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Ice adhesion of an aqueous solution including a surfactant with stirring on cooling wall: ethylene glycol—a silane coupling agent aqueous solution

Hiki Hong[a,1], Jong Hyeon Peck[b,2], Chaedong Kang[c,*]

[a]*School of Mechanical and Industrial System Engineering, KyungHee University, Yongin 449-701, South Korea*
[b]*KITECH, HVAC/R Research Team, Chonan 330-820, South Korea*
[c]*Department of Mechanical Engineering, Chonbuk National University, 664-14 Duckjin-dong, Jeonju 561-756, South Korea*

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Abstract

Ice adhesion to the cooling wall directly hinders continuous ice formation or system performance due to the increase of flow and thermal resistance. In order to obtain the basic characteristics of two- or three-component aqueous solutions with EG, SCA and water on freezing, the influence of component ratio and supercooling degree on those solutions was investigated through a batch type freezing process of the solutions. Moreover, the strength of ice adhesion was shown to vary by stirring power. Ice adhesion was suppressed when (1) the solution including the additive SCA at comparatively high concentration; (2) the solution had high initial concentration of EG or SCA; (3) the brine temperature was higher; (4) the supercooling degree was comparatively small in the lower concentrations. No ice adhesion occurred at the stirring power below 30 W. Furthermore, particle size of the ice slurry was smaller in higher concentrations.

Keywords: Ice; Wall; Aqueous solution; Additive; Surface tension; Mixture; Two-phase mixture

Adhérence de la glace, formée à partir d'une solution aqueuse contenant un agent tensio-actif, soumise à l'agitation, sur une paroi refroidie—solution aqueuse contenant un couple à base de silane

Mots clès : Glace ; Paroi ; Solution aqueuse ; Additif ; Tension superficielle ; Mélange ; Mélange diphasique

1. Introduction

Ultimately, high heat transfer rate and transportability are the merits of a system with continuous ice formation and storage, a so-called dynamic-type ice storage system (DISS) [1]. The peculiarity comes from fine particle ice of micrometers scale. However, ice adhesion to the cooling

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* Corresponding author. Tel.: +82-63-270-2318; fax: +82-63-270-2315

E-mail addresses: hhong@khu.ac.kr (H. Hong), pjh6240@kitech.re.kr (J.H. Peck), ckang@chonbuk.ac.kr (C. Kang).

[1] Tel.: +82-31-201-2925; fax: +82-31-202-2625.

[2] Tel.: +82-41-589-8343; fax: +82-41-589-8330.

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Nomenclature

C_{pw}	specific heat of water (kJ kg^{-1} K^{-1})
C_a	equivalent specific heat of solution ($\approx C_{pw}$)
m	mass, total mass (g, kg)
$q(\tau)$	cooling heat transfer per unit mass just after dissolution of supercooling (kJ kg^{-1})
$T_{\mathrm{fp}}(\tau)$	freezing point (°C)
T_{sc}	supercooled temperature just before freezing (°C)
ΔT_{sc}	supercooling degree, $T_{\mathrm{fp}}(0)-T_{\mathrm{sc}}$ (K)
x	concentration (mass%)
Greek	
λ	latent heat (kJ kg^{-1})
τ	freezing time starting from dissolution of supercooling (h)
ω	stirring velocity (rpm)
subscript	
a	component of EG and SCA
brine	cooling brine
EG	ethylene glycol
fp	freezing point
ice	ice
mixture	mixture of ice slurry and solution during freezing
SCA	silane coupling agent (3-aminoprophyltri-ethoxysilane)
sc	supercooled, supercooling
water	water

wall continuously or intermittently has been an issue in DISS. The reason is that the ice adhesion directly affects the performance and stability of the DISS due to the increase of flow and thermal resistance.

