Preparation of Nano- and Microemulsions using Phase Inversion and Emulsion Titration Methods

MASSEY UNIVERSITY

A thesis presented in partial fulfilment of the requirements
for the degree of Master of Food Technology
at Massey University, Auckland, New Zealand

Yue Wang

2014

Abstract

The formation of nano- and microemulsions with droplet size smaller than 100 nm in diameter and stabilised by non-ionic surfactants was investigated by using two different methods, emulsion phase inversion and emulsion titration. A series of ternary systems consisting of three components (lemon oil, Tween 20 or 80 and water) were prepared at different ratios via gentle agitation by the phase inversion composition method involving the spontaneous formation of microemulsion. The phase behaviour and nano- and microemulsion formation of the ternary mixtures prepared were characterised by visual observation for their phase separation and optical clarity (e.g. transparency and opacity). The samples were also analysed for their particle size and size distribution, viscosity, conductivity and birefringence. As a consequence, phase diagrams based on two different types of small molecule surfactants (Tween 20 or Tween 80) were constructed which define the ratios of three components in the composition of the ternary mixtures that allow the formation of oil-in-water (o/w) or water-in-oil (w/o) nano- and microemulsions. Overall, the o/w microemulsions were found to form at a small region of the ternary phase diagrams with a relatively large ratio of water, compared to w/o nanoemulsion, along dilution lines 1 and 2. On the other hand, w/o microemulsions were determined at the corner of surfactant-rich region along dilution lines 1, 2, 3 and 4 in the ternary phase diagrams. Between the two ternary phase diagrams based on Tween 20 and 80, there were some differences in their composition regions responsible for the formation of nano- and microemulsions as well as for other types of phases formed, including bi- and multiphase, liquid crystals, gel and coarse emulsions. In this study, nano- and microemulsions were also produced by a method called ‘titration method’ involving a two-step process; i) preparation of non-ionic small molecule surfactant-stabilised o/w emulsions by high pressure homogenisation and ii) titration of the o/w emulsions into non-ionic surfactant micelle solutions. Types and concentrations of surfactants (Tween 20, 40, 60 and 80) and oils (sunflower oil, lemon oil, tributyrin, isopropyl myristate and Imwitor 308) were investigated for their influence on the solubilisation of oil molecules from emulsion droplets into surfactant micelles, thus the formation of nano- and microemulsion. The results showed that Tween 60 and Tween 80 had the better capacity of oil droplet solubilisation compared to Tween 20 and Tween 40. The system containing a higher concentration of 2 wt% Tween 80 micelles had the larger capacity of droplet solubilisation than the other systems

containing 0.5 wt% or 1 wt% Tween 80 micelles. In terms of the types of oil used, microemulsions could be fabricated using lemon oil, tributyrin, isopropyl myristate and Imwitor 308, whereas it could not be formed by using sunflower oil due to its high viscosity. Among the oils with relatively low viscosities, the order of the maximum amount of oil incorporated in 1 wt% Tween 80 micelles was Imwitor 308 > lemon oil > isopropyl myristate > tributyrin. This implies the lower viscosity oil has a higher rate of solubilisation in non-ionic surfactant micelles. The effects of pH, salt concentration and heat treatment on the stability of microemulsions were also determined. The results found that the nano- and microemulsion systems prepared by the emulsion titration method were relative stable to pH and ionic strength but sensitive to thermal treatment. This study provides useful information for the rational design of transparent nano- and microemulsions as delivery systems potentially for bioactive compounds for applications in food, beverage and non-food areas.

Acknowledgements

I would like to express my deepest sense of gratitude to my supervisor, Dr. Sung Je Lee, for his guidance, encouragement and generous help throughout the entire project. It was a rewarding experience to work under his supervision and this thesis would not have appeared in its present form without his assistance, support, ideas and substantial corrective comments.

I would like to thank Associate Professor Marie Wong, Ms. Helen Matthews, Mr. PC Tong, Ms. Ninghui (Rachel) Liu, and Mrs Yan Wang for providing trainings, ordering chemicals, laboratory demonstrations, technical help, encouragements and scientific advices and for allowing me to effectively use the lab instrument and facilities.

I also wish to thank Dr. Jingyuan Wen and Alvin Zhou from the University of Auckland for their friendship and support.

Finally, I wish to express my sincere thanks to my mother，Ling Ma, my father, Gongping Wang, and my dear friends for giving me the strength to take up this work and complete it to the best of my ability. Without them, it would have not been possible for me to finish this fantastic journey.

Table of Contents

List of Figures

List of Tables

List of Appendices