Original Research Article

Effect of Activity Coefficient of polyvalent ionic salt solution on Demulsification of soy Lecithin based Oil-in-Water Emulsion

ABSTRACT

Lecithin based Oil-in-Water (O/W) emulsion finds a lot of applications in food and cosmetic industries. The stability of O/W emulsion depends on properties of the emulsifying solution and the ionic impurities present. The present experiment well explained the role of polyvalent Lewis acid salt solution in establishing the stability of emulsion. Lewis acid salts solutions of NaCl, KNO_3 $BaCl_2$ $MgCl_2$, $Al_2(SO4)_3$ and $FeCl_3$ were used in the preparation of lecithin based O/W emulsion. The ionic activity coefficient calculated from Debye-Hückel limiting law (DHLL) for Lewis metal ions Na^+, K^+, Ba^{2+}, Mg^{2+}, Al^{3+} and Fe^{3+} were found to be 0.955, 0.965,0.869,0.872, 0.737 and 0.737 which directly decrease the pH value of the ionic solutions to 6.9,7.0,7.14,7.04, 2.2 and 2.5 respectively. The conductance of ionic solution increases with decrease in ionic activity coefficient. These trends very strongly destabilize the lecithin O/W emulsion showing a significant order of breakup time of different emulsion systems as 3.5, 2.0,1.4,1.0, 0.5 and 0.25 minutes. The effect of ionic activity coefficient on emulsion destabilization was well projected by microscopic view. Measurement of droplet diameter was conspicuous for monovalent and divalent metal ions ranging from 14-156 micrometer and number of larger droplets increases with increasing in the valence. However, the higher valence Al^{3+} and Fe^{3+} ionic solutions totally break the emulsion with immediate separation of oil and water layer having lower activity coefficients. Thus, the increased valence and conductance, decreased ionic activity coefficient and pH value of the emulsifying solution destabilize the soy lecithin based O/W emulsion significantly by increasing the interfacial tension and retarding the binding ability of the emulsifier.

Key words: *Emulsion, Lecithin, Lewis acid salt, Debye-Hückel limiting law, Ionic activity coefficient, Valence, breakup time*

1. INTRODUCTION

An emulsion is a colloidal dispersion between two immiscible liquids as in Oil-in-water (O/W) emulsions. Oil-in-water emulsions are two-phase systems in which water is the continuous phase and oil is dispersed phase. Emulsions find revolutionary effect in food, pharmaceutical, metallurgical and cosmetic industries. The stability and physicochemical properties of food grade emulsion play a key role in determining the application of the emulsion. In O/W emulsion the droplets interfacial membrane surrounding the droplets hugely decides shelf life, texture, appearance, and flavor [1]. The interfacial membranes are bound through the surface active molecules known as emulsifiers comprising surfactants, phospholipids, proteins and polysaccharides [2,3]. The probability of forming stable O/W emulsions containing oil droplets surrounded by multiple layer interfacial membranes was achieved by using food grade emulsifiers such as 0.5 wt % corn oil, 0.1 wt % lecithin, 0.0078 wt % chitosan, 0.02 wt % pectin, and 100 milliMole acetic acid, pH 3.0 in presence of salt concentrations (pH 4-8 at 0 milliMole NaCl and pH 3-8 at 100 milliMole NaCl) [4,5]. The assessment of stability of an emulsion is studied by simple observations like creaming volume measurement and microscopic view. Emulsifying ability is based on their chemical structures and include synthetic, natural and dispersion of droplets by surfactants as soap and detergent (6). Stability of O/W emulsion is enhanced by guar gum and lecithin concentrations (1, 0.75, and 0.5%wt). All emulsions can be stabilized at the studied concentrations of stabilizers. The interaction of the stabilizers in the mixture with each other at the interface binds the droplet and stabilizes the emulsions [7]. Soy lecithin with bio-silica stabilizes the O/W emulsion forming healthy food giving assured quality. The varying concentration of soy lecithin (0.05- 1.5%) and bio-silica (0.5- 1.5%) from rice husks shows stabilization of the emulsion and kinetics of destabilization is reported as first order. The destabilization rate of the emulsion was decreased by 25-50% in the presence of emulsifier mixture [8, 9]. The optimization of the formation and stability of an O/W emulsion was met by a mixture of emulsifiers lecithin, xanthan gum and sunflower oil. Optimum concentrations of Xanthan gum and lecithin were found to have significant influences on emulsion stability and mean droplet diameter [10,11]. The interfacial features of a surfactant are influenced by ions present in the pool of emulsion. The effect of magnesium salts ($MgCl_2$ and $MgSO_4$) and sodium salts (NaCl and Na_2SO_4) on interfacial characteristics of hyrdroxylated soybean lecithin shows that the magnesium salt more stabilize the emulsion than the sodium salt as Mg^{2+} ion strongly interconnects negatively charged head groups of the emulsion in oil-water interface by increasing surface elasticity and viscosity [12]