Several studies of fine particle ice formation on a cooling wall have been performed using aqueous solutions or some additives concerned with cold preserving [1–6]. Corresponding with natural convection, ice removal conditions of EG aqueous solution on various cooling walls were observed by Hirata [7]. Alcoholic materials such as ethylene glycol (EG), propylene glycol (PG) and ethanol are well known as brines, which are not only easy to mix in water, but also have well-opened properties. EG consists of hydroxyl and ethylene. The hydroxyl part bonds with inorganic material or water molecules. In contrast, the ethylene part with hydrophobic material displaces to the empty space between water molecules. Thus, EG disperses well in water. Due to these merits, the EG solution also has been used in ice slurry making.

An additive, anti-freeze protein (AFP) [3], was reported as an effective material in the role of suppressing ice growth. Surfactant is useful for reducing the unbalance of free energy state on interface. Another respect of ice formation using an emulsion that mainly consists of water, silicone oil and an amount of surfactant, i.e. silane coupling agent (SCA), was proposed by Okada et al. [8,9]. From the ice formation, it was revealed that the mixture was considerably effective to suppress ice adhesion to the cooling wall, and ice adhesion condition was expressed to the material of the cooling surface and cooling heat flux. Especially, it is noticeable that the cohesion between ice particles was suppressed during long-term ice storage [9]. The SCA reacts chemically with water and forms an alcoholic material called silanol in a determined ratio. The silanol has both the hydrophilic and hydrophobic part in itself, and hence lies between organic and inorganic material. Such a peculiar characteristic like amphoteric surfactant contributes to suppress ice growth in the solution.

In previous studies, SCA in emulsion was effective to suppress ice adhesion; moreover, it is possible to obtain continuously ice slurries with high IPF [10–12]. At present, several practical ice slurry systems with aqueous solutions are operated with a mechanical driven slurry-forming device. However, it is an obstacle to simplify and stabilize the system.

When the EG of a coolant and the SCA of a surfactant mix in water, a new aqueous solution with three components is formed. It is worth observing that the ice formation process of the solution is applicable to understanding the freezing problem on a functional solution system. It is worth-while to investigate the role of the EG and the SCA which have been known to as a material of the suppression of ice growth in emulsion [10–12]. However, the related study of the contribution of the EG and the SCA in aqueous solution have been reported yet. In this study, therefore, in order to obtain the basic characteristics of two- or three-component aqueous solutions on freezing, the influence of component ratio and supercooling degree of two or three component aqueous solution with EG, SCA and water was investigated during freezing of the solution. The strength of ice adhesion was shown at variations of stirring power.

2. Experiments

2.1. Freezing point of solution

When a freezing process occurs in an aqueous solution with three components, the freezing point of the silanol solution helps in observing the freezing state and the amount of ice formation in the solution [8] Because the properties, such as chemical formula, phase diagram and so on, of the three components (water–EG–SCA) mixing in solution have been nearly unknown because of their complexity, it is difficult to find the effect of component changes on the system. Thus the quantity of one of the components is constant in this experiment. It was ascertained that 3 mass% of silanol aqueous solution is sufficient to obtain the effect of the surfactant by Okada et al. [8]. In order to observe the freezing point of each concentration, a series of solution

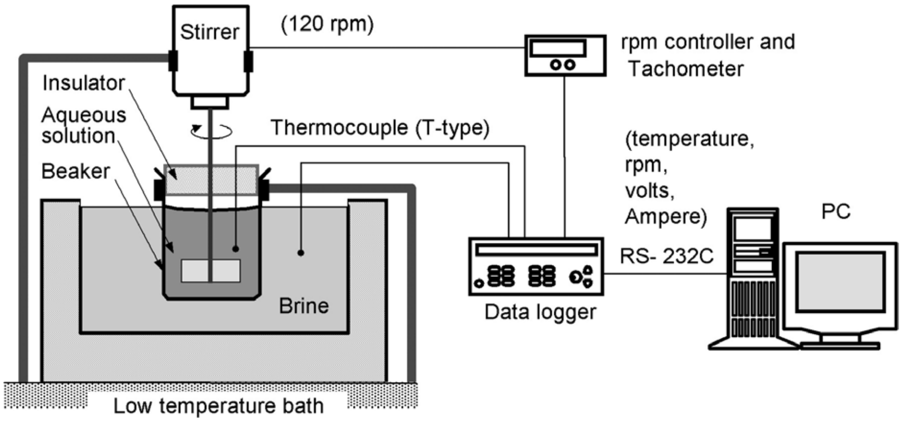


Fig. 1. Experimental apparatus of solution freezing.

