

2014 NIMS Hot Topics Workshop

British Council Researchers Links Workshop on Soft Matter:
Analysis, Applications and Challenges
funded by British Council and NIMS

NIMS, Daejeon, South Korea
March 18 ~ March 21, 2014

Organizers

- Apala Majumdar, University of Bath, UK
- Jinhae Park, Chungnam National University

Mentors

- Hyeonbae Kang, Inha University
- Yong-Jung Kim, KAIST
- Ping Lin, University of Dundee, UK
- Tim Sluckin, University of Southampton, UK

Invited Speakers

- Gareth Alexander, University of Warwick, UK
- **Deski Beri, Universitas Negeri Padang, Indonesia**
- John Biggins, University of Cambridge, UK
- Oleksandr Buchnev, University of Southampton, UK
- Flynn Castles, University of Oxford, UK
- Sunho Choi, KAIST
- Linsey Corson, University of Strathclyde, UK
- Yunkyong Hyon, NIMS
- Narina Jung, UNIST
- Hyeonbae Kang, Inha University
- Junseok Kim, Korea University
- Yangjin Kim, Konkuk University
- Yong-Jung Kim, KAIST
- Yong-Woon Kim, KAIST
- Ping Lin, University of Dundee, UK
- Halim Kusumaatmaja, Durham University, UK
- Apala Majumdar, University of Bath, UK
- Yogesh Murugesan, University of Southampton, UK
- Yasumasa Nishiura, Tohoku University, Japan
- Ben Outram, University of Oxford, UK
- Xingbin Pan, East China Normal University, China
- Jinhae Park, Chungnam National University
- Wei Wang, Peking University, China
- Tim Sluckin, University of Southampton, UK
- Tim Spencer, Sheffield Hallam University, UK
- Yoshimi Tanaka, Yokohama National University, Japan
- Sumesh Thampi, University of Oxford, UK
- Adriano Tiribocchi, University of Edinburgh, UK
- Christophe Trabi, Nottingham Trent University, UK
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For more information, please check <http://open.nims.re.kr/events/SoftMatter>



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March. 17, 2014

Name: Professor Deski Beri

Affiliation: Chemistry Department, Universitas Negeri Padang, Indonesia

I hereby declare that British-Korea Research Link Workshop on Soft Matter's board have reviewed your article entitled "Phase Dynamics and Solubility of Dyes in Microemulsions and Liquid Crystals of Water, CTAB and Penthanol Systems" and it gives me great pleasure to invite you to give a scientific talk in "NIMS Hot Topics Workshop on British-Korea Research Link Workshop on Soft Matter: Analysis, Applications and Challenges" to be held at NIMS, Daejeon, Korea during March 18~21, 2014.

This visit provides the delegates an opportunity to contribute their expertise as well as interact with other scholars. I am sure that your participation will enhance the quality of the conference and you and other participants will benefit from the conference immensely.

NIMS is a government institution, aims to promote research activities in pure and applied mathematics, serving as a research hub inter linking mathematics, sciences, engineering, technology and industry. The institute has a long vision of research in mathematical sciences providing original theories to be utilized for future technology.

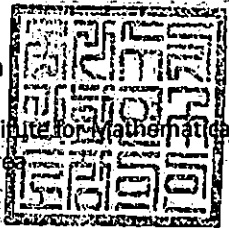
International flight, local expenses will be supported by British Council.

If you have any questions, please do not hesitate to contact us.

Looking forward to seeing you

Sincerely,

Dongsu Kim
Director
National Institute for Mathematical Sciences
Daejeon, Korea



Phase Dynamics and Solubility of Dyes in Microemulsions and Liquid Crystals of Water, CTAB and Pentanol Systems

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Deski Beri^a, Angla Pratami^a and Ali Amran^a,

Interaction between dyes and surfactant compound is an interesting research field due to dyes can be used for probing the microstructure of colloidal association structures. In this study, we figured out the area formation of microemulsions and liquid crystals of water, CTAB and pentanol systems at pH = 4.5 and pH = 9.5. The results showed that there is a phase transition occurs in the area of the hexagonal liquid crystal with increasing pH. Dyes solubility (methyl red in the systems of pH=4.5 and methylene blue in the systems of pH=9.5) indicated that solubility of dyes will be higher when the number of pentanol increased both in acidic and alkaline solution. Particularly for methyl red, lamellar liquid crystal phase was the best medium (0.0036 g w/w solutions) followed by w/o microemulsions (0.0012 g w/w solutions) and o/w microemulsion (0.0006 g w/w solutions), respectively whereas for methylene blue w/o microemulsions was the best solvent (0.0009 g w/w solutions), followed by lamellar liquid crystals (0.0006 g w/w solutions) and o/w microemulsions (0.0005 g w/w solutions), respectively. Dyes solubility in hexagonal liquid crystal phase was not investigated due to high viscosity. Refractive index and viscosity measurement showed that the refractive index tended to be high with increasing number of pentanol and surfactant proportional with the addition of dyes. Finally, evaporation rate measurement showed that all association structure evaporated in pseudo first order reaction.

