a linear relationship between the temperature during ultrasound irradiation and the nucleation temperature in the liquid samples. Furthermore, ultrasound had a more prominent effect on the heat transfer rate during the freezing process of liquid sample, due to its uniform property.

In general, in both HPF and UAF, samples are normally submerged in cooling liquid media, and therefore they can be regarded as special immersion freezing enhancing techniques. During the freezing process, the cooling media that could not be frozen at ultra-low temperatures are required. The cooling media play an important role on removing the heat generated during freezing, resulting in the formation of small and uniform ice crystals. Currently, alcohol-water, glycol-water and polyethylene-water are the three common media used for HPF and UF. It should be pointed out that these cooling media are not suitable for direct contact with foods, and other cooling media should be used if foods are in direct contact with the media.

4. Electrically and magnetically disturbed freezing

In the last decade, three electromagnetic technologies, including high-voltage electric field, magnetic fields, and radiofrequency, have also emerged for food freezing (Hozumi *et al*., 2003; Orlowska *et al*., 2009; Petersen *et al*., 2006; Xanthakis *et al*., 2013).

4.1 Electrically disturbed freezing (EF)

Electric field can be divided into two categories, namely oscillating electric field (OEF) and static electric field (SEF). The former is mainly used for pure water heating (dielectric heating), while the latter is mainly used for product freezing. The mechanism of OEF is water molecule rotating in response to an applied electric field, as water molecule belongs to a kind of dielectric material due to the characteristics of its intrinsic electric dipole moment. OEF at radio or microwave frequencies has been regarded as a preferred method for rapid thawing of cryo-materials (Wusteman *et al*., 2004). For frozen foods, OEF is able to suppress ice formation and enhance the supercooling level, which is probably ascribed to the re-distribution of water molecules in the electric field. Jackson *et al*. (1997) investigated the effect of OEF on the verification of ethylene glycol solutions, and found that OEF at 2.45 GHz was able to reduce the amount of ice formed. The study of Sun *et al*. (2006) showed that OEF at 50 kHz could reduce the size of ice crystal to the minimum.

On the contrary, freezing under SEF with high voltage has also been used to increase ice nucleation temperature and improve the quality and microstructure of frozen food (Hozumi *et al*., 2003). Generally, water molecules are strongly polar due to the characteristic dipole structure. Because of the dipole moment, in the presence of electric field, the dipole polarization of water can be re-arranged and water molecules move in the direction of the electric field. There are clusters of molecules in liquid water, which are linked by hydrogen bonds and seemed as solid configuration. Due to reorientation phenomenon, hydrogen bonds become stronger. As a result, the structure of water clusters can be changed, as evidenced by molecular dynamic simulations conducted by some researchers (Shevkunov & Vegiri, 2002; Sun *et al*., 2006; Vegiri, 2004a; Vegiri, 2004b; Zangi & Mark, 2004). For instance, Shevkunov and Vegiri (2002) illustrated that the water cluster converged into an almost aligned state at the electric field strength of $1.5*10^7$ V/m, owing to the dipoles in the direction less than 90^o to the direction of the field. Ice nuclei are related to the aggregation of small stable molecules, which would be promoted during SEF process. However, in the early studies, there were two different opinions about the final changes of water structure in SEF process. On one hand, in SEF process water molecules would be well-aligned and became solid-ice mixture structures, but still in the liquid stage (Jung *et al*., 1999). On the other hand, water medium might turn into solid state, such as ice I, when the magnitude of homogeneous electric field reached up to $5.0*10^9$ V/m (Svishchev & Kusalik, 1994). Later, Sun *et al*. (2006) simulated a set of molecular at -15 ^{o}C under strength of $0\text{-}8.0*10^9$ V/m and reported that during SEF process the water system had an ice-like structure. Although the results obtained in the both hands are different in some aspects, both of them had demonstrated that SEF was effective to initiate the ice nucleation and had overturned the result that SEF do not have positive effect on freezing in previous studies (Shichiri & Araki, 1986; Dolittle & vali, 1975; Shichiri & Nagata, 1981). Furthermore, the study of Orlowska *et al*. (2009)

exhibited that the nucleation temperature increased with the increase of applied voltage. The strength of SEF also had a positive relationship with the supercooling degree according to the study of Sun *et al*. (2008). In general, the food structure can be maintained once ice nucleation and supercooling degree can be controlled.