were prepared in glass tubes (ϕ13.4×120 mm) with a volume of 13 ml and agitated a few seconds before cooling. The temperature of the test solution in the glass tube, which was cooled continuously from room temperature to a prescribed supercooling degree, was measured with Copper–Constantan thermocouple (ϕ0.3 mm). The highest temperature which was obtained from just after dissolution of supercooling, was regarded as the freezing point of every solution. During the cooling process all the data, i.e. the temperature of the solution, the low temperature bath and surrounding, were measured by a data logger (Agilent 34970A, 16 data channels), with a period of 5 s, and saved to a personal computer (P-233 MHz) with HP-VEE measuring software.

2.2. Freezing process

Fig. 1 shows the experimental apparatus for ice formation and measurement system. A series of aqueous solutions for freezing were used, which consisted of an additive (SCA: 3-aminoprophyltriethoxysilane, molecular weight of 221, Toshiba Chemical Co.), water and ethylene glycol (EG). The total mass of each solution was set at 300 mg.

Each solution had a different component ratio as shown in Table 1, and was stirred with a stirrer in a beaker (SUS304, ϕ 73.0±0.5×120 mm -1t). Total concentration of mass percentage from cases 1 to 4 is equal. On the other hands, the effect of the EG concentration in three-component solution was observed from the case 2, 5 and 6. The stirrer consisted of a motor and vertical plate type impeller of 30 mm×20 mm (three pieces with 120° pitches). From preliminary experiments, no waving on the solution top surface was observed under about 150 rpm. However, as ice formation occurred and continued in the beaker, the rotational speed decreased because of the increase of the stirring torque. For all experiments the stirring velocity of solution was kept to 120 rpm. The variations of the stirring velocity and stirring power are important in measuring the physical behavior of ice formation and growth. The outer wall of the beaker was cooled by brine of −7.5 °C in a low temperature bath. Heat transfer from the surroundings was restrained by a polystyrene foamed cap. During the cooling process the temperatures of the solution and the brine were measured with copper–constantan thermocouple. All the measured data was recorded every 5 s and stored by a data acquisition system, which consisted of a data logger (Agilent 34970a) and PC. When the solution temperature reached the target supercooling degree ($\Delta T_{sc}=2$ K in Table 1), the dissolution of supercooled solution was compulsorily initiated by putting ice particles of less than one gram into the solution. This freezing process started just after the dissolution of the supercooled solution to freezing point. Then, ice particles in the solution increased over time. However, the amount of ice product is difficult to measure because of the complexity of the thermo-physical property and geometry of ice slurries. In order to decide the time to finish the freezing, the cooling continued until the stirrer met with a hard ice layer in the beaker.

Table 1
Concentration of aqueous solution (mass%)

Component	Cases					
	Case 1	Case 2	Case 3	Case 4	Case 5	Case 6
EG	4	3	1	0	4	7
SCA	0	1	3	4	3	3
Water	96	96	96	96	93	90

