Effect of Electrolytes on the Formation and Stability of n-Dodecane Nanoemulsions by the Phase Inversion Temperature (PIT) Method

By

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A thesis submitted to the University of Adelaide

For the degree of Master in Engineering Science

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April 2010

Dedicated to my beloved father

Mr. Boon Ling LIEW

17/9/1951 – 20/7/2008

DECLARATION

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Jeffery C LIEW

Date: 9 April 2010

ACKNOWLEDGEMENT

My gratitude goes to many individuals who have and several organisations, which have contributed much to my research in nanoemulsions for the past 2 years. Firstly, to A/Professor Dzuy Nguyen and Dr. Yung Ngothai, thank you for the outstanding supervision, inspiration and support.

Thank you to all the staff in the School of Chemical Engineering, especially Mrs Mary Barrow, for the help in administration and providing the needs for my study. Without the help from the staff of the University‟s workshop, Mr. Jason Peak, Mr. Peter Kay and Mr. Jeffrey Hiorns, my experiments would never be completed. To the staff from Adelaide Microscopy, thank you for your time in helping me in my experiments, especially in Scanning Electron Microscope. The help for editing of the thesis by Dorothy Missingham is gratefully acknowledged.

To all my friends, thank you for your companionship and support during these two years, especially Mr. Shiau Ming Ng for his kindness in providing accommodation, for which I will never forget.

Furthermore, to my lovely lady, Miss Mei Ling Soo, thank you for your support and belief during my candidature. Last but not least, I am grateful to my family for their irreplaceable emotional and financial support during my postgraduate study. My accomplishment of this thesis would not be easy without your blessing.

ABSTRACT

This research focuses on the effect of sodium chloride (NaCl) or calcium chloride ($CaCl_2$) on the formation and stability of n-dodecane/non-ionic surfactant/aqueous nanoemulsions produced by the Phase Inversion Temperature (PIT) method. It is because there are only few works done to investigate the effect of electrolyte on the production of nanoemulsions, especially those produced by the PIT method. Furthermore, in this research, the ability for ageing nanoemulsions to reverse their physical properties to freshly-prepared state has also been investigated..

The nanoemulsions were produced by heating aqueous-continuous emulsions (O/W) to oil-continuous (W/O) emulsions, followed by a quenching process to produce O/W nanoemulsions. Pure milliQ water and concentration from 0.001M to 0.1M NaCl or $CaCl_2$ were used as continuous phase. The non-ionic surfactants used were polyoxyethylene (4) lauryl ether (Brij30) and sorbitan monooleate (S80). The stability was determined by dynamic light scattering technique by measuring the growth of the droplet size and size distribution (PdI) before the nanoemulsions were phase separated, which was determined by visual observation. The reversibility testing was done by measuring the droplet size and PdI as a function of temperatures for a three-day ageing nanoemulsion system.

Droplet size as small as 65nm was produced from a brine system while there was 77nm in a pure milliQ water system, with PdI lower than 0.2. The PIT temperature was found to be depressed when: (1) oil or surfactant concentration increased; (2) HLB number of surfactant system decreased; and (3) NaCl or $CaCl_2$ was added. The transitional temperature (ΔT_{trans}), a temperature difference between the temperature for producing O/W and W/O emulsions, has linked with the stability of the nanoemulsions. The largest ΔT_{trans} in a emulsion system with low oil concentration (R=0.2) appeared at 7wt% Brij30 at pure milliQ water system but at 6wt% Brij30 at NaCl system, showing that the surfactant concentration used to produce the most stable nanoemulsions was reduced by the aid of compression effect from NaCl. When the oil concentration increased, more NaCl was needed to produce stable nanoemulsions. Nanoemulsions produced by a mixture of hydrophilic and hydrophobic non-ionic surfactant was extremely unstable, with a big difference in PIT temperature and ΔT_{trans} compared to a pure surfactant system. It was found that NaCl was a better electrolyte than $CaCl_2$ to produce nanoemulsions with smaller droplet sizes and PdI and higher stability. For the most stable nanoemulsions, 20^{o}C was better than 10^{o}C, to keep the sample from phase separation for more than 30 days. Additionally, the addition of $CaCl_2$ was found to have no difference in the production of nanoemulsions by adding it either before or after the emulsification process.

It has been found that only system with NaCl appeared to have the ability to revert the droplet size and PdI, from ageing to freshly-prepared nanoemulsions. Furthermore, the reversibility ability was governed by phase inversion process as only system experiencing heating process could be reverted. However, nanoemulsions with high stability were partially reversible, where only droplet size was reversible.

The positive effects (increasing stability, smaller droplet size and PdI, and reversibility ability) from the addition of NaCl could only be achieved at certain electrolyte and surfactant concentrations.

PUBLICATIONS

Publication arising from this thesis

1. **Jeffery Chin Long Liew**, Q. Dzuy Nguyen, Yung Ngothai: "The stability of n-dodecane/brine nanoemulsions produced by phase inversion temperature method", Conference Proceedings of *Chemeca 2008: towards a Sustainable Australasia*, Newcastle, NSW, 28 September to **1 October 2008**.

2. **Jeffery Chin Long Liew**, Q. Dzuy Nguyen, Yung Ngothai: "Effect of sodium chloride on the formation and stability of n-dodecane nanoemulsions by the PIT method". Conference Proceedings of *Chemeca 2009: Engineering our future: are we up to the challenge*, Perth, WA, **27-30 October 2009.**

3. **Jeffery Chin Long Liew**, Q. Dzuy Nguyen, Yung Ngothai, (**2009**), "Effect of sodium chloride on the formation and stability of n-dodecane nanoemulsions by the PIT method". *Asia Pacific Journal*, In Press.

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