1 Introduction

Some non-interacting liquids which is immiscible each other (i.e. water and oil) could be treated as a macroscopically homogeneous phase (i.e. microemulsions and liquid crystals) by addition of some surfactants and it's mixtures^{1,2,3}. Microemulsions and liquid crystals research attracted much interest due to the wide application of the materials in research, industries and technology, and both amphiphilic association structures have some unique properties in the wide interface area, thermodynamical stability, and the ability to dilute immiscible liquids^{4,5}. Therefore, i). microemulsions has wide application such as; in oil recovery, fuel distilled, textile immersion and finishing, detergency, cosmetics, agriculture, food chemistry, pharmacy, environmental restoration, detoxification, microporous materials, analytical chemistry, liquid membranes, biotechnology and dyes industry⁶. Whereas ii). Liquid crystals has a wide application in cosmetics, pharmaceutical industry, pharmaceutical delivery, manufacturing industry, electronics, liquid crystal display, nanoparticles, dyes and dispersed polymeric liquid crystals film, etc^{7,8,9}.

Our research focused on determining the stability of some dyes in the microemulsion's and liquid crystal's phases¹⁰. The chemical and physical properties of association structure in water (pH=4,5 and pH=9,5), surfactants (anionic=SDS, cationic=CTAB and non-ionic=Brij-35) and cosurfactant (pentanol) systems¹¹. Our research was conducted in three steps; there were: i) preparation of phase diagram of water (pH=4,5 and pH=9,5), surfactants (anionic=SDS, cationic=CTAB and non-ionic=Brij-35) and cosurfactant (pentanol) systems, than ii) determine the solubility of methyl red in o/w and w/o microemulsions and lamellar liquid crystals from previous determined systems of water (pH=4,5), surfactants (anionic=SDS, cationic=CTAB and non-ionic=Brij-35) and cosurfactant (pentanol) and solubility of methylene blue in o/w and w/o microemulsions and lamellar liquid crystals from previous determined systems of water (pH=9,5), surfactants (anionic=SDS, cationic=CTAB and non-ionic=Brij-35) and cosurfactant (pentanol)¹². Third, iii) determine the refractive indexes, viscosity and evaporation rate of association structure in water (pH=4,5 and pH=9,5), surfactants (anionic=SDS, cationic=CTAB and non-ionic=Brij-35) and cosurfactant (pentanol) systems¹³.

2 Experimental and Methods

A. Materials and Methods

In this research we used cationic surfactant CTAB (cetyl trimethyl ammonium bromide) GR, nitric acid fuming, potassium hydroxide GR and n-pentanol, methylene blue were purchased from Merck KgaA, Germany, methyl red was purchased from Wako Pure Chemical Industries Ltd. Double distilled water was purchased from Rafi Medika. Double distilled water were used in this experiment; water pH=4.5 was prepared by addition of nitric acid 0.1M gradually dropped into stirred double distilled water, which simultaneously measured by Lab 850 BENCHTOP pH METER. Whereas to prepare water pH=9.5, KOH = 0.1 M were used similarly. Freshly liquids were used to prepare phase diagram.

B. Phase Diagram Preparation

Phase area of microemulsions, both o/w and w/o microemulsions and liquid crystals (lamellar and hexagonal) from water (pH=4.5 and 9.5), CTAB and pentanol system was determined by water titration method at ambient temperature. Phase diagram were plotted with varying water/cosurfactant/surfactant (w/c/s) ratios. The total component mass was kept 0.5 g in various w/c/s system ratio. Started from water excess systems, determination steps was developed to oil and surfactant excess. The procedure was by weight all three components in certain composition than homogeneously mixed by using thermolyne® mixer for 3-5 minutes. Than phase formation was observed by using visuals, parafilm and Hund-Wetzlar® optical polarizing microscope. The observation result than plotted as a mark in the diagram. Finally, after carefully phase boundary determined, than all phase formation point was ajoin to form phase area or phase island.

C. Solubility of Methyl Red and Methylene Blue in Microemulsions and Liquid Crystals

Solubility of methyl red was determined for o/w and w/o microemulsions and lamellar liquid crystals for water (pH=4.5), CTAB) and pentanol system, whereas solubility of methylene blue was determined for o/w and w/o microemulsions and lamellar liquid crystals for water (pH=9.5), CTAB and pentanol system. Because of technical problems, solubility of methyl red and methylene blue in the hexagonal liquid crystal were not determined. The procedure is small quantities of methyl red/methylene blue were added gradually in to a tube which was filled by given composition phase of interest. The addition was observed visually, and red laser for methyl red and blue laser for methylene blue addition was used to show the solubility in visual. The addition was stopped when the precipitated of dyes were formed.

D. Refractive Index Measurement

Temperature control ABBE Refractometer 2WAJ from PCE Instruments UK, Ltd., was used to determined refractive index area of interest. Every blank phase and addition of methyl red phase of interest was observed by refractometer. *Procedure:* a small liquid of interest from o/w and w/o microemulsions and lamellar liquid crystals was treated in prism and than observed in temperature control and double distilled water calibrated instruments. Than the similar composition of interest with addition of methyl red was

treated in similar procedures, the observed refractive index was record and than analysed.