Most studies (Le-Bail *et al*., 2011; Orlowska *et al*., 2009; Petersen *et al*., 2006) about SEF are mainly carried out in model food systems or aqueous solutions instead of real food products, in order to minimize convective heat transfer phenomena inside the product. Recently, Xanthakis *et al*. (2013) investigated the freezing process of pork meat assisted by SEF at 12 kV with the supercooling degree varied from 3.93 ± 1.3 to 1.92 ± 1.45 °C. The results revealed that the size of ice crystals was significantly reduced from 32.79 ± 4.04 μm to 14.55 ± 8.20 μm, when the electric field intensity was increased from 0 to 12 kV. All these above studies of SEF had provided strong evidence to indicate that SEF had a positive effect on the food freezing process, including the initiation of ice nucleation and the alleviation of damage in frozen food structure.

Besides the original state of the samples to be frozen, factors affecting the initiation of ice nucleation by SEF include the characteristics of electrode such as electrode shape and material and electric field parameters. Even if the freezing process is carried out at the same high voltage, the actual magnitude of electric field dissipated may vary in different systems. Hozumi *et al*. (2003) investigated the impact of material of electrode on the SEF-assisted freezing process. Six materials, including aluminum, copper, argentum, aurum, platinum and carbon, were used. It was found that the lowest degree of supercooling was obtained when aluminum was used, whereas the application of carbon resulted in the highest supercooling degree. Hozumi *et al*. (2005) compared the effects of electrode of two different shapes (sharp and flat) on the freezing process

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and revealed that the supercooling degree was lower when the electrode with flat surface was utilized. Besides the application of SEF at high voltage, low voltage is also occasionally applied. Okawa *et al*. (Thermal Engineering Conference, 1999) reported that SEF at considerably low voltages between 30-120 V could enhance the immersion freezing process at both -6 °C and -8 °C. However, the electric enhancement of freezing process was insignificant when SEF at 50 V and -2 ^{o}C was used (Hozumi *et al*., 2003).

It is notable that the aforementioned SEF processes are all about DC electrical field. Moreover, AC electrical field also shows some potential to influence the freezing process, including enhancing the supercooling degree and prohibiting or delaying the spontaneous ice nucleation (Woo & Mujumdar, 2010). For example, AC-EF could be used for inhibiting the formation of ice crystals on ice cream surface, as claimed by the patent developed by Kim *et al*. (2013), showing that AC-EF at radio frequency voltage could shock the dipole water molecules. As can be seen, the effect of freezing assisted by AC-EF is different from that of freezing assisted by DC-EF. According to our best knowledge, there are no studies reporting the application of AC-EF on real food freezing.

4.2 Magnetically disturbed freezing (MF)

Magnetic field has been developed to achieve rapid freezing, specifically for the products having magnetocaloric properties (Aleksandrov *et al*., 2000). Similar to the electric field, magnetic field can also be divided into two categories, namely oscillating magnetic field (OMF) and static magnetic field (SMF).

Water is characterized by the diamagnetic property without intrinsic magnetic dipole moment. Hence, it is susceptible to develop a magnetic dipole moment under a magnetic field.

When it comes to freeze samples in the presence of MF, special attention should be paid to the initiation of a magnetic moment and the subsequent maintenance of this magnetic moment. Since the hydrogen bonds of water molecules would become stronger under the magnetic field, they are usually well-distributed and stable. The melting point and thermal conductivity of water also increased simultaneously (Inaba *et al*., 2004; Norio & Satoru, 2001). The study of Iwasaka and Ueno (1998) found that the enhancement of the strength of hydrogen bonds at 14 T SMF was associated with near-infrared spectrum of water molecules.