3. Results and discussion

3.1. Freezing point

Fig. 2 shows the freezing point just after dissolution of supercooling $T_{\mathrm{fp}}(0)$ for each aqueous solution with the given concentration ratio. The concentration means the percentage of the mass of EG+SCA to total mass. Although the freezing point decreases in quadratic form with the increase of concentration, it was assumed as a linear function in comparatively low concentration regions below 12 mass%. Thus, the relation between the freezing point, $T_{\mathrm{fp}}(\tau)$ and the concentration, $x(\tau)$ is obtained by Eq. (1) with ± 0.12 °C (reliability 98.5%). However, for SCA only solution (case 4), the Eq. (1) was adopted from the relation of Ref. [8], because it was different from that of the EG+SCA one. Then the ice amount, $m_{\mathrm{ice}}(\tau)$ is predicted by Eq. (2). With the assumption that the specific heat of the EG is equal to that of the SCA, the heat transfer for cooling during ice formation $q(\tau)$ for the mixture temperature $T_{\mathrm{fp}}(\tau)$, relates to the ice amount $m_{\mathrm{ice}}(\tau)$ by Eq. (3). In addition, the amount of ice just after the dissolution of supercooling was ignored due to the small quantity in comparison to the total mass, 300 g. The latent heat of the ice obtained from the EG and SCA mixing solution might be different than that of the pure ice. Previously, the latent heat of 240 kJ/kg, which is the experimental result [8] of an SCA solution, was revealed. Instead of the unknown property of the mixture of EG and SCA aqueous solution, the latent heat of SCA solution was used to this experiment.

$$T_{\mathrm{fp}}(\tau) = Ax(\tau) + B \tag{1}$$

where

$$x(= x_{\mathrm{EG}} + x_{\mathrm{SCA}}) \geq 4 \text{ mass\%}$$

$A = -0.356$, $B = 0.093$ for mixing solution of EG and SCA; $A = -0.292$, $B = 0$ for SCA only solution (Ref. [8])

$$x(\tau) = \frac{m_{\mathrm{a}}}{m_{\mathrm{total}} - m_{\mathrm{ice}}(\tau)} \times 100\ [\%] \tag{2}$$

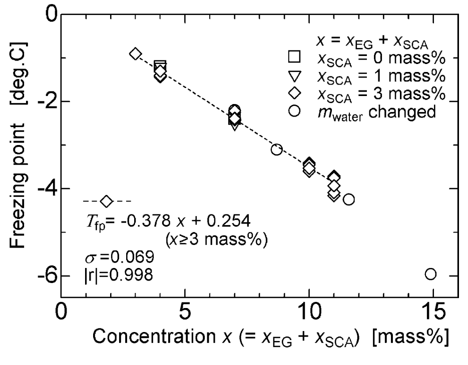


Fig. 2. Freezing point of EG+SCA aqueous solution to SCA composition.

where

$$m_{\mathrm{total}} = m_{\mathrm{w}}(0) + m_{\mathrm{a}}, \quad m_{\mathrm{a}} = m_{\mathrm{EG}} + m_{\mathrm{SCA}}$$

$$q(\tau) = \lfloor m_{\mathrm{a}} C_a (T_{\mathrm{fp}}(\tau) - T_{\mathrm{fp}}(0)) - (m_{\mathrm{ice}}(\tau) + m_{\mathrm{ice,sc}})\lambda_{\mathrm{ice}} \rfloor / m_{\mathrm{total}} \tag{3}$$

where

$C_{pw}(= 4.18 \text{ kJ/kg K}) \approx C_a = (C_{\mathrm{EG}} + C_{\mathrm{SCA}})/2$,

$\lambda_{\mathrm{ice}} = 240$ kJ/kg and

$$m_{\mathrm{ice,sc}} = -m_{\mathrm{w}} C_{pw} (T_{\mathrm{fp}}(0) - T_{\mathrm{sc}})/\lambda_{\mathrm{ice}}$$

$$m_{\mathrm{ice}}(\tau) = m_{\mathrm{total}} - \frac{100 A m_{\mathrm{a}}}{T_{\mathrm{fp}}(\tau) - B} \tag{4}$$