E. Viscosity Measurement

Schott-Geräte 509 03 Oswald viscosimeter was used to measure kinematic viscosity area of interest. Every blank phase and addition of methyl red and methylene blue phase of interest was observed. *Procedure:* a small (3 mL) liquid of interest from o/w and w/o microemulsions was treated in viscosimeter and than time of liquid to pass through the certain length were observed in temperature control and double distilled water calibrated instruments. Than the similar composition of interest with addition of methyl red was treated in similar procedures, the observed liquid diffusion through the certain length was record and computed by Poiseoule formula as:

$$v = \frac{\eta}{\rho} = \frac{\pi R^4 g h_m}{8LV} t$$

Since $\frac{\pi R^4 g h_m}{8LV} = K$, so viscosity could be simplified as $v = K.t$,

from instrument we get kinematic viscosity formula as $v = K(t - \vartheta)$

, where ϑ called as Hagenbach correction = $\vartheta = \frac{0.12}{K.t}$, and

$K = 0.004$. Using the formula presented above, we compute the kinematic viscosity.

F. Evaporation Rate of Microemulsions

Evaporation rate was determined by room temperature evaporation of phase of interest (o/w and w/o microemulsions and lamellar liquid crystals) with and without dyes addition. Daily weight reduction was recorded and analysed.

3 Result and Discussion

A. Phase Diagram of Water, CTAB and Pentanol

Determination of phase diagram for water (both in pH=4.5 and pH=9.5); CTAB and pentanol system resulted four main phase areas, i.e., o/w and w/o microemulsions, lamellar (LLC) and hexagonal liquid crystals (HLC) as shown in Fig.1 for water pH=4.5 and Fig.2 for water pH=9.5.

W/o microemulsions area for water pH=4.5; CTAB and pentanol system formed a wide microemulsion band around 0-63% water; 0-52% CTAB and 22-100% pentanol. O/w microemulsions in excess of water formed in area of 73-100% water; 0-23% CTAB and 0-12% pentanol. Lamellar liquid crystals (LLC) was formed in the area of 11-53% water; 25-69% CTAB and 6-33% pentanol. Hexagonal liquid crystals (HLC) was formed in area of 36-71% water; 25-69% CTAB and 6-33% pentanol, respectively. Completed picture of area formation could be seen in Fig.1.

W/o microemulsions area for water pH=9.5; CTAB and pentanol system formed a similar wide microemulsion area around

0-62% water; 0-51% CTAB and 21-100% pentanol. O/w microemulsions in excess of water formed in area of 65-100% water; 0-22% CTAB and 0-16% pentanol. Lamellar liquid crystals (LLC) was formed in area of 21-62%; 19-54% CTAB and 9-39% pentanol. Hexagonal liquid crystals (HLC) was formed in area of 9-39% water; 49-61% CTAB and 2-34% pentanol, respectively. Completed picture of formation area could be seen in Fig.2.

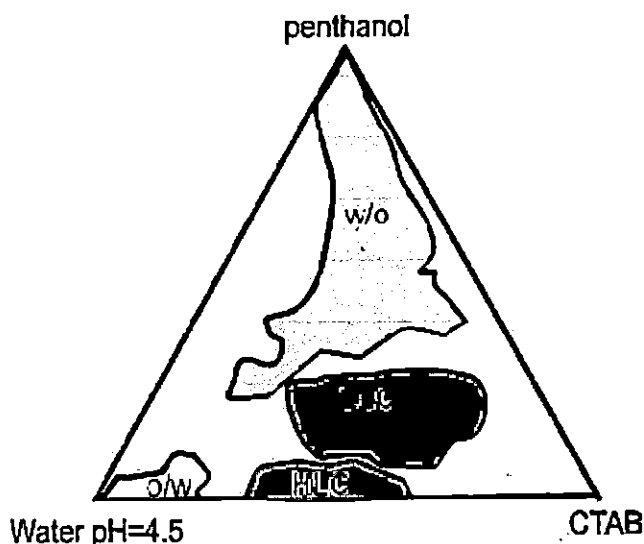


Fig. 1 Phase diagram of water pH=4.5, CTAB and pentanol system: every colour represent phase formation in the system

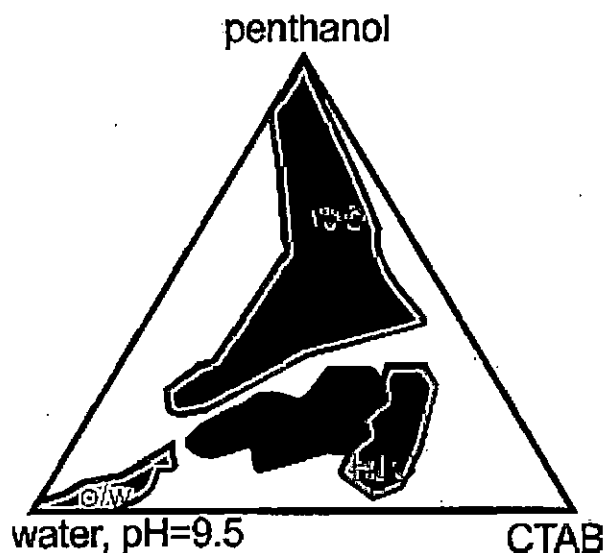


Fig. 2 Phase diagram of water pH=4.5, CTAB and pentanol system: every colour represent phase formation in the system