An emerging separation technology of emulsion liquid membrane (ELM) technique, found by Li in 1968 used as in the fields like hydrometallurgy, environmental engineering, biochemical engineering, pharmaceutical engineering and food technology [13,14]. Polyvalent metal ions destabilize the O/W emulsion and leads to the separation of oil and solute layers. Thus, the solutes can be separated and also concentrated [15]. Different types of chemicals that can be separated or recovered from industrial streams applying ELMs are organic acids, phenols, cresols, and amines, as well as metallic ions such as lead, copper, cadmium and mercury [16,17,18]. A similar technique was used to extract silver ions(Ag^+) from aqueous solution by emulsion liquid membrane from feed solution [19]. The valence of the ions directly related to the rate of breaking of emulsion. For O/w emulsion monovalent ion in NaCl slightly increase the absolute value of zeta potential with increasing NaCl electrolytic concentration [20,21]. Low concentrations of bivalent calcium in calcium chloride electrolyte have strong effect on zeta potential. The possible assumption is that the Cl^- ions believed to adsorb to the surface more strongly than Na^+ is negligible compared to the neutralizing effect of Ca^{2+} ions. Calcium ions also have a low degree of hydration in aqueous solution, making them even more prone to adsorb to negatively charged species than those of high degree of hydration [22]. Emulsion Stability depends on thermodynamic parameters and these are thermodynamically unstable systems. An emulsion can rapidly or slowly separates into two immiscible phases with time. There are some known common phenomenon of emulsion destabilization like droplet-droplet coalescence, flocculation, creaming, and Ostwald ripening [23]. Aggregation of droplets conspicuously affects shelf life and texture of emulsions [24]. The research review reports clearly indicate the factors influence the formation of O/W emulsion, destabilization and application of lecithin based oil-in-water emulsions. The survey also clarifies the physicochemical characterization of emulsions through various instrumental methods. Many literatures were discussed about the effect of polyvalent metal ions on the zeta potential which greatly affect the interfacial surface elasticity and viscosity along with microscopic image of different systems.

Present work was focused on the influence of activity coefficient from Debye-Hückel limiting law (DHLL) on lecithin oil-in water emulsion. The activity coefficient at unit ionic strength is variably destabilizing the emulsion. The effective nuclear charge increases the activity coefficient of an ion.

Conductance of the unit concentration of Na^+, K^+, Ba^{+2}, Mg^{+2}, Al^{+3} and Fe^{+3} were determined against valances.' A comparative study was enunciated between the conductance, effective nuclear charge and activity coefficient of the ionic solutions with stability of the emulsion. Emulsion break up time was determined over a time in relation with creaming and separation of two immiscible layers against effective nuclear charge on cations. The study is also concentrated on the coalescence of emulsion by microscope images and viewed the dispersed droplet-droplet aggregation to form the large droplet.

2. MATERIALS AND METHODS

2.1. CHEMICALS

AR grade NaCl, KNO_3, $Al_2(SO_4)_3$, $MgSO_4$, $BaCl_2$, Fe_2O_3 and LR grade hexane, Acetone, Chloroform, Acetic acid were purchased from Merck suppliers. Double distilled water from glass distillation plant and refined commercial sunflower oil from local market.