3.2. Freezing experiments

Fig. 3 shows the freezing process of EG 4 mass% solution without SCA (case1). The solution in the beaker was stirred to 120 rpm by impeller, and was cooled by low temperature brine at -7.5 °C. When the solution reached the prescribed supercooling state, ice particles of about 1 mg were inserted in the solution under stirring and the supercooling was dissolved immediately with ice formation, i.e. freezing started in the solution and on beaker wall. As the freezing started, the temperature of the mixture of ice and solution returned to the freezing point, -1.2 °C at the initial concentration of the solution. After that, the freezing point decreased gradually proportionally to the concentration of solution. Corresponding to the decrease of the freezing point, the rotating speed abruptly decreased from the resistance of the ice contact. At the time of 50 min from the dissolution, the impeller in the beaker stopped with the resistance of hard ice growing from the beaker wall. Ice adhesion occurred in the beaker. At that time, the extent of ice adhesion to the cooling wall was investigated by reversing the beaker. When the ice in the beaker was weakly attached to the wall, it slipped and separated from

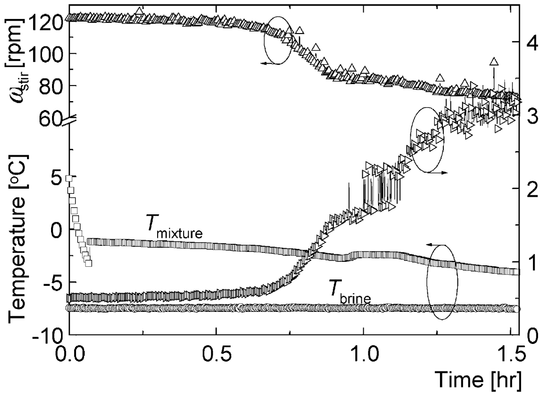


Fig. 3. Time history of temperature, power and stirring velocity during EG 4 mass% solution freezing.

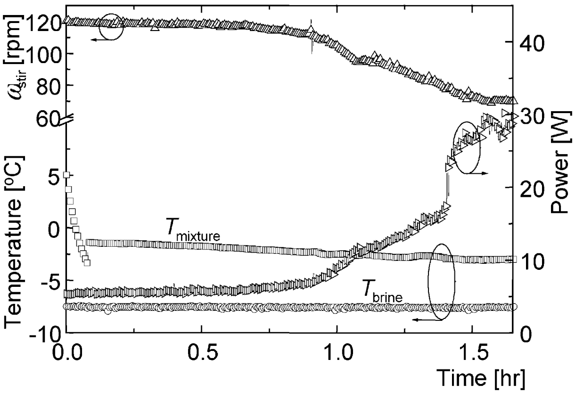


Fig. 4. Time history of temperature, power and stirring velocity during EG 3 mass% + SCA 1 mass% solution freezing.

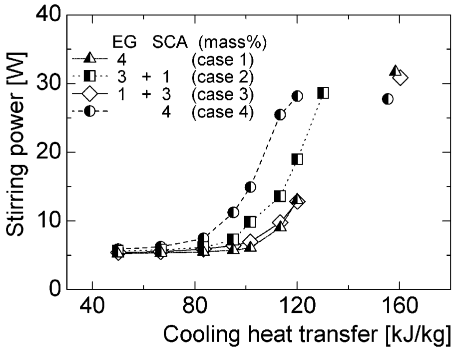


Fig. 5. The stirring power variation to cooling heat transfer for total concentration of 4 mass% ($\Delta T_{sc} = 2$ K, $T_{brine} = -7.5$ °C).

the beaker, denoting the ice was in a non-adhesion state. However, it only showed qualitatively whether the ice adhesion occurred or not. Several measurements of the intensity of sheet-like ice adhesion to metal surface [7], or to the stress between score micrometers of ice particles [13,14], have been reported. In this experiment, a quantitative