There was no significant difference between w/o and o/w microemulsions for phase areas in the system pH=4.5 and pH=9.5. However, for liquid crystals areas both lamellar(LLC) and hexagonal (HLC) were different. The LLC phase for water pH=9.5 system is thinner and shift to water excess area compared to LLC phase of water pH=4.5. Phase transition was take place to HLC, where increasing pH would change the position of formation area for hexagonal liquid crystals. The increasing pH means the increasing

number of hydroxyl ions (OH⁻) tends to dissolved surfactant (CTAB) molecules. This would minimizes tensions between water molecules and CTAB and therefore along with water and CTAB line, there is no liquid crystal formation in system of water pH=9.5. However, the existence/availability of pentanol molecules reducing the solubility due to the increasing carbon numbers. So that in the middle area overlapped to LLC, the HLC was formed again.

B. Solubility of methyl red and methylene blue in microemulsions and liquid crystals

Experiments showed that the solubility of methyl red and methylene blue were displayed in Table 1 and Table 2.

Table 1. Solubility Data for Methyl Red in Microemulsions and Liquid Crystal of Water pH=4.5, CTAB and Pentanol System

Phase Area	Average Solubility (gram)
O/W Microemulsions	0.0006
W/O Microemulsions	0.0012
Lamellar Liquid Crystal	0.0036

Data in Table 1 presented that the average solubility of methyl red in lamellar liquid crystal, w/o microemulsions and o/w microemulsions were 0.0036, 0.0012, and 0.0006 grams over 0.5 grams of solutions respectively.

Table 2. Solubility Data for Methylene Blue in Microemulsions and Liquid Crystal of Water pH=9.5, CTAB and Pentanol System

Phase Area	Average Solubility (gram)
O/W Microemulsions	0.0005
W/O Microemulsions	0.0009
Lamellar Liquid Crystal	0.0006

Whereas data in Table 2 presented the average solubility of methylene blue in w/o microemulsions, lamellar liquid crystal and o/w microemulsion were 0.0009, 0.0006, and 0.0005 grams over 0.5 grams of solutions respectively.

As shown in the tables the solubilisation of methyl red in lamellar liquid crystal was better compared to w/o and o/w microemulsions in system of water pH=4.5. But in system of water pH=9.5 the solubilisation of methylene blue in lamellar was less soluble compared to w/o microemulsions.

Hence methyl red and methylene blue solubilisation in the water; CTAB and pentanol systems was characteristics. The particular pH=4.5 and pH=9.5 were chosen due to the bright red of methyl red was characterized to pH range about 4.4-6.2, as shown in Fig.3 whereas bright blue colour from methylene blue were characterized in the basic solution.

CTAB structure in Fig. 5 has a long carbon chain with an ammonium head group represented as circle, whereas carbon chain represented as long tail group. This polar structure would arranged in water and oil medium. However pentanol structure in Fig. 7 could be considered as oil due to the semipolar properties. By using the illustrative model we proposed the solubility mechanism as well in Fig. 6 and Fig. 8.