Although the mechanism of effect of MF on water molecules is different from that of effect of EF, MF is also able to reduce the supercooling degree, which is the main advantage of MF. Aleksandrov *et al*. (2000) observed that the critical degree of supercooling decreased with the increase of SMF in the range of 0-0.5 T, which was similar to the effect of DC-SEF as mentioned previously. The supercooling could be negligible and the solidification occurred once the MF intensity was higher than 0.5 T. On the other hand, the patent developed by Norio and Satoru (2001) claimed that water could stay in a metastable state in the presence of MF and a high degree of supercooling could be generated, and therefore samples could be frozen rapidly if the temperature decreased to a low degree or the MF was removed instantaneously. Rotating magnetic field is a periodic transverse magnetic field, which rotates azimuthally about the center line of the melt. A number of magnet poles that are placed at equally spaced azimuthal positions and connected to successive phases of a multiphase AC power source can produce rotating magnetic field. Therefore besides the effect of SMF on the supercooling degree, rotating magnetic freezing can control the ice growth and lead to the formation of uniform ice crystals without the occurrence of crystal grouping. The study of Sato and Fujita (2006) found that small and uniform ice crystals were formed under rotating magnetic field with low intensity. Similar results were obtained by Wang *et al*. (2006), when both rotating magnetic field and the combination of steady magnetic field and electric field were used during the vertical gradient freezing process. Wang *et al*. (2006) also showed that the effect of the combination of steady magnetic field and electric field was more prominent.

OMF is another magnetic technology that can influence ice formation. The effect of OMF on freezing is mainly due to the OEF generated by OMF, according to the Maxwell Faraday equation (Wowk, 2012). Mochimaru *et al*. (2008) reported that OMF could improve the cryo-preservation of porcine ovarian tissue, but OMF had no effect on the freezing point of distilled water. Up to now, no studies about the OMF-assisted freezing of real food products have been reported.

The main affecting factor of MF is the intensity of magnetic field, which cannot be too low or too high. For example, there was no significant difference between food frozen with a 0.0005 T magnetic field and control blank experiments (Suzuki *et al*., 2009), however, if the MF intensity increased, water started to levitate. Tagami *et al*. (1999) investigated the growth of crystal in the magnetic field and found that the water globules with the diameter of 6 mm levitated in a magnetic field at 18 T, and the globule still stayed in a supercooled liquid state at -10 °C. This result indicated the insignificant effect of the degree of supercooling. Except for this, as the above, the material, shape and size of the device also affect the effectiveness of MF.

Although claim was made that better food preservation could be obtained by using commercial freezers with a magnetic field generator (Suzuki *et al*., 2009), the application of MF on food freezing is scare, and most of the studies (Suzuki *et al*., 2009; Woo and Mujumdar, 2010;

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Aleksandrov *et al*., 2000) are just on water.

4.3 Radiofrequency freezing (RF)

Radiofrequency belongs to electromagnetic field, which has been rarely used in freezing. RF waves can generate torque in water molecules through changing their equilibrium relationships in the ice cluster (Anese *et al*., 2012), and water dipole rotation can thus be exploited to control the size of ice crystal during freezing. Anese *et al*. (2012) utilized RF at low voltage pulses (2 kV) to assist cryogenic freezing with liquid nitrogen of pork, and results were compared with air blast and cryogenic fluid flow freezing, showing that the RF-frozen meat exhibited a better cellular structure, less intercellular voids and less cell disruption. Meanwhile, small ice crystals were formed and well-distributed the intracellular region. The better microstructure of pork after RF-assisted freezing was due to the ability of RF to decrease the freezing point and consequently promote the ice nucleation. Similar effect on the freezing point is also obtained by the utilization of MF during freezing, as discussed previously. It should be pointed out that the liquid nitrogen used as a cooling media during RF freezing is expensive, and other freezing media could be investigated in future study.