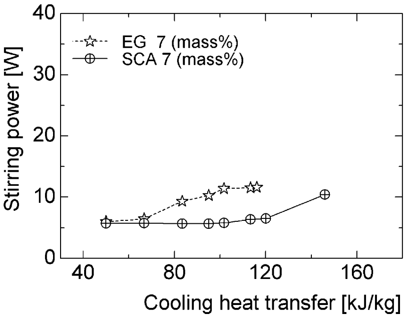
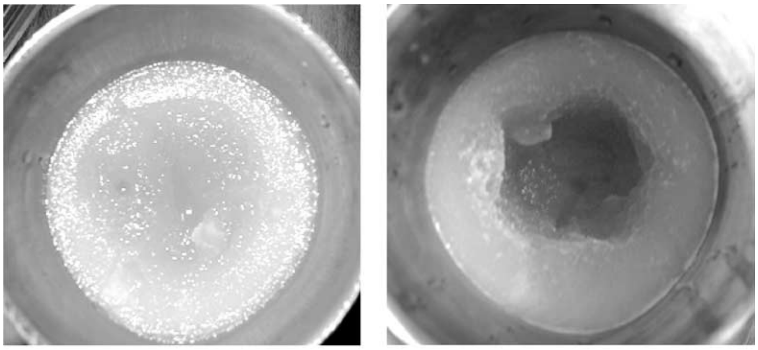


Fig. 7. The stirring power variation to cooling heat transfer for total concentration of 7 mass% ($\Delta T_{sc} = 2$ K, $T_{brine} = -7.5$ °C).

approach, i.e. measuring the power (or torque) for stirring was tried during the ice formation process. For relative comparison, stirring power during water freezing was measured in the same beaker. In Fig. 3, the power was kept constant before the ice growing from the cooling wall reached the stirring blade. It increased gradually as the blade came into contact with the hardened ice. Fifty minutes later, an abrupt and unstable increase appeared, i.e. the stirring resistance of the blade to the edge of growing ice increased suddenly. After that, the power was settled at about 32 W. The time for estimating the cooling process was determined from the cooling heat transfer in Eq. (3). However, according to circumstances, the finish time for freezing may be faster by ice adhesion than that by the prescribed heat transfer for cooling.

Fig. 4 shows the temperature and the stirring power of the solution in case 2 (SCA 1 mass% added to EG 3 mass% solution) during freezing. In Fig. 4, the stirring power decreased less than Fig. 3 (case 1). Moreover, a stiff power jump appeared later than that of case 1 (Fig. 3). This shows that the SCA affects ice cohesion among the ice particles.

Fig. 5 shows the variation of the stirring power to the cooling heat transfer of the case 1–4 during freezing. The symbols in the upper right of the figure indicated the final state of freezing. For the comparatively low concentration solution (4 mass% of total concentration), the stirring power



(a) Just after freezing (b) Slurry removed state

Fig. 6. The state of ice formation in beaker (case 3, $\Delta T_{sc} = 2$ K, $T_{brine} = -7.5$ °C).

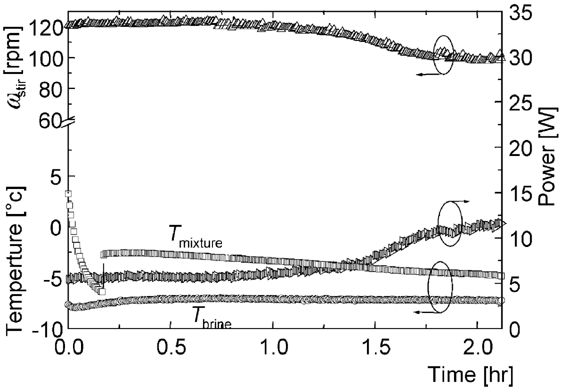


Fig. 8. Time history of temperature, power and stirring velocity during EG 4 mass% + SCA 3 mass% solution freezing ($\Delta T_{sc} = 4$ K, $T_{brine} = -7.5$ °C).

was settled from 25 to 35 W within the cooling heat transfer of 160 kJ/kg. In detail, the stirring power of the SCA-only or SCA-mixed solution (cases 2–4) is higher than the EG-only solution (case 1) below the cooling heat transfer of 120 kJ/kg. This shows that, in the low concentration solution, the SCA had nearly no role to suppress ice adhesion to the SUS wall. In all cases of total concentration of 4 mass%, ice adhesion occurred physically. Thus the stirring power near 30 W indicates skeptical ice adhesion to the SUS cooling wall.