2.2. PREPARATION OF SOYA LECITHIN

100gm of soya bean seeds with 2-3 percent moistures was ground to fine powder. The powder was packed in the What Mann filter paper and placed in thimble of the soxhlet extractor. LR grade hexane was used as extracting solvent and extraction was continued for one hour. The oil was recovered from the solvent. Soya bean oil was stirred with 30 ml acetone and acetone insoluble matter (AIM) allowed to settle. The supernatant solution was decanted, and this process was repeated for another three times. The resulting AIM was flushed with nitrogen gas to remove traces of acetone [**25**]. Light pinkish powdery AIM obtained was used as soya lecithin and preserved in an air tight bottle.

2.3. SAMPLING

The following sets emulsion were prepared using commercial sunflower oil as shown in the Table1

Table 1 Sampling

Sample set	Soya lecithin(mg)	Sunflower oil (ml)	Volume of 0.001M Ionic solution
S_1	100	5	10 ml water
S_2	100	5	10 ml NaOH
S_3	100	5	10 ml NaCl
S_4	100	5	10 ml KNO_3
S_5	100	5	10 ml $BaCl_2$
S_6	100	5	10 ml $MgSO_4$
S_7	100	5	10 ml $Al_2(SO_4)_3$
S_8	100	5	10 ml $FeCl_3$

2.4. PREPARATION SOY LECITHIN BASED O/W EMULSION

Series of emulsions were prepared as shown in the Table 1. Volume/Volume and Volume/Weight percent of sunflower oil and soya lecithin were taken in a sterilized mortar and homogenized using a piston. When homogenized mixture was formed, Volume/Volume percent of salt solution was added with little portions and homogenizing till it forms primary emulsion. The resulting primary emulsion properly diluted and collected in 30ml graduated test tubes. The colloidal emulsion was allowed to stand at 25-28^{0}C for one hour.

2.5. DETERMINATION OF THE RATE OF CREAMING

The well homogenized emulsions in the graduated test tubes were allowed to stand over a time period of one hour. Separation of immiscible layers' starts as coalesces with emulsion. The volume of separations was noted per minute intervals till a constant level of volume reached [26]. Percentage of creaming volume was measured using the formula.

$$\% \text{ Creaming} = \frac{(V_E - V_C)}{V_E} 100$$

V_E = Total volume of the emulsion V_C = Creaming volume

2.6. pH MEASUREMENT

Exactly 100ml 0.001 molar salt solutions were prepared by using double distilled water in the cleaned volumetric flask. pH value of each solution were determined using standardized pH meter. 10 ml of this solution was used in the of emulsion. Guang Wang and Tong Wang's method with slight change was used to determine pH values [27].

2.7. DETERMINATION OF CONDUCTANCE

Conductance of each salt solution was determined using digital systronic conduct meter of 1mirco Siemen (**μS**) accuracy. The experimental salt solutions were prepared by series dilution method for accuracy. 100 ml of 0.1 M of each salt solution was prepared as stock solution and 10ml of this solution was diluted to 100ml gives 0.01M solution.10ml of the diluted solution give 0.001 M solution on further dilution to 100ml and whose conductance was determined.

2.8. IONIC STRENGTH AND ACTIVITY COEFFICIENT

Ionic strength electrolytic solutions were determined using respective concentration and stable valency of the ions using the formula

$$I = \frac{1}{2} \sum_{i=1}^{n} c_i z_i^2$$

Activity coefficient of polyvalent metal cations was determined by applying Debye-Hückel limiting law (DHLL)

$$-\log \gamma_i = \frac{0.51 z_i^2 \sqrt{I}}{1.33 + \alpha_i \sqrt{I}}$$

I = Ionic Strength of the solution (molekg^{-1}), Ci = Ionic concentration ith ion, Zi=Charge of the ith ion
γ_i= Activity coefficient, α_i = Effective diameter of the hydrated ion i in nanometers (10^{-9} m)

2.9. MICROMETRIC DETERMINATION OF DROPLET DIAMETER

Emulsion droplet diameter was determined by ocular micrometric method. Ocular micrometer was calibrated using stage micrometer [28,29]. One division of the ocular micrometer was calibrated using the formula

$$\text{One division of Oculometer} = \frac{ds}{d_O} Ds$$

d_S = Coinciding division on stage micrometer, d_O = Coinciding division on the oculometer (om),
Ds = One division of stage micrometer = 10 µm
Diameter of the droplet = d = No. div. across droplet X 1 div.om