4.4 Microwave assisted freezing (MWF)

Just like the principles of EF and MF, intrinsic electric dipole moment of water molecule can be changed by microwave, and there microwave assisted freezing can impact on ice formation. Jackson *et al*. (1997) suggested that the combination of microwave radiation and a cryo-protectant could potentially control ice formation in cells and tissues, thus improving the long-term cryo-preservation of biomaterials. However limited investigations had been reported in this field. Chaplin (2013) suggested that lower frequency MV (915 MHz vs 2450 MHz) had significant and lasting effects on liquid water. Xanthakis *et al*. (2014) investigated the MWF of a food matrix and selected pork samples and indicated that the degree of supercooling and the average ice crystal size was decreased by about 92% and 62%, respectively, under the tested conditions as compared to the conventional freezing process. It should be pointed out that this was the first application of MWF on a real food system, and further studies are needed confirm the outcome.

5. Dehydrofreezing

Dehydrofreezing is a technique that includes two independent steps: namely osmotic dehydration and freezing, and thus dehydrofreezing is also termed as osmo-dehydro-freezing (ODF). This method is mainly used for freezing of fresh fruit and vegetables, as these samples often contain abundant water, which can result in the delay and impede the progress of freezing process. For fruit and vegetables, their cell walls are less elastic than their cell membrane. As a consequence, large ice crystals can form easily during freezing process. The dehydration treatment prior to freezing can remove part of cellular water in fresh samples, thus resulting in the decreases of freezing point and freezing time during the subsequent freezing process. Due to the acceleration of freezing process, the food quality can be improved. The main advantages of ODF can be summarized as low energy consumption, low cost for packaging, distribution and storage, and improved food quality (Piotrowski *et al*. 2004; Ramallo & Mascheroni, 2010). Currently, salts, sugars and corn syrups are widely used as osmotic solution for dehydration. Among them, salts are mainly used for vegetable dehydration, while sugars and corn syrups are usually used for fruit dehydration.

Occasionally, osmotic dehydration (OD) has a positive effect on the instantaneous food freezing-thawing process. However, the dehydration pre-treatment cannot benefit the cryo-preserving process of frozen food. Giraldo *et al*. (2003) investigated the impact of ODF on the volatile profile of kiwi, and showed that ester contents increased, whereas the contents of aldehydes and alcohols decreased after the OD treatment. After one-month storage, the difference in the volatile composition between frozen samples with and without dehydration pre-treatment was insignificant. On the other hand, the study of Blanda *et al*. (2009) found that the dehydration pre-treatment resulted in the enrichment of aromatic compounds in frozen strawberries, whereas some native aromatic compounds was lost after freezing, it was thus supposed that dehydration could remove some peculiar smell of food and provide food new fragrance. Some nutritional compounds in foods may be lost during dehydration, due to the mass transfer of food solutes from samples to the surrounding solution, as confirmed by the study of Peiró *et al*. (2006) and Rizzoloa *et al*. (2007), who indicated that the dehydration treatment resulted in the decrease of contents of ascorbic acid, citric acid, galacturonic acid, and other components in pineapple and grapefruit.

The main factors affecting ODF are the permeable membrane (Clark & Bihan, 2000), concentration and agitation degree of the osmotic solution, besides the type, size and geometry of the sample (Taiwo *et al*., 2001). In addition, ultrasound and pressure are combined with ODF in order to enhance the freezing process. Since OD treatment cannot reduce the water activity completely and prevent the proliferation of microorganisms, the shelf life of processed product is normally short. High hydrostatic pressure can increase the mass transfer rates and cell permeability and therefore the combination of OD and high hydrostatic pressure had been used to process various food products, such as strawberries (Nuñez-Mancilla *et al*., 2013;