Fig. 6 shows the ice adhesion to the SUS beaker when the freezing process of the EG 1—SCA 3 mass% solution (case 3) was finished. It was confirmed that the ice adhesion to the beaker wall is not so hard but thick (the right of Fig. 6). For concentrations of EG 7 mass% or SCA 7 mass% higher than the 4 mass% of total concentration (Fig. 5), the stirring power was shown in Fig. 7. In the concentration, the SCA solution acted to suppress the stirring power in higher concentration than the EG one. Therefore, it is necessary to observe the freezing process greater than 4 mass% concentration which occurred with the ice adhesion.

For EG 4 mass% plus SCA 3 mass% solution (case 5) the freezing point and the stirring power during freezing, are shown in Fig. 8. The stirring power increased gradually at nearly 80 min and became 12 W at 2 h during freezing. When the freezing process finished, there was no ice adhesion to the beaker wall.

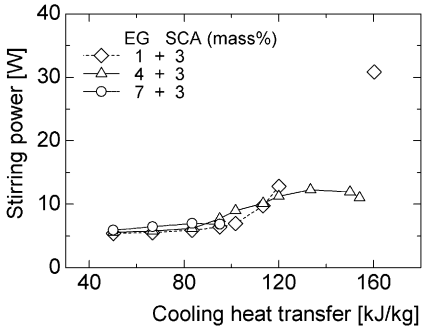


Fig. 9. The stirring power variation to cooling heat transfer for total concentration of 4, 7 and 10 mass% ($\Delta T_{sc} = 2$ K, $T_{brine} = -7.5$ °C).

Fig. 9 shows the stirring power at the initial concentration (cases 3, 5 and 6). In the figure, the stirring power of case 3 (rhomboid) was higher than those of case 5 or 6 when the cooling heat transfer was released above 120 kJ/kg. It was considered that the ice adhesion more easily occurs in a low concentration solution than in a high concentration one. On the contrary, no ice adhesion occurred in cases 5 and 6. Thus, at least, no ice adhesion to the beaker occurred with a stirring power of less than 12 W. However, the highest concentration in this experiment (case 6) caused a small cooling heat transfer, i.e. small ice formation because of the freezing point depression, even though no ice adhesion occurred.

Fig. 10 shows the snapshots of ice slurries pictured by CCD camera just after dissolution of supercooling. In the figure, the size of the ice particles ranges from 100 to 500 μm. Furthermore, the particle size decreased as the initial concentration increased.

The cooling time during freezing was influenced by the brine temperature. As the concentration increases, the cooling heat transfer decreases proportionally to the difference between the freezing point and the brine temperature.

Fig. 11 shows the stirring power of the EG 4% + SCA 3 mass% solution (case 5) to the various brine temperatures. In each case, the supercooling degree is equal to 1 K. From the figure, the stirring power in the brine temperature of −5.5 °C was smaller than that in −7.5 or −9 °C for each cooling heat transfer. Furthermore, the gap became larger as the cooling

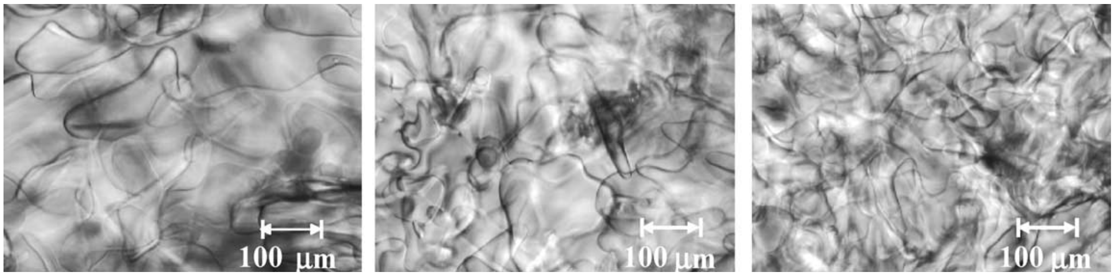


Fig. 10. The shape of ice of just after freezing for the case 3, 5 and 6 ($\Delta T_{sc} = 1$ K, $T_{brine} = -7.5$ °C).

