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学 位 論 文 要 旨
Dissertation Abstract

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学位論文題目：
Title of Dissertation　A Study of Food Nano-Emulsification Using Low-Energy Method in Aqueous Phase/Polyoxyethylene Sorbitan Fatty Acid Ester/Vegetable Oil Systems
（ポリオキシエチレンソルビタン脂肪酸エステル水溶液/食用油系における省エネルギーなナノエマルション調製に関する研究）

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Low-energy emulsification methods are advantageous to prepare nano-emulsion, since they do not require any expensive special equipment. In low-energy emulsification method, the optimization of phase behavior are very important for nano-emulsion properties because the phases changed during emulsification play a key role in obtaining nano-emulsion with a minimum droplet size and low polydispersity. However, the studies of this emulsification methods using food ingredient are limited. The objectives of the thesis are to study food nano-emulsification using low-energy method in aqueous phase/polyoxyethylene sorbitan fatty acid ester/vegetable oil systems for more understanding the effect of phase behaviors on emulsification. Water or 20% sucrose solution was used as an aqueous phase, four types of polyoxyethylene sorbitan fatty acid esters or Tweens®: Tween20, Tween40, Tween60 and Tween80 were used as surfactants and vegetable oil was used as oil phase.

The effect of sucrose on phase behavior and droplet size in an aqueous phase/Tween®/vegetable oil system was studied. The results showed that addition of sucrose had minimal effects on the phase behavior of Tween20 system. On the other hand, sucrose changed phase behaviors in Tween40, 60, and 80 systems clearly. The sponge phase (L_3) region expanded, the cubic (I_1) and hexagonal (H_1) phases decreased, and the coexisting liquid crystalline (L_c), micellar (W_m), and oil (O) phases were formed by adding sucrose in Tween40 and Tween80 systems. In Tween60 system, Solid of Tween60 (Solid T60) existed for both water and sucrose solution systems. An L_c phases changed to lamellar phase (L_a). In Tween20 systems, the phase transition during emulsification was the main mechanism for preparing a nano-emulsion. In Tween40 and Tween80 systems, an L_3 phase is necessary to produce small emulsion droplet sizes, while the big droplet sizes could have been produced

passing through the H_1 and/or I_1 phase. The emulsifications in Tween60 systems were unsuccessful because of existence of Solid T60 at 25 °C.

For better understanding about the emulsification unsuccessful phenomenon in Tween60 system, the effect of temperature (25, 40, 50, 60, and 70 °C) on phase behavior changes and on droplet size of prepared emulsions was studied. The results showed that Solid T60 at 25 °C disappeared by increased temperature from 25 to 40 °C which resulted in the emulsification was successful. Moreover, increasing temperature caused phase transformations of L_a to H_1 or L_3 or L_c phase, H_1 to L_3, L_3 to S+O or W_m phase, and L_c and I_1 phase to W_m phase. Small droplet sized emulsions with low polydispersity (SD_d) resulted in emulsification process passed through L_3 structure, and avoided the H_1 and I_1 structures.

Furthermore, the effect of temperature (25, 40, and 60 °C) in water/Tween20, 40, and 80/vegetable oil systems was studied. In addition, the emulsions prepared using low-energy emulsification method were compared with the one using high-energy emulsification method. The results clearly showed that smaller droplet sizes emulsion could be prepared by the low-energy emulsification method, whereas very large droplet sizes, bigger than 10,000 nm, were obtained using the high-energy method. For low-energy method, temperature did not show obviously effect on phase behavior of Tween20 system. But the droplet size of emulsions of Tween20 system increased as temperature increased. In Tween40 and Tween80 systems, increasing temperature had an effect on phase transformations and on both droplet sizes and homogeneity of the prepared emulsions as also observed in Tween60 system.

The emulsion which has composition of aqueous phase:Tween®:vegetable oil equal to 0.8:0.1:0.1 exhibited good stability without increased in the droplet size over of storage period for 4 months. Then, the emulsions prepared by two emulsification course, starting form aqueous phase to Tween® ratio at 0.3:0.7 (C-course) and 0.4:0.6 (D-course), in all Tween® systems were diluted to 0.5 and 1.0 wt% of surfactant by pure water and kept at 25 °C for 4 weeks. Diluted emulsions showed highly stable through storage time. However, the size fluctuation and creaming separation occurred in samples whose droplet size was bigger than 500 nm.

In overall results indicate that food nano-emulsification using low-energy method is useful and successively lead to high emulsion stability. Therefore, it is important to select conditions such as starting composition, preparation pathway, and temperature that involve with L_3 phase and avoid H_1 and I_1 phases during the emulsification process.