Nuñez-Mancilla *et al*., 2011) and chicken breast (Leng *et al*., 2013). On the other hand, for melon, apple, kiwifruit and pineapple, the OD treatment in the presence of ultrasound irradiation has been demonstrated to intensify the mass transfer process, increase the water diffusion coefficient and better preserve the food original quality (Fernandes *et al*., 2008; Cárcel *et al*., 2007; Nowacka *et al*., 2014; Fernandes *et al*., 2009). ODF had been applied on many vegetables and fruits, such as kiwifruits (Talens *et al*., 2003), strawberries (Blanda *et al*., 2009), mangos (Chiralt *et al*., 2001), apples and pears (Marani *et al*., 2007). Although the dehydration pre-treatment can benefit the subsequent freezing process and the processed food products, ODF also has some negative influences. For example, the study of Marani *et al*. (2007) found that ODF was able to preserve the color and texture of apples and decrease the drip loss of apples, whereas unsatisfactory results were obtained in the case of kiwi freezing by ODF, however ODF was still regarded as an effective and significant method for frozen apples.

Besides experimental studies on the quality of final products by ODF as discussed above, mathematical modelling on the heat and mass transfer process occurring during ODF has also been carried out (Goula & Lazarides, 2012). In general, heat and mass transfer occurs in the ODF process. The mass transfer process involves the migration of cellular compounds and water between the food samples and the osmotic solution, while the heat transfer occurs accompanied by the phase change during freezing. The mass transfer process usually affects the thermo-physical properties of food, thus influencing the heat transfer profoundly. There are many studies focused on modeling the mass transfer during OD process, as well as the heat transfer during freezing (Rizzoloa *et al*., 2007; Marani *et al*., 2007; Giraldo *et al*., 2003; Floury *et al*., 2008; Pham 2006; Wang *et al*., 2007). However, limited studies are available to investigate both

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the heat and mass transfer processes simultaneously during ODF. Agnelli *et al*. (2005) developed a model to describe the heat transfer process by using the enthalpy formulation with a finite volume method, with several hypotheses made about the dehydration step and the results showed a good quantitative agreement between the experimental and calculated data. Moreover, in the study of Goula and Lazarides (2012), the developed models as functions of OD and freeze time were accurate (R^2=0.997-0.999; *RMSE*=0.156-0.252) to predict the temperature and phase changes, as well as the concentration of solute and water during ODF. These studies indicate that modelling the heat and mass transfer process is necessary to enlarge the application of ODF in the food industry.

6. Future trends

The characteristics of each novel freezing method have summarized in Table 1. All methods have shown potentials for applying in freezing foods. For HPF, the cost of the high pressure facilities should be reduced in order to further promote its applications. The feasibility of HPF to freeze some seasonal fruits, such as litchi, longan, peach, etc. should be explored. On the other hand, more researches are expected to understand the advantage of ultrasound for enhancing the freezing process of solid samples and the ice nucleation mechanism. Furthermore, the strategy of varying either ultrasound frequency for UAF or pressure level for PSF at one ice stage, as well as several consecutive ice stages, should be explored in future studies. For both HPF and UAF, it is necessary to develop low cost and safe low temperature media that are not harmful to either the freezing equipment or the food samples.

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For SEF, more studies should be performed to understand the effect and mechanism of SEF on the freezing of various food products. In addition, the scale-up of the electrical equipment, the shape and material of electrode used in the freezing equipment should also be explored in future studies. For MF, further investigations on the mechanism of MF to food systems as well as the potential of OMF for food freezing are needed. As for RF and MWF, future studies should be concerned on the effect of these techniques on freezing of various food products under different processing conditions.

About ODF, future studies can explore the potential of the combination of OD with HPF, UF or other novel methods, and the relationship between the formation and distribution of ice crystals during ODF process and the water content before freezing in order to better understand and utilize the ODF.

7. Conclusions

Food freezing is a complicated process, involving heat transfer, ice nucleation, ice growth, ice distribution, and the changes in food physical and chemical properties.

HPF can increase the degree of supercooling, especially PSF, resulting in the instantaneous occurrence of ice nucleation, rapid growth of ice crystals and desired distribution of ice crystals throughout the sample. UAF aims at decreasing the supercooling degree and increasing the freezing point, which can initiate the ice nucleation and reduce the energy consumption. The ultrasonic cavitation effect can also promote the formation of small and homogeneous ice crystals. As a result, both HPF and UAF can improve the quality of final product. However, the requirements of special freezing media and containers also limit their applications in the food industry.

