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

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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.
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Table of Contents

ABSTRACT ................................................................................................................................. I

ACKNOWLEDGEMENTS ............................................................................................................. III

LIST OF FIGURES ...................................................................................................................... VIII

LIST OF TABLES ........................................................................................................................ XII

LIST OF APPENDICES ................................................................................................................ XIII

CHAPTER 1 INTRODUCTION ........................................................................................................ 1

CHAPTER 2 LITERATURE REVIEW ............................................................................................. 4

2.1 INTRODUCTION .................................................................................................................... 4

2.2 CLASSIFICATION OF EMULSIONS ...................................................................................... 5

  2.2.1 Conventional emulsion ......................................................... 6
  2.2.2 Nanoemulsion ................................................................. 6
  2.2.3 Microemulsion ............................................................... 6

2.3 EMULSION INGREDIENTS ................................................................................................... 7

  2.3.1 Aqueous Phase ............................................................... 7
  2.3.2 Oil Phase ........................................................................ 7

    2.3.2.1 Sunflower oil .............................................................. 9
    2.3.2.2 Lemon oil ................................................................. 9
    2.3.2.3 Isopropyl myristate ................................................... 10
    2.3.2.4 Tributyrin ................................................................. 11
    2.3.2.5 Imwitor 308 .............................................................. 11
    2.3.2.6 Beta-carotene ........................................................... 12

  2.3.3 Emulsifiers ........................................................................ 13

    2.3.3.1 Small molecule surfactants ....................................... 13
    2.3.3.2 Protein emulsifiers .................................................... 18
    2.3.3.3 Polysaccharide emulsifiers ....................................... 19

  2.3.4 Cosurfactants/cosolvents ...................................................... 19

2.4 FORMATION OF EMULSIONS ......................................................................................... 20

  2.4.1 High energy methods ........................................................... 23
    2.4.1.1 High pressure homogenization .................................. 23
    2.4.1.2 Microfluidization ....................................................... 24
2.4.1.3 Ultrasonication ................................................................. 24
2.4.1.4 Homogenization and solvent displacement/evaporation method .... 25
2.4.2 Low energy methods ................................................................. 26
  2.4.2.1 Phase inversion temperature (PIT) ...................................... 26
  2.4.2.2 Phase inversion composition (PIC) .................................... 28
  2.4.2.3 Emulsion inversion point (EIP) ........................................ 29
2.4.3 Emulsion titration/dilution method ........................................... 30
2.5 TERNARY PHASE DIAGRAM .......................................................... 32
2.6 TWO MAIN MECHANISMS OF EMULSION STABILITY ............... 36
  2.6.1 Electrostatic repulsion ......................................................... 36
  2.6.2 Steric hindrance ................................................................. 37
2.7 EMULSION STABILITY ................................................................. 37
  2.7.1 Gravitational separation ....................................................... 39
  2.7.2 Flocculation ........................................................................ 40
  2.7.3 Coalescence ........................................................................ 40
  2.7.4 Ostwald ripening ............................................................... 41
  2.7.5 Phase inversion ................................................................. 42
  2.7.6 Factors affecting the stability of emulsion ............................ 43
    2.7.6.1 pH ............................................................................ 43
    2.7.6.2 Ionic strength ........................................................... 44
2.8 CHARACTERIZATION OF EMULSION PROPERTIES .................... 44
  2.8.1 Light scattering ................................................................. 44
  2.8.2 Particle size and size distribution ...................................... 45
  2.8.3 Zeta potential ................................................................. 46
  2.8.4 Birefringence ..................................................................... 47
  2.8.5 Electrical conductivity ...................................................... 48
  2.8.6 Viscosity .......................................................................... 49
  2.8.7 Turbidity .......................................................................... 50
2.9 CONCLUSIONS ............................................................................. 51

CHAPTER 3 FORMATION AND CHARACTERISATION OF TRANSPARENT
MICROEMULSIONS BY PHASE INVERSION METHOD ....................... 54

3.1 INTRODUCTION ........................................................................ 54
3.2 MATERIALS AND METHODS .................................................... 56
4.3.1 Dilution of stock emulsion in 1% Tween surfactant solution .................. 82
4.3.2 Particle size of secondary emulsions with time after preparation .......... 86
4.3.3 Effect of surfactant type on droplet solubilisation ................................. 90
4.3.4 Effect of concentration of surfactant micelle solutions on droplet
solubilisation ........................................................................................................... 97
4.3.5 Effect of oil type on droplet solubilisation .............................................. 101
4.3.6 Effect of some environmental factors on droplet solubilisation ............ 107
  4.3.6.1 Influence of pH ..................................................................................... 107
  4.3.6.2 Influence of ionic strength .................................................................... 110
  4.3.6.3 Influence of thermal treatment ............................................................... 112
4.3.7 Phase diagram ............................................................................................. 115
4.4 CONCLUSIONS ............................................................................................ 116