3. RESULTS AND DISCUSSION

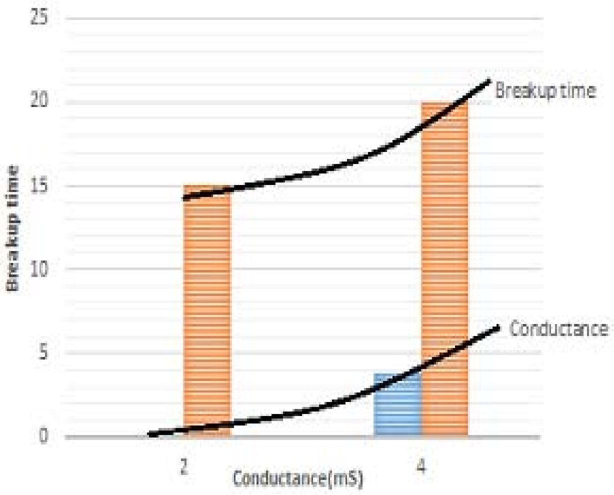
3.1. CONDUCTANCE OF IONIC SALT SOLUTION AND EMULSION BREAKUP TIME

Conductance of each salt solution was determined and compared with complete emulsion break up time as shown in the Table 2.

Table 2 Conductance of salt solution and emulsion break up time

Salt solution	Conductance (mS)	Emulsion break up time(minute)
Control(without salt)	0.108	15.0
NaOH	3.86	20.0
NaCl	0.286	3.5
KNO_3	0.241	2.0
$BaCl_2$	0.273	1.4
$MgSO_4$	0.220	1.0
$Al_2(SO_4)_3$	0.403	0.5
Fe_2Cl_3	0.755	0.25

The conductance of 0.001M salt solutions was increased with the increase in the valence (effective nuclear charge). A graph was plotted conductance versus breakup time as clear separation of oil and aqueous layer.



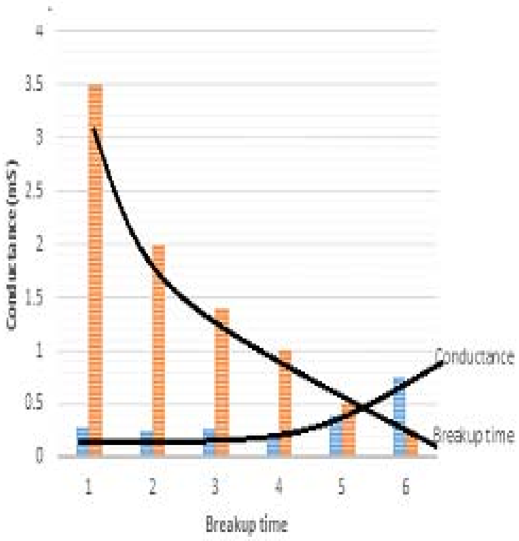


Figure 1a

Figure 1b

a = Breakup time with conductance of lecithin and NaOH emulsion
b = Conductance of ionic salt solution with emulsion breakup time

The plot explains the stability of the emulsion. Conductance increases as the valence of metal ions of the salt solution increases (Fig 1b), in turn the stability of the emulsion decreases. When NaOH solution was used in the emulsion preparation, lecithin O/W emulsion was stabilized having long breakup time as compared with emulsion in distilled water (Fig 1a). NaOH solution acts as Bronsted base and decreases the interface surface tension on the droplets. Al^{3+} and Fe^{3+} ionic solution shown higher conductance (0.403mS, 0.755mS) in comparison with bivalent Ba^{2+}, Mg^{2+} (0.273mS, 0.220mS) ionic salt solution. Monovalent Na^{+}, K^{+} ionic salt solutions have recorded relatively higher conductance (0.286mS, 0.241mS) than bivalent ions. However, Na^{+} and K^{+} ions are smaller in size which attributes a stable emulsion with larger breakup time (3.5min, 2.0min). The overall trend observed was that an increased conductance of the ionic salt solution with variable valence destabilized the emulsion. The salt as Lewis acids destabilizes the emulsion or decreases the life time of the emulsion.