Both EF and MF have shown the potential to control the spontaneous nucleation process. Strong electric field can promote ice nucleation, as well as the crystallization of supercooled water. The mechanisms of DC-EF and AC-EF on the freezing process are different. The former can initiate ice nucleation, while the latter can postpone ice nucleation. However, detailed effects of EF and MF on food freezing have not been well understood yet. Most of the studies were focused on the freezing of water, instead of real food products. Generally speaking, the application of SEF for food freezing is a relatively new topic. On the other hand, RF can control the size of ice crystal during freezing, but it is rarely used, which may be due to the difficult operation and condition of experiments. In addition, MWF cannot control the nucleation temperature, but can improve the final quality of frozen food by less damage to the microstructure and tissue, which is achieved by decreasing the average size of ice crystals and supercooling degree.

As a pre-treatment prior to the freezing process, OD can remove some water in the sample, enhance the freezing process and reduce the damage of freezing for the plant tissue. ODF has been successfully used in freezing of vegetables and fruits. Furthermore, high pressure and ultrasound could be incorporated into the ODF process to further accelerating the freezing process.

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Table 1. Comparison among various new freezing methods

Method	Principles	Advantages	Applications	Freezing type	Availability
HPF	Increasing supercooling degree; spontaneous nucleation	Formation of intracellular, small and large amount of ice crystals, less damage to food microstructure	Normally for solid foods including meats, seafoods and fruits	Immersion	PAF/PSF/PIF
UAF	Decreasing supercooling degree	Same as with HPF	More suitable for liquid foods	Immersion	High/low frequency
EF	Decreasing supercooling degree	Inducing nucleation	Emerging technique, few applications in foods	Plate	AC/DC
MF	Controversy about decreasing or increasing supercooling degree	Uniform ice growth	Same with EF	Plate	OMF/SMF
ODF	Reducing water content	Shortening freezing time, lowering freezing loads	Fruits and vegetables	Immersion	Different membranes
MWF	Decreasing supercooling degree	Small ice crystal size	Emerging technique, few applications in foods	Plate	-

HPF: high pressure freezing; UAF: ultrasound-assisted freezing; EF: electric field freezing; MF: magnetically distributed freezing; MWF: microwave assisted freezing; ODF: osmo-dehydro-freezing; PAF: pressure assisted freezing; PSF: pressure shift freezing; PIF: pressure induced freezing; DC: direct current; AC: alternating current; OMF: oscillating magnetic field; SEF: static magnetic field.

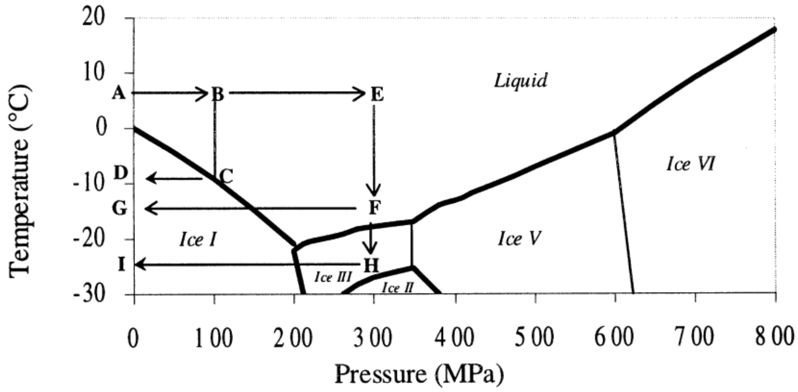

Fig. 1. Phase diagram of water (Le-Bail *et al*., 2002).