CHAPTER 5 OVERALL CONCLUSIONS AND RECOMMENDATIONS ....... 118
CHAPTER 6 REFERENCES .................................................................................... 120
APPENDICES .......................................................................................................... 133
List of Figures

Figure 2.1 Two types of emulsions, oil-in-water emulsion and water-in-oil emulsion.... 4
Figure 2.2 Chemical structures of some major compounds found in lemon oils.......... 10
Figure 2.3 Chemical structure of isopropyl myristate............................................ 11
Figure 2.4 Chemical structure of tributyrin............................................................. 11
Figure 2.5 Chemical structures of glyceryl caprylate (Imwitor 308)....................... 12
Figure 2.6 Chemical structure of (A) retinol (vitamin A) and (B) β-carotene............. 13
Figure 2.7 A schematic structure of small molecule surfactants............................ 14
Figure 2.8 Some typical structures formed due to the self-association of surfactant molecules.............................................................. 15
Figure 2.9 Chemical structures of small molecule surfactants (Tween 20, 40, 60 and 80). .......................................................... 17
Figure 2.10 (A) A bench top scale of APV-2000 two-stage high pressure homogenizer and (B) schematic illustration of a two-stage high pressure homogenizer used to produce emulsions.......................................................................................... 23
Figure 2.11 (A) Microfluidics-M-110EH microfluidizer (Microfluidics 2013) and (B) schematic illustration of microfluidizer used to produce emulsions................................. 24
Figure 2.12 (A) QSONICA-Q125 sonicator (Qsonica 2013) and (B) schematic illustration of ultrasonic probe homogenizer used to produce emulsions........................ 25
Figure 2.13 Schematic illustration of the process of a combined method of homogenization and solvent evaporation to form nanoemulsions............................................. 26
Figure 2.14 Effect of changing CCP caused by temperature changes on the phase inversion of a system containing small molecule surfactant. γ is the partial molar volume of the hydrophobic tail of surfactant, a is the head group area of surfactant and l is the hydrophobic chain length of surfactant tail group............................................. 28
Figure 2.15 A schematic representation of an experiment where emulsion droplets are titrated into a surfactant micelle solution to produce microemulsion..................... 31
Figure 2.16 Two types of an o/w microemulsion system............................................. 32
Figure 2.17 Schematic diagram of the free energy of nanoemulsion and microemulsion systems compared to the phase separated state (McClements 2012)....................... 33
Figure 2.18 Ternary phase diagram of an oil-surfactant-water system (Cannon 2011). 34
Figure 2.19 Schematic diagram illustrating the distribution of ions around charged emulsion droplets. .................................................................................................................. 36
Figure 2.20 Adsorption of emulsifiers (small molecule surfactants, flexible biopolymers and globular biopolymers) at the surface of oil droplet............................................ 37
Figure 2.21 Schematic illustrations of different types of emulsion destabilization..... 38
Figure 2.22 A schematic diagram of the electrical charge (zeta potential) of droplets in emulsion measured in mV at the slipping plane.................................................. 47

Figure 3.1 Ternary phase diagram composed of lemon oil, surfactant (Tween 20 or 80) and water................................................................. 57
Figure 3.2 Phase diagrams created for ternary systems formulated with different levels of lemon oil, water and Tween 20 (A) or Tween 80 (B). Abbreviations: ME represents microemulsion, CE conventional emulsion, NE nanoemulsion and LC liquid crystals. 60
Figure 3.3 Schematic representation of phase inversion from w/o to o/w microemulsion through bicontinuous structure formation (e.g. liquid crystalline lamellar phase). ...........62

Figure 3.4 Visual appearance of samples derived from dilution lines 1, 2 and 3 (1:9, 2:8 and 3:7 ratio of oil to surfactant) of the phase diagram for lemon oil/Tween 20/water ternary systems. Numbers from 0 to 90 labelled on vial caps indicate water weight fraction (% w/w); ME represents microemulsion; CE coarse emulsion; NE nanoemulsion and PS phase separation including creaming. The pictures shown in D, E and F indicate some differences in the rheological properties (e.g. viscosity and gelation) between samples. A pattern used in the photo background was to show and compare the degree of clarity and opacity between samples. .........................................................64

