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	キーワード (Ja):
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	作成者: Klinmalai, Phatthranit
	メールアドレス:
	所属:
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Name: Miss Phatthranit Klinmalai Student ID: 1461020 Course of Applied Marine Biosciences Advanced Food Science and Technology

## RECRYSTALLIZATION BEHAVIOR OF ICE CRYSTALS IN SUGAR-BASED SOLUTIONS

## Summary

Freezing is regarded as one of widely used method for long term food preservation. At the low temperatures used for many frozen food products storage and distribution, an increase in the mean size of ice crystals by recrystallization is a major difficulty leading to quality deterioration such as degrading the smooth texture in ice cream and increasing the cell structure damage leading to drip loss in meat. Recrystallization is defined as any change in the size, shape, orientation, or perfection of individual crystals after the completion of solidification. Therefore, controlling and predicting ice recrystallization is important to increase the quality of frozen food. The aim of this work was to investigate the systematic understanding of ice recrystallization behavior in sugar-based solution containing mono-, di-, and trisaccharides, polysaccharides and protein that represent simple model food.

Saccharides are widely used in the basic component in many foods. Dielectric relaxation spectroscopy of series of mono and disaccharide solutions (maltose, sucrose, glucose, and fructose) were carried out as a tool to evaluate free water mobility in these solutions. Moreover, the correlation between the fast dielectric relaxation time which may be originated from the movement of free water and ice recrystallization rate constant in these solutions were examined. The results revealed that the dielectric relaxation spectroscopy was a useful method to obtain parameter for predicting and controlling ice recrystallization rate constant in these solutions. The difference of ice recrystallization rate constant was explained well by the difference of the fast dielectric relaxation in freeze-concentrated matrix although the combination effects of solutions such as types of solutes, temperatures, and concentrations existed. The difference of ice recrystallization rate between di and trisaccharide solutions (trehalose, sucrose, and mixture of raffinose and trehalose) was also explained well by the fast dielectric relaxation time in freeze-concentrated matrix. Proton spin-spin relaxation time of water molecules obtained by using nuclear magnetic resonance (NMR) can also explain in fairly good manner and predict the difference of ice recrystallization rate between sucrose and trehalose. It was suggested that the observed trend of smaller ice recrystallization rate constants in trehalose solution and mixture solution of raffinose and trehalose were originated from smaller water mobility in freeze-concentrated matrix of these solutions.

Ice recrystallization behavior in sucrose solutions containing locust bean gum (LBG) and xanthan gum (XG) were investigated as a model of polysaccharide system. The effects of LBG and XG on ice recrystallization behavior were discussed by using the measured physical properties of water mobility, rheological, and microstructure of freeze-concentrated matrix. Increasing the concentrations of polysaccharide did not always give smaller ice recrystallization rate constant. Initial sucrose concentration (20% and 30%) had effect on ice recrystallization behavior in polysaccharide system. It was suggested that high concentration of LBG and XG caused the cryogelation in freeze-concentrated matrix. Elastic behavior of 20% sucrose solution containing 0.50% LBG was higher than that of 20% sucrose solution containing 0.50% XG. Therefore, the content of sucrose in LBG solution entrapped inside gel network may be higher than that in XG solution. The difference of recrystallization rate constant between the higher and lower concentrated sucrose solution in the presence of LBG was larger than that of XG. These results led to the heterogeneity of ice crystals in LBG solution and increasing ice crystals size and ice recrystallization rate in XG solution when compared with sucrose solution as a control sample. Moreover, the difference of structural heterogeneity in freeze-concentrated matrix agreed to the shape of dielectric relaxation spectra and the fitting results of dielectric relaxation, respectively.

The effect of protein addition on ice recrystallization rate constant in 20% sucrose solution was examined by using bovine serum albumin (BSA) as a model protein. The properties of freeze-concentrated matrix were estimated by dielectric relaxation spectroscopy and

differential scanning calorimetry (DSC) in order to discuss the mechanism causing effect of protein addition on ice recrystallization behavior. The addition of BSA reduced the initial ice crystals size but ice recrystallization rate constants of these samples were not significantly different. The fast dielectric relaxation time in BSA system did not change by increasing BSA concentration from 0 to 4% in sucrose solutions. Conversely, the fast dielectric relaxation time in sucrose solution containing 6% BSA was higher than other samples, indicating that the free water mobility increased. It was suggested that this was caused by the incorporation of loosely bound waters in the outer of hydration shell of BSA into free waters. However, ice recrystallization rate constants of 20% sucrose solution containing 6% BSA was not significantly different with other solutions when increasing protein concentration. The reason why the recrystallization rate constant did not change in spite of increased water mobility may be the decreasing the ice content by the addition of BSA.

The knowledge presented herein would be helped as guidance for developing these application of saccharide, polysaccharides, and proteins in not only food freezing but also pharmaceutical and biomedical fields.