3.2. pH VALUE OF IONIC SOLUTION

pH value of each ionic solution used for emulsion preparation was determined as shown in the Table 3

Table 3 pH value of 0.001M ionic solutions

Ionic solution	P^H value	Emulsion breakup time
NaCl	6.9	3.5
KNO_3	7.0	2.0
$BaCl_2$	7.14	1.4
$MgSO_4$	7.04	1.0
$Al_2(SO_4)_3$	2.2	0.5
Fe_2Cl_3	2.5	0.25

The Table 3 records that the over al pH value decreases with increasing valence of the Lewis acid metal ions. Because, oxidation state of ions increase. As the pH value increases the emulsion breakup time decreases remarkably for divalent ionic solutions (1.4 -1.0 min) with some anomalies that the pH value increases for Ba^{2+} and Mg^{2+} ionic solutions. However, the pH values for Al^{3+} and Fe^{3+} were abruptly come down to 2.2 and 2.5 with nearly zero breakup time. It is concluded that the pH value increases with the increase of the valence of the Lewis acid salt metal ions and in turn the stability of the emulsion decreases significantly.

3.3. RATE OF CREAMING

Each homogenized emulsion was shaken well in the graduated (30 ml) test tube and allowed to stand at room temperature (28 - 30^{0}C). The percentage of creaming was noted down against time interval and a graph was plotted time against percentage of creaming.

Table 4 Percentage of creaming volume with time duration

Time interval	Samples							
	S_1	S_2	S_3	S_4	S_5	S_6	S_7	S_8
0	0	0	0	0	0	0	26.7	33.3
2	0	0	3.3	3.3	6.7	6.7	36.7	63.3
4	6.7	0	10	13.3	13.3	13.3	50	66.7
6	18.7	6.7	36.7	37.3	38.7	33.3	56.7	66.7
8	36	33.3	50	52	46.7	48.7	60	66.7
10	43.3	41.3	53.3	54.7	56.7	53.3	61.3	66.7
12	48	46.7	53.3	55.3	60	56.7	63.3	66.7

When the emulsion start breaking, the segregation of the droplet takes place. Over a time, the droplets combined to form enlarged or bigger one. As the result rate of creaming increases with time. A graph was plotted time in minutes versus percentage of creaming as shown in the Figure 2.