Figure 3.5 Visual appearance of samples from dilution lines 1, 2 and 3 (1:9, 2:8 and 3:7 ratio of oil to surfactant) of the phase diagram for lemon oil/Tween 80/water ternary systems. Numbers from 0 to 90 labelled on vial caps indicate water weight fraction (% w/w). Abbreviations: ME represents microemulsion, CE coarse emulsion, LC liquid crystals and PS phase separation including creaming. The pictures shown in D, E and F indicate some differences in the rheological properties (e.g., viscosity and gelation) between samples........................................................................................................65

Figure 3.6 The microscopic appearance of liquid crystals, represented by birefringence, found in some samples (L120 and L130 of dilution line 1) of a ternary system of lemon oil, Tween 80 and water. The image was taken at 20 × magnification under a polarized light microscopy. ........................................................................................................................................68

Figure 3.7 Particle size distributions of microemulsions prepared from ternary systems of lemon oil, surfactant (Tween 20 or Tween 80) and water........................................................................................................70

Figure 3.8 Electrical conductivity versus water weight fraction along (A) the dilution line 1 of lemon oil-Tween 20-water system and (B) the dilution lines 1 and 2 of lemon oil-Tween 80-water system. ........................................................................................................................................72

Figure 3.9 Viscosity of samples along the dilution line 1 for the lemon oil-Tween 80-water system measured at 25°C. Samples: (A) Newtonian fluid and (B) non-Newtonian fluid. ........................................................................................................................................74

Figure 3.10 Variation in dynamic viscosity as function of water content along the dilution line 1 for the lemon oil-Tween 80-water system at 25°C. The values of viscosity were taken at a shear rate of 12 s⁻¹. ................................................................................................................75

Figure 4.1 Particle size distributions of mixed emulsion-surfactant solutions (1% w/w Tween 80) with different IPM oil concentrations after overnight storage at ambient temperature........................................................................................................................................84

Figure 4.2 Appearance of secondary emulsions (1% w/w Tween 80) with increasing IPM concentration and stock emulsion (1% w/w Tween 80 and 10% IPM). Pictures were taken after one day storage at 20°C. Numbers refers to IPM concentration. .................84

Figure 4.3 Influence of oil (IPM) concentration on the mean particle diameters of mixed stock emulsion-surfactant solutions (i.e. secondary emulsions with 1% Tween 80) after overnight storage at ambient temperature..........................................................86

Figure 4.4 Visual appearance of secondary emulsions (1% w/w Tween 80) with increasing IPM concentration and stock emulsion (1% w/w Tween 80 and 10% IPM). Pictures were taken before (A) and after 1 day (B) storage at 20°C. Numbers refers to IPM weight fraction. ........................................................................................................87

Figure 4.5 Changes in the mean particle diameters (A and B) and turbidity of secondary
emulsions (C) over time during 24 hrs of storage at 20°C.

**Figure 4.6** Influence of surfactant type and oil concentration on the mean particle diameter of secondary emulsions prepared by titrating an emulsion (10% w/w IPM, 1% w/w surfactant) into a 1% w/w surfactant solution (Tween 20, 40, 60, and 80).

**Figure 4.7** Influence of surfactant type and oil concentration on the mean particle diameter of secondary emulsions prepared by titrating an stock emulsion (10% w/w IPM, 1% w/w Tween 20, 40, 60, or 80) into the same surfactant solution (1% w/w surfactant).

**Figure 4.8** Influence of surfactant type and oil concentration on the particle size distributions of secondary emulsions prepared by titrating stock emulsions produced by 10% w/w IPM and 1% w/w surfactant into 1% w/w same surfactant solutions. (A) Tween 20, (B) Tween 40, (C) Tween 60 and (D) Tween 80.

**Figure 4.9** Visual appearance of secondary emulsions prepared by titrating the stock emulsion produced by 10% w/w IPM and 1% w/w surfactant into 1% w/w surfactant solutions. (A) Tween 20, (B) Tween 40, (C) Tween 60, and (D) Tween 80. Numbers refer to IPM oil weight fraction (wt%). These pictures were taken after 1 day storage at 20°C.