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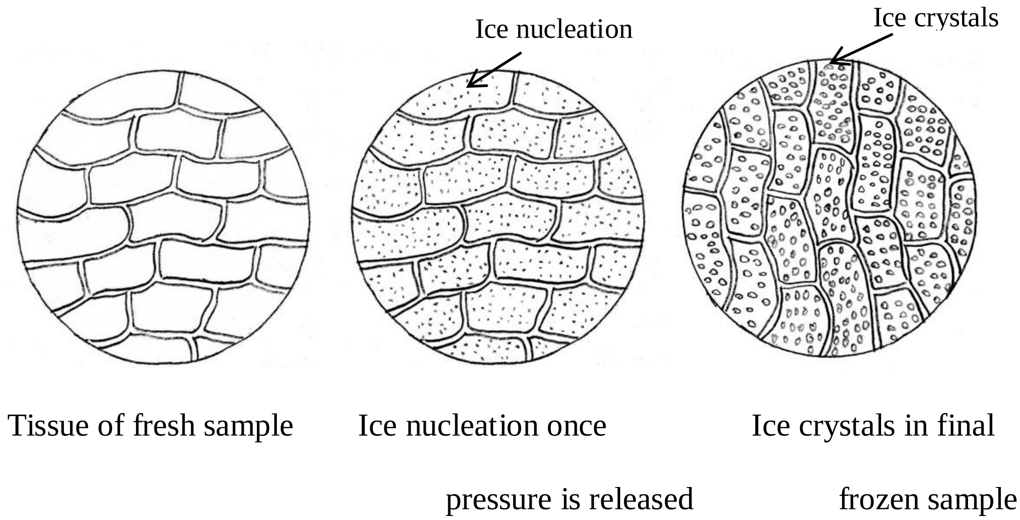

Fig. 2. Formation of ice crystals in tissue during PSF process.

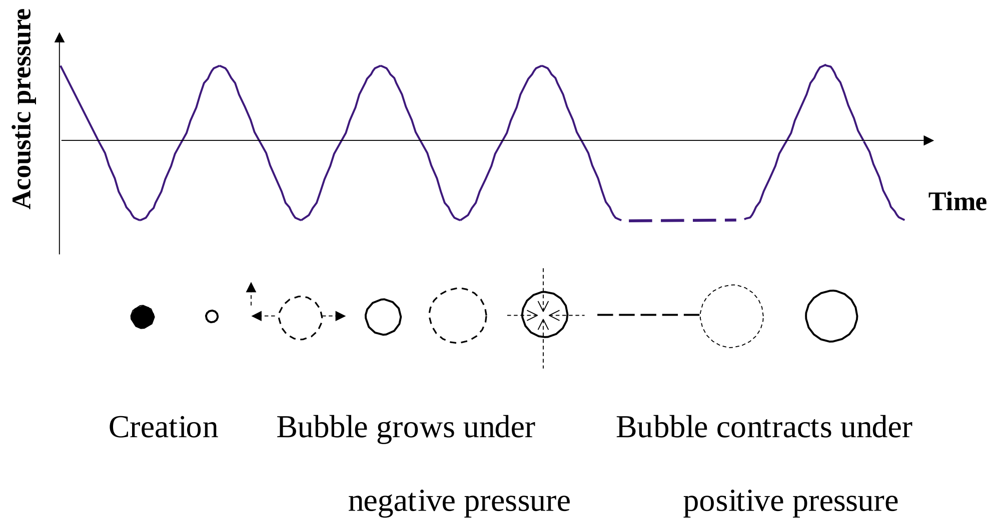

Fig. 3. Motions of bubbles during cavitation.

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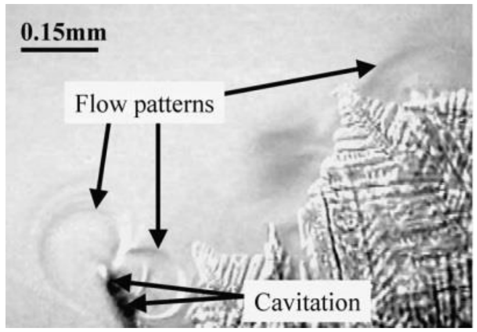

Fig. 4. Circular flow patterns observed around two cavitation bubbles in a 15 wt% sucrose solution (Chow *et al.*, 2003).

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