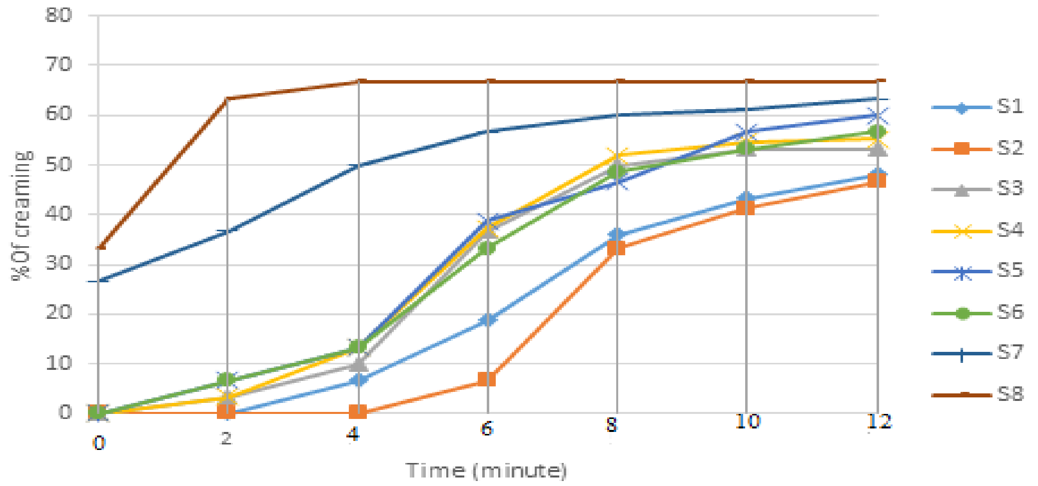


Figure 2 Rate of Creaming with time

The graph clearly explains the trend of separation of oil and aqueous layers. For samples S_1 and S_2 breakup of emulsion initiated after two and four minutes. Emulsion S_2 having sodium hydroxide shows decreased breakup time with the percentage of creaming responding higher stability of the emulsion. Emulsion S_1 having only oil and emulsifier follows the stability of the emulsion S_2. Rate of creaming increases moderately for monovalent Na^+, K^+ and bivalent Ba^{2+}, Mg^{2+} ions in the emulsions S_3, S_4, S_5 and S_6 respectively. Percentage of creaming was abruptly increased from zero to 26.7 and 33.3 % for the trivalent ions Al^{3+} and Fe^{3+} respectively showing zero breakup time. The trends in the graph with variable valences significantly explain the stability of the emulsion. As the valence of the metal ion in its salt increases, the stability of the emulsion decreases.

3.4. IONIC STRENGTH AND IONIC ACTIVITY COEFFICIENT

Ionic strength and ionic activity coefficient of the variable valence Lewis acid salt solutions were calculated using Debye-Hückel limiting law (DHLL) as shown in the Table 5

Table 5 ionic activity coefficient against emulsion breakup time

Ion	**Concentration mole/dm^3**	$I = \frac{1}{2}\sum_{i=1}^{n} c_i z_i^2$	α_i	Y_i	Emulsion Breakup time (min)
Na^+	0.001	0.001	0.4	0.955	3.5
K^+	0.001	0.001	0.3	0.965	2.0
Ba^{2+}	0.001	0.004	0.5	0.869	1.4
Mg^{2+}	0.001	0.004	0.8	0.872	1.0
Al^{3+-}	0.001	0.015	0.9	0.737	0.5
Fe^{3+}	0.001	0.015	0.9	0.737	0.25

A bar graph was plotted ionic activity coefficient with increasing time against emulsion breakup time as shown in the Figure 3

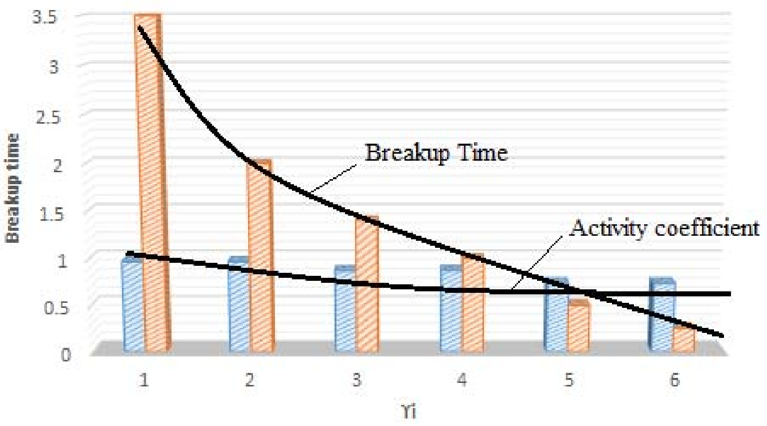


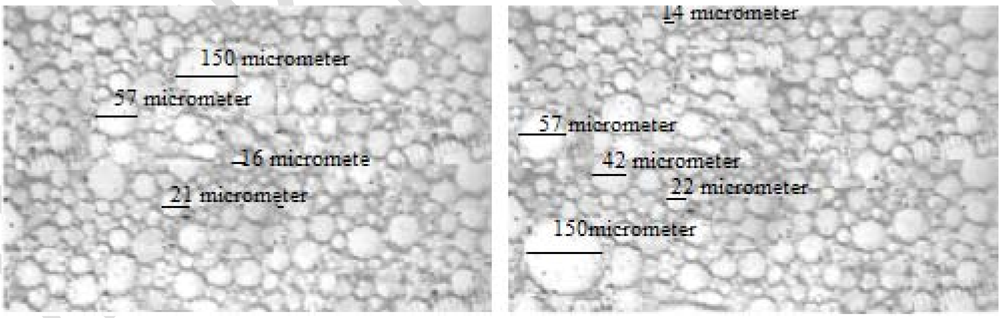
Figure 3 Plot of ionic activity coefficient versus emulsion breakup time