**Figure 4.10** Physical appearance of secondary emulsions prepared by titrating an emulsion (10% w/w IPM, 1% w/w Tween 80) into four different concentrations of surfactant solutions: (A) 0% w/w Tween 80 (water); (B) 0.5% w/w Tween 80; (C) 1% w/w Tween 80; (D) 2% w/w Tween 80. Numbers refer to IPM weight fraction (wt%). The pictures were taken after overnight storage at 20°C.

**Figure 4.11** Influence of surfactant micelle and oil concentrations on the turbidity of secondary emulsions prepared by titrating an emulsion (10% w/w IPM, 1% w/w Tween 80) into four different concentrations of surfactant solutions (0%, 0.5%, 1%, and 2% w/w Tween 80). The data was obtained by measuring absorbance at 600 nm after overnight storage at ambient temperature.

**Figure 4.12** Influence of surfactant micelle solution and oil concentration on the mean particle diameter of secondary emulsions prepared by titrating an emulsion (10% IPM and 1% Tween 80) into four different concentrations of surfactant solutions (0%, 0.5%, 1%, and 2% Tween 80). The samples were measured after overnight storage at ambient temperature.

**Figure 4.13** Influence of oil type on the particle size distributions of the initial stock emulsions produced from 10% w/w oil and 1% w/w Tween 80.

**Figure 4.14** Physical appearances of secondary emulsions prepared by titrating an emulsion (10% w/w oil, 1% w/w Tween 80) into 1% w/w Tween 80 surfactant solutions (A) Sunflower oil; (B) Tributyrin; (C) Lemon oil; (D) Imwitor 308. Numbers refers to oil weight fraction (wt%) in the secondary emulsions. The pictures were taken before (1) and after storage overnight (2) at 20°C.

**Figure 4.15** Influence of oil type and concentration on the turbidity of secondary emulsions prepared by titrating a stock emulsion (10% oil and 1% Tween 80) into 1% Tween 80 surfactant solution. The turbidity was measured at 600 nm after overnight storage at ambient temperature.

**Figure 4.16** Influence of oil type and concentration on the mean particle diameter of secondary emulsions prepared by titrating a stock emulsion (10% oil and 1% Tween 80) into 1% Tween 80 surfactant solution. The measurement was done after overnight storage at ambient temperature.

**Figure 4.17** Physical appearances of secondary emulsions (1% w/w Tween 80) with
different pH value. (A) 0.05% w/w IPM; (B) 0.15% w/w IPM; (C) 0.5% w/w IPM. Numbers refer to pH values of the secondary emulsions. The pictures were taken after overnight storage at ambient temperature.

Figure 4.18 Effect of pH on the mean particle diameter of secondary emulsions (0.05%, 0.15% and 0.5% w/w IPM: 1% w/w Tween 80).

Figure 4.19 Effect of pH on the particle size distributions of secondary emulsions (1% w/w Tween 80) (A) 0.05% w/w IPM; (B) 0.15% w/w IPM; (C) 0.5% w/w IPM.

Figure 4.20 Effect of salt (NaCl) concentration on the mean particle diameter of secondary emulsions (0.025%, 0.075% and 0.25% w/w IPM; 1% w/w Tween 80). 

Figure 4.21 Physical appearances and particle size distributions of secondary emulsions (1% Tween 80) containing different concentration of salt.(A) 0.025% IPM; (B) 0.075% IPM; (C) 0.25% IPM. The size and pictures were taken after overnight storage at 20°C.

Figure 4.22 Physical appearances of 1% w/w Tween 80 secondary emulsions or stock emulsions at 20 °C (blank), after heating at 50 °C, and after heating at 80 °C (A) 0.05% w/w IPM; (B) 0.15%; (C) 0.5% w/w IPM; (D) 10% w/w IPM (stock emulsions). Numbers refer to the storage day of emulsions.

Figure 4.23 Effect of thermal processing on the mean particle diameter (Z-Average) of 1% w/w Tween 80 secondary emulsions or stock emulsions at 20 °C (blank), after heating at 50 °C, and after heating at 80 °C (A) 0.05% w/w IPM; (B) 0.15%; (C) 0.5% w/w IPM; (D) 10% w/w IPM (stock emulsions).

Figure 4.24 Schematic diagram of the temperature influence on emulsion properties (McClements & Rao 2011).