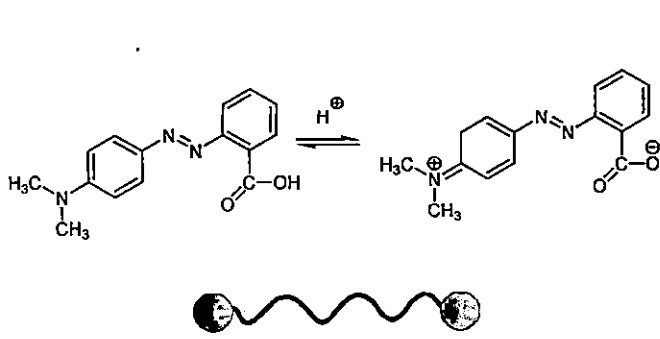


Fig. 3 Methyl red structure in acidic solution and the illustrative model

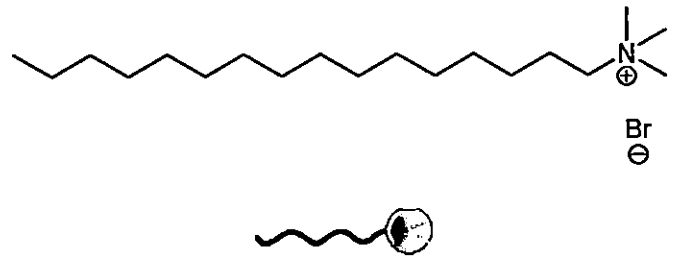


Fig. 5. Cetyltrimethyl ammonium bromide and the illustrative model

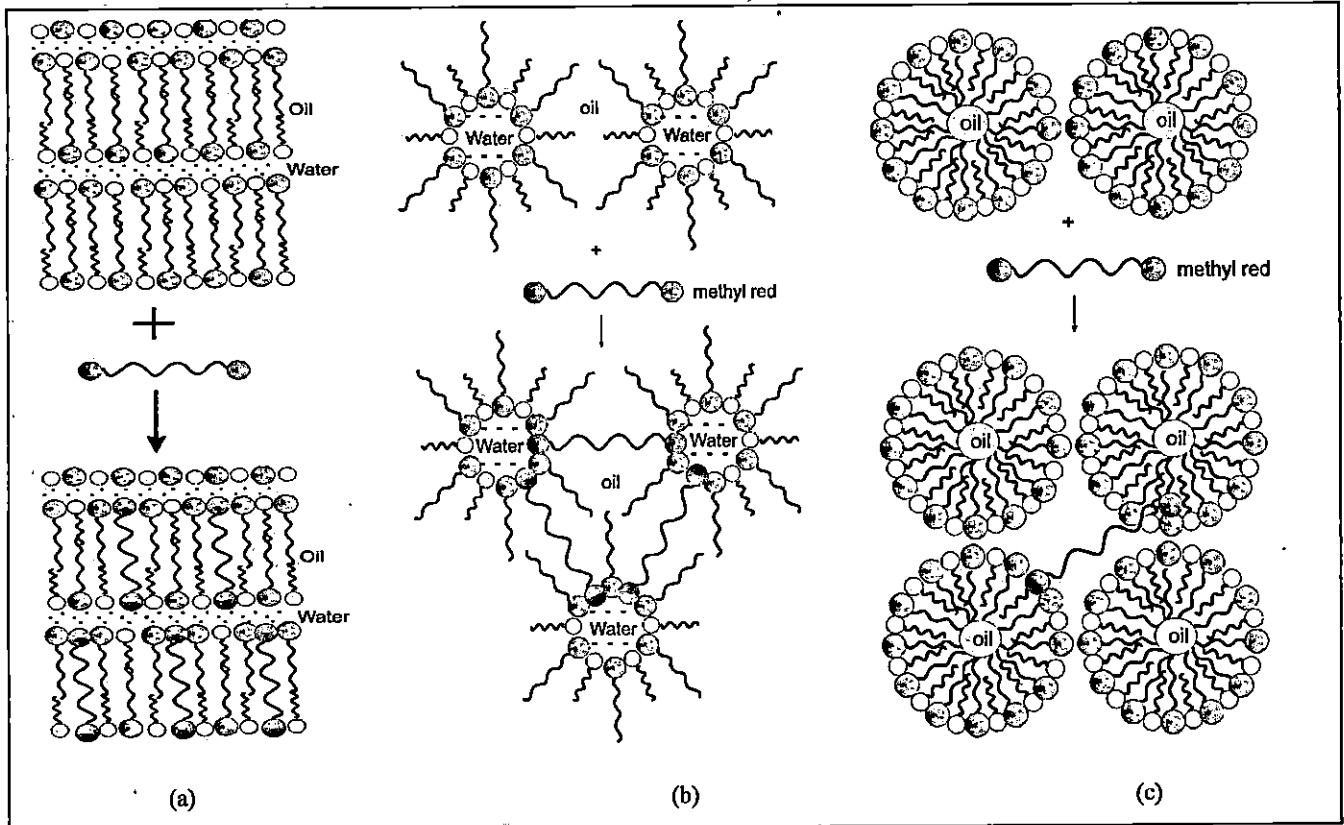


Fig. 6. Solubilization mechanisms of methyl red in (a) lamellar liquid crystal, (b) w/o microemulsion and (c) o/w microemulsion system water pH=4.5, CTAB and pentanol

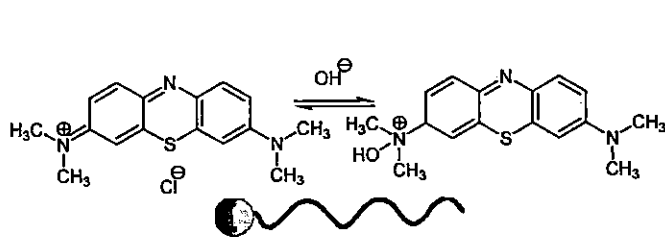


Fig. 4 Methylene blue structure in basic solutions and the illustrative model

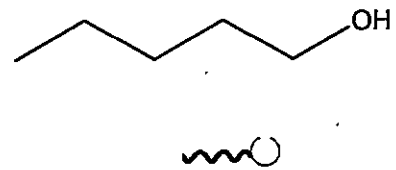


Fig. 7. N-pentanol as cosurfactant and the illustrative model

Methyl red structure in Fig.3 has a good bipolar structures, so it has good potentials junction in both end. This pre-assumption bring us to the thesis that methyl red structure will be soluble in the layer structures (Fig. 6a), however the less polar species in methyl ammonium $(CH_3)_2N^-$ group would be soluble in oily rich or w/o

microemulsions (Fig. 6b). However, the existence of $-COO^-$ group in methyl red structure force this structure to be miscible in the limited number of o/w microemulsions as shown in Fig. 6c.