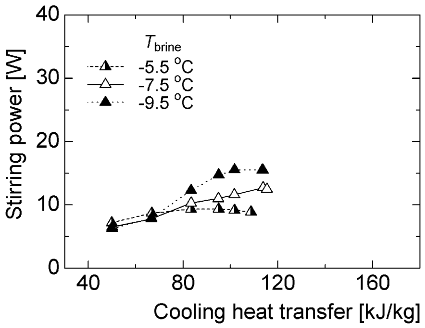


Fig. 11. The stirring power variation to cooling heat transfer for the brine temperature of −5.5, −7.5 and −9.0 °C (case 5, $\Delta T_{sc}=2$ K).

heat transfer increased. This shows that low brine temperature brings the solution to large cooling heat transfer, and leads the ice growth to be rapid rather than gradual.

Fig. 12 shows the relations between the stirring power and the supercooling degree. In the lower concentrations, higher stirring power on the large supercooling degree (4 K) was obtained than on the small one (2 K). It is considered that the ice adhesion is easier to occur because the larger supercooling degree helps to ice growth more rapidly in lower concentration. However, there was no influence of the supercooling on the solution with higher concentrations.

In the experiment, the possibility of suppressing ice adhesion to the cooling wall in an EG aqueous solution with SCA was confirmed. However, there was little or no improvement in the suppression of ice adhesion to a metallic cooling wall and the mitigation of the ice cohesion in aqueous solution without organic—immisible to water or hydrophobic—material (e.a.) because the SCA has the characteristic of an amphoteric surfactant. Therefore, more various additives and other improved material on the cooling wall are needed to supplement the demerits of the SCA, such as high cost and chemical toxicity.

4. Conclusion

Two- or three-component (EG, SCA or EG+SCA) aqueous solutions were stirred and frozen simultaneously in SUS beakers. Through the study of the aqueous solutions, the results for freezing behavior and ice formation were obtained as follows:

The shape of ice particle was smaller as the concentration of EG in solution increased. Furthermore, ice adhesion was suppressed when

(1) the additive SCA included in the total concentration was 7 mass% (case 5) rather than 4 mass% (case 1–4)
(2) (the total concentration of 4 mass% solution, case 1–4),in the EG solution than in the SCA one, the cooling heat transfer was below 115 kJ/kg
(3) the solution had high initial concentration for EG or SCA
(4) the brine temperature was higher
(5) the supercooling degree was small at the low concentration of 4 mass%
(6) furthermore, the stirring power indicated 30 W approximately to skeptical ice adhesion and below 15 W to no ice adhesion
(7) the cooling heat transfer shrank as the brine temperature or the initial concentration increased
(8) the particle size of the ice slurry was smaller in higher concentrations

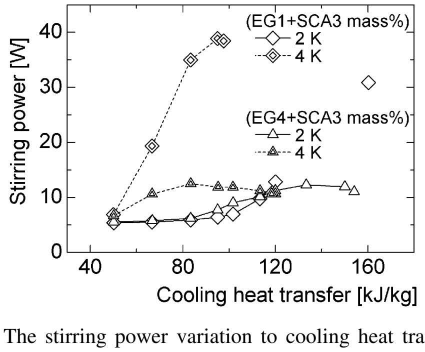


Fig. 12. The stirring power variation to cooling heat transfer for supercooling degree of 2 and 4 K (case 3 and 5, $T_{brine}=-7.5$ °C).

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