Figure 4.25 Ternary phase diagram based on the dilution process from point A (stock lemon oil emulsion) to point B by titration of conventional Tween 80-stabilised stock emulsion into 1 wt% Tween 80 micelle solution. CE (coarse emulsion, 10% lemon oil and 1% Tween 80); ME (microemulsion, 0.05% lemon oil and 1% Tween 80).
List of Tables

Table 2.1 Some physicochemical properties of sunflower oil, lemon oil, isopropyl myristate (IPM), tributyrin and Imwitor 308 and their comparison with water...............8

Table 2.2 Some chemical and physical properties of Tween 20, 40, 60 and 80a............17

Table 3.1 Composition and types of phase behaviour of ternary systems composed of lemon oil, surfactant (Tween 20 or Tween 80) and water from dilution lines 1, 2 and 3 of ternary phase diagrams. A few samples from dilution lines 4 and 5 that formed w/o microemulsions are also included.................................................................66

Table 3.2 Average particle size in diameter of selected microemulsions from ternary systems of lemon oil, surfactant (Tween 20 or Tween 80) and water.........................69

Table 4.1 Mean particle diameter (Z-Average) and polydispersity index (PdI) of surfactant solution (1% Tween 80), stock emulsion (10% IPM and 1% Tween 80) and secondary emulsions (different concentrations of IPM and 1% Tween 80) measured after storage for 1 day at 20\(^{\circ}\)C.................................................................83

Table 4.2 Mean particle size of stock emulsions, expressed as Z-average in diameter (d.nm), that were prepared with 10% (w/w) IPM and 1% (w/w) Tween surfactants by using a two-stage high pressure homogenizer at 500/50 bar (first/ second stage pressure)........................................................................................................92

Table 4.3 The particle size of initial stock emulsions (10% w/w oil and 1% w/w Tween 80) which were used to prepare the secondary emulsions......................................102
List of Appendices

**Appendix 1** Real composition corresponding to Line 1 (1 : 9 dilution line) of Lemon oil/Tween 20/Water systems.................................................................134

**Appendix 2** Visual appearance of Line 1 (1 : 9 dilution line) of Lemon oil/Tween 20/Water system.................................................................134

**Appendix 3** Real composition corresponding to Line 2 (2 : 8 dilution line) of Lemon oil/Tween 20/Water systems.................................................................135

**Appendix 4** Visual appearance of Line 2 (2 : 8 dilution line) of Lemon oil/Tween 20/Water system.................................................................135

**Appendix 5** Real composition corresponding to Line 3 (3 : 7 dilution line) of Lemon oil/Tween 20/Water systems.................................................................136

**Appendix 6** Visual appearance of Line 3 (3 : 7 dilution line) of Lemon oil/Tween 20/Water system.................................................................136

**Appendix 7** Real composition corresponding to Line 4 (4 : 6 dilution line) of Lemon oil/Tween 20/Water systems.................................................................137

**Appendix 8** Visual appearance of Line 4 (4 : 6 dilution line) of Lemon oil/Tween 20/Water system.................................................................137

**Appendix 9** Real composition corresponding to Line 5 (5 : 5 dilution line) of Lemon oil/Tween 20/Water systems.................................................................138

**Appendix 10** Visual appearance of Line 5 (5 : 5 dilution line) of Lemon oil/Tween 20/Water system.................................................................138

**Appendix 11** Real composition corresponding to Line 6 (6 : 4 dilution line) of Lemon oil/Tween 20/Water systems.................................................................139

**Appendix 12** Visual appearance of Line 6 (6 : 4 dilution line) of Lemon oil/Tween 20/Water system.................................................................139

**Appendix 13** Real composition corresponding to Line 7 (7 : 3 dilution line) of Lemon oil/Tween 20/Water systems.................................................................140

**Appendix 14** Visual appearance of Line 7 (7 : 3 dilution line) of Lemon oil/Tween 20/Water system.................................................................140

**Appendix 15** Real composition corresponding to Line 8 (8 : 2 dilution line) of Lemon oil/Tween 20/Water systems.................................................................141

**Appendix 16** Visual appearance of Line 8 (8 : 2 dilution line) of Lemon oil/Tween 20/Water system.................................................................141

**Appendix 17** Real composition corresponding to Line 9 (9 : 1 dilution line) of Lemon oil/Tween 20/Water systems.................................................................142

**Appendix 18** Visual appearance of Line 9 (9 : 1 dilution line) of Lemon oil/Tween 20/Water system.................................................................142

**Appendix 19** Real composition corresponding to Line 10 (Oil free) of Lemon oil/Tween 20/Water systems.................................................................143