In contrast, at methylene blue structure (Fig.4), we have both less potential junction at the end. The increasing pH would increase the polar properties of one group and it would increase the solubility of oily group or w/o microemulsions Fig. 8b. The methyl ammonium $(CH_3)_2N^+$ group with influence of basic group (OH^-) tended to form active species like a surfactant as well, so this properties would not significantly increase the solubility in o/w microemulsion Fig.8c and in layer structure at lamellar liquid crystals Fig. 8a.

As shown in Fig. 8 the solubility of methylene blue was less soluble in lamellar liquid crystal and o/w microemulsions due to the ability to minimized junction and also to capabilities to broke association structure since, the former association structure was

stable, but than it should be destabilized by the addition of methylene blue. However methylene blue need an extra force to reformed association structure as figured in Fig. 8c.

C. Refractive Index and Viscosity

Refractive index measurement for water pH=4.5, CTAB and pentanol system both for o/w and w/o microemulsions presented in Table 3. The datas was collected for w/o and o/w microemulsions

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{N}$$

and averages was computed by $\bar{x} = \frac{\sum_{i=1}^n x_i}{N}$. Data showed that

refractive index will be directly proportional to the the oil content. The high number of oil in the system, the refractive index would tended to rise. The addition of methyl red was less significant to increase the refractive index. It could be seen that the increase of methyl red would increase 0.13% for o/w microemulsion and 0.2%

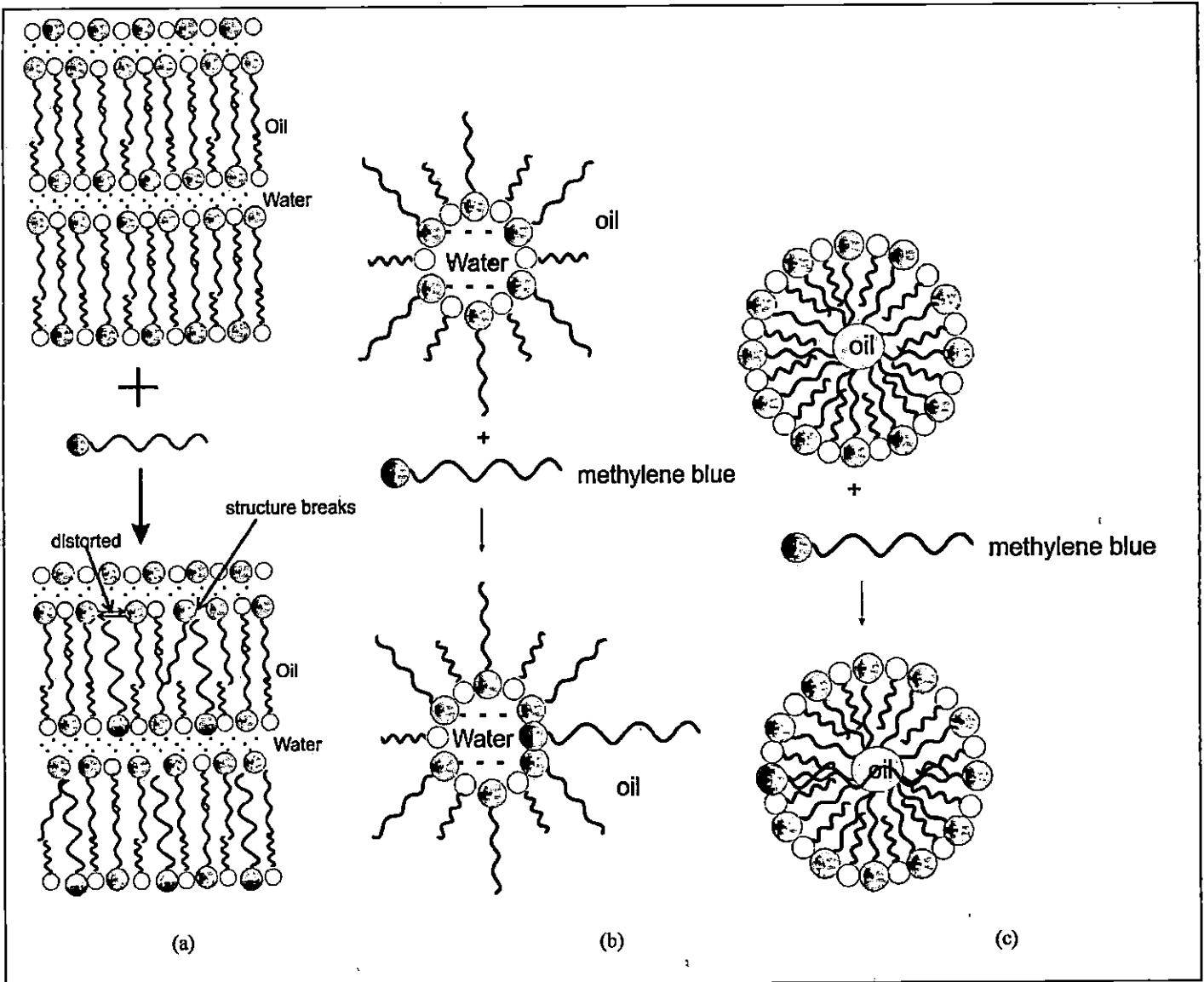


Fig. 8. Solubilization mechanisms of methyl red in (a) lamellar liquid crystal, (b) w/o microemulsion and (c) o/w microemulsion system water pH=4.5, CTAB and pentanol

for w/o microemulsion.

Table 3. Refractive index measurement with and without addition methyl red for microemulsions in water pH=4.5, CTAB and pentanol system.

No	Phase	Refractive index		Δ
		Without methyl red	With methyl red	
1	o/w microemulsion	1.352056	1.353361	0.0013
2	w/o microemulsion	1.410157	1.412243	0.002

Refractive index measurement for water pH=9.5, CTAB and pentanol was done without addition of methylene blue due to the limitation of instrument. Comparing data in Table 3 and Table 4 could be seen there was no significant different between acid and basic systems.

Table 4. Refractive index measurement with and without addition methylene blue for microemulsions in water pH=9.5, CTAB and pentanol system.

No	Phase	Refractive index		Δ
		Without methylene blue	With Methylene blue	
1	o/w microemulsion	1.349267	N/A	-
2	w/o microemulsion	1.404947	N/A	-

Viscosity measurement for o/w and w/o microemulsions showed datas as presented in Table 5 for water pH=4.5, CTAB and pentanol system and with the addition of methyl red and Table 6 for water pH=9.5, CTAB and pentanol system and the addition of methylene blue. It could be seen that the oil number would increase viscosity, the addition of dyes would increase viscosity both for acid and basic systems. Methyl red addition would increase 1.3% in average to viscosity in o/w microemulsion and 2.9% in w/o microemulsion. Whereas data in Table 6 presented the almost similar condition, where methylene blue addition would increase 1.3% in average to viscosity in o/w microemulsion and 1.4% in w/o microemulsion. Both datas in acid and basic condition was not significantly different in viscosity.

Table 5. Viscosity measurement with and without addition methyl red for microemulsions in water pH=4.5, CTAB and pentanol system.

No	Phase	Viscosity (mpa.s)		Δ
		Without methyl red	With methyl red	
1	o/w microemulsion	0.074857	0.087934	0.0131

2	w/o microemulsion	0.113656	0.142692	0.029
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Table 6. Refractive index measurement with and without addition methylene blue for microemulsions in water pH=9.5, CTAB and pentanol system.

No	Phase	Refractive index		Δ
		Without methylene blue	With Methylene blue	
1	o/w microemulsion	0.076019	0.089118	0.013099
2	w/o microemulsion	0.123479	0.137752	0.014273

D. Evaporation rate measurement

Evaporation rate measurement was done for two or three months and measurement was done everyday. Data was plotted as weight loss vs time in hours. Measurements was done for o/w and w/o microemulsions with and without dyes. Data analysis was done by first order kinetics as:

1) *o/w microemulsions in water pH=4.5, CTAB and pentanol system*

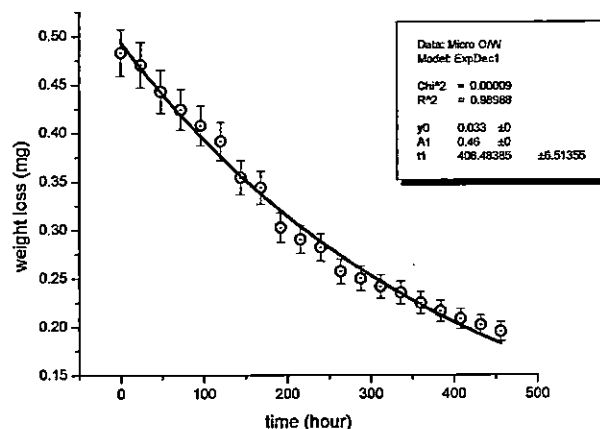


Fig. 9. o/w microemulsions plot for water pH=4.5, CTAB and pentanol system

Curve fitting for Fig. 9 would give the formula as

$$y = Ae^{-kt}$$

2) *o/w* microemulsions in water pH=4.5, CTAB and pentanol system in addition of methyl red

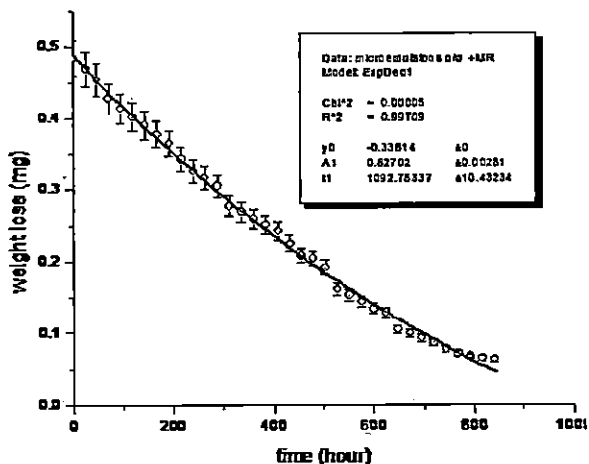


Fig. 10. *o/w* microemulsions plot for water pH=4.5, CTAB and pentanol system in addition of methyl red