**Appendix 20** Visual appearance of Line 10 (Oil free) of Lemon oil/Tween 20/Water system.................................................................143

**Appendix 21** Real composition corresponding to Line 11 (surfactant free) of Lemon oil/Tween 20/Water systems.................................................................144

**Appendix 22** Visual appearance of Line 11 (surfactant free) of Lemon oil/Tween 20/Water system.................................................................144
Appendix 23 Real composition corresponding to Line 1 (1 : 9 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 145
Appendix 24 Visual appearance of Line 1 (1 : 9 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 145
Appendix 25 Real composition corresponding to Line 2 (2 : 8 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 146
Appendix 26 Visual appearance of Line 2 (2 : 8 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 146
Appendix 27 Real composition corresponding to Line 3 (3 : 7 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 147
Appendix 28 Visual appearance of Line 3 (3 : 7 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 147
Appendix 29 Real composition corresponding to Line 4 (4 : 6 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 148
Appendix 30 Visual appearance of Line 4 (4 : 6 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 148
Appendix 31 Real composition corresponding to Line 5 (5 : 5 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 149
Appendix 32 Visual appearance of Line 5 (5 : 5 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 149
Appendix 33 Real composition corresponding to Line 6 (6 : 4 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 150
Appendix 34 Visual appearance of Line 6 (6 : 4 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 150
Appendix 35 Real composition corresponding to Line 7 (7 : 3 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 151
Appendix 36 Visual appearance of Line 7 (7 : 3 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 151
Appendix 37 Real composition corresponding to Line 8 (8 : 2 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 152
Appendix 38 Visual appearance of Line 8 (8 : 2 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 152
Appendix 39 Real composition corresponding to Line 9 (9 : 1 dilution line) of Lemon oil/Tween 80/Water systems ................................................................. 153
Appendix 40 Visual appearance of Line 9 (9 : 1 dilution line) of Lemon oil/Tween 80/Water system ................................................................. 153
Appendix 41 Real composition corresponding to Line 10 (oil free) of Lemon oil/Tween 80/Water systems ................................................................. 154
Appendix 42 Visual appearance of Line 10 (oil free) of Lemon oil/Tween 80/Water system ................................................................. 154
Appendix 43 Real composition corresponding to Line 1 (1 : 9 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems ................................................................. 155
Appendix 44 Visual appearance of Line 1 (1 : 9 dilution line) of Lemon oil/Tween 80/Ethanol/Water system ................................................................. 155
Appendix 45 Real composition corresponding to Line 2 (2 : 8 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems ................................................................. 155
Appendix 46 Visual appearance of Line 2 (2 : 8 dilution line) of Lemon oil/Tween 80/Ethanol/Water system ................................................................. 155
oil/Tween 80/Ethanol/Water systems..........................156

**Appendix 46** Visual appearance of Line 2 (2 : 8 dilution line) of Lemon oil/Tween 80/Ethanol/Water system. .................................................................156

**Appendix 47** Real composition corresponding to Line 3 (3 : 7 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................157

**Appendix 48** Visual appearance of Line 3 (3 : 7 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................157

**Appendix 49** Real composition corresponding to Line 4 (4 : 6 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................158

**Appendix 50** Visual appearance of Line 4 (4 : 6 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................158

**Appendix 51** Real composition corresponding to Line 5 (5 : 5 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................159

**Appendix 52** Visual appearance of Line 5 (5 : 5 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................159

**Appendix 53** Real composition corresponding to Line 6 (6 : 4 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................160

**Appendix 54** Visual appearance of Line 6 (6 : 4 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................160

**Appendix 55** Real composition corresponding to Line 7 (7 : 3 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................161

**Appendix 56** Visual appearance of Line 7 (7 : 3 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................161

**Appendix 57** Real composition corresponding to Line 8 (8 : 2 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................162

**Appendix 58** Visual appearance of Line 8 (8 : 2 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................162

**Appendix 59** Real composition corresponding to Line 9 (9 : 1 dilution line) of Lemon oil/Tween 80/Ethanol/Water systems........................................163

**Appendix 60** Visual appearance of Line 9 (9 : 1 dilution line) of Lemon oil/Tween 80/Ethanol/Water system.........................................................163

**Appendix 61** Real composition corresponding to Line 10 (oil free) of Lemon oil/Tween 80/Ethanol/Water systems.......................................................164

**Appendix 62** Visual appearance of Line 10 (oil free) of Lemon oil/Tween 80/Ethanol/Water system.................................................................164