3) *o/w* microemulsions in water pH=9.5, CTAB and pentanol system

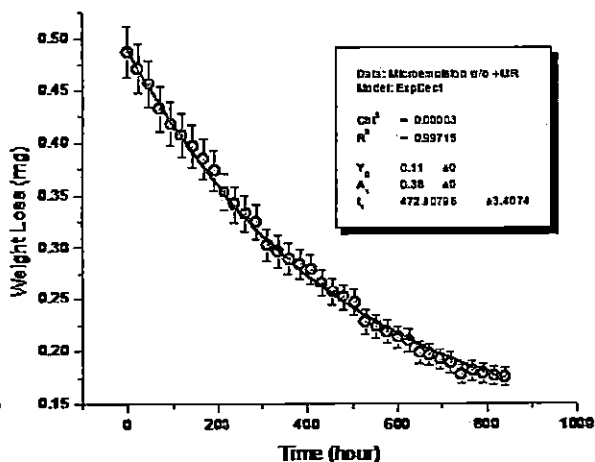


Fig. 11. *o/w* microemulsions plot for water pH=9.5, CTAB and pentanol system

4) *w/o* microemulsions in water pH 9.5, CTAB and pentanol system

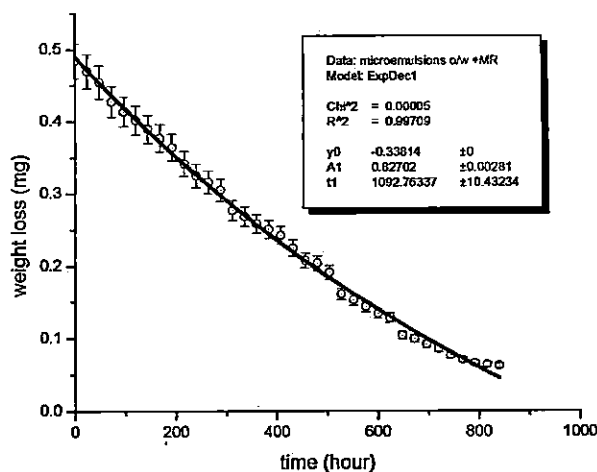
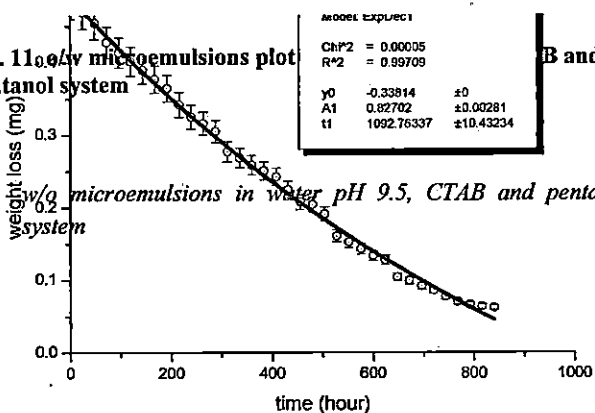


Fig. 12. Fig. 13. *w/o* microemulsions plot for water pH=4.5, CTAB and pentanol system

Conclusions

In this research we have figured out the phase area of water pH=4.5 and pH=9.5, CTAB and pentanol. Our finding is they were four phase areas i.e. *o/w* and *w/o* microemulsions, lamellar and hexagonal liquid crystals, the boundary phase were; *w/o* microemulsions area for water pH=4.5; CTAB and pentanol systems formed a wide microemulsion band around 0-63% water; 0-52% CTAB and 22-100% pentanol. *O/w* microemulsions in excess of water formed in area of 73-100% water; 0-23% CTAB and 0-12% pentanol. Lamellar liquid crystals (LLC) was formed in area of 11-53% water; 25-69% CTAB and 6-33% pentanol. Hexagonal liquid crystals (HLC) was formed in area of 36-71% water; 25-69% CTAB and 6-33% pentanol, respectively. Whereas *w/o* microemulsions area for water pH=9.5; CTAB and pentanol system formed a similar wide microemulsion area around 0-62% water; 0-51% CTAB and 21-100% pentanol. *O/w* microemulsions in excess of water formed in area of 65-100% water; 0-22% CTAB and 0-16% pentanol. Lamellar liquid crystals (LLC) was formed in area of 21-62%; 19-54% CTAB and 9-39% pentanol. Hexagonal liquid crystals (HLC) was formed in area of 9-39% water; 49-61% CTAB and 2-34% pentanol, respectively. The solubility of dyes in the phase area was characteristics, which for system water pH=4.5 the highest solubility was given by lamellar liquid crystal, followed by *w/o* and *o/w* microemulsions respectively, whereas for system water pH=9.5 the solubility of methylene blue was given by *w/o* and followed by lamellar liquid crystal and *o/w* microemulsion respectively. Addition of dyes was not significantly changed refractive index and viscosity in average. Finally the evaporation rate measurement for systems indicated that system was followed the pseudo first order kinetics.

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