With increasing ionic strength, the ionic activity coefficient decreases. In turn, the valence of Lewis acid cations increases. The Figure 3 clearly explains the trend of ionic activity with breakup time of the emulsion. The monovalent ions Na^+ and K^+ have higher ionic activity coefficient (0.765, 0.755) and shows larger breakup time (3,5 and 2.0 min). The divalent Ba^{2+} and Mg^{2+} recorded the activity coefficient as 0.869 and 0.872 respectively having less breakup time 1.4 and 1.0 min. The trivalent ions Al^{3+} and Fe^{3+} gave lower activity coefficient 0.737 each showing lowest breakup time 0.5 – 0.25 min. The results discussed evidently correlate the activity coefficient of variable valence decrease with increasing valence. In turn, breakup time of the emulsion decreases with decrease in activity coefficient calculated from DHLL. Thus, activity coefficient from DHLL directly relates the destabilization of the emulsion. That is the emulsion was destabilized more and more with decrease activity coefficient. Attention was drawn towards the zeta potential which adversely affected by polyvalent charges on the metal cation of the salt. It is also observed that the average diameter of colloidal droplets measured with time. The stable and unstable emulsions were as shown in the Figure 4a, b

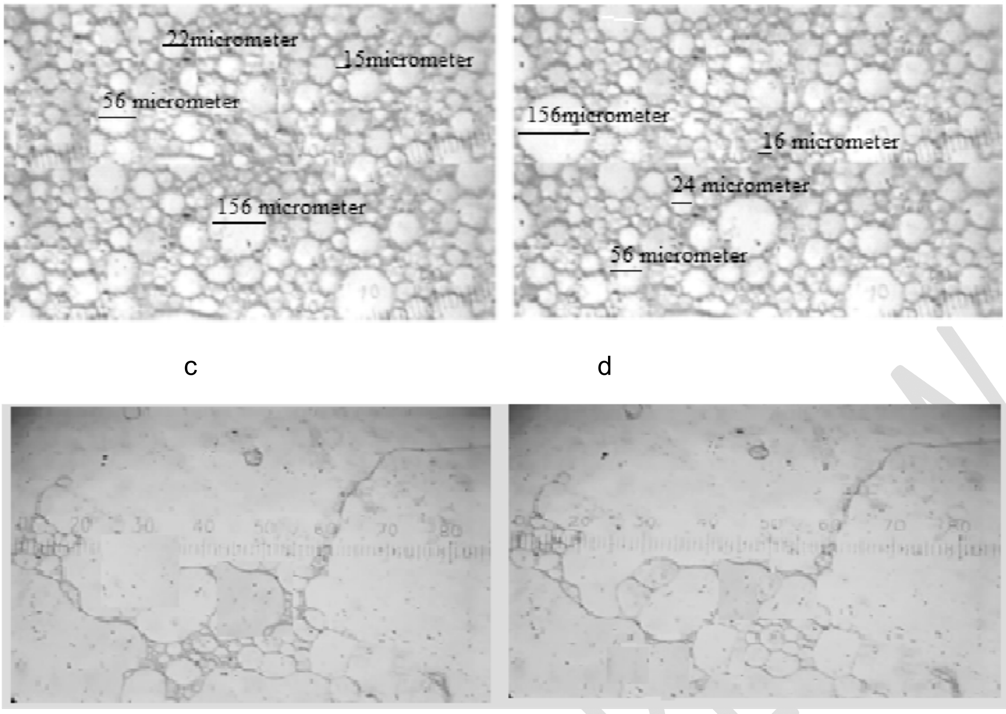
Figure 4 a. Stable emulsion due to Na^+ and Ba^{2+} b. Unstable emulsion due to Al^{3+} and Fe^{3+}

3.5. MICROMETRIC VIEW AND MEASUREMENT OF DROPLET DIAMETER

Homogenized emulsions were used in the micrometry to measure the diameter of the oil droplet dispersed in water. A small drop was mounted on the oculometer slide and observed using micrometer. The microscopic view of emulsion was as shown in the Figure 5



a b



c d

e f

Figure 5 Microscopic view of emulsion

a, b = Emulsion in Na^+ and K^+ solution c, d = Emulsion in Ba^{2+} and Ma^{2+} solution

e, f = Emulsion in Al^{3+} and Fe^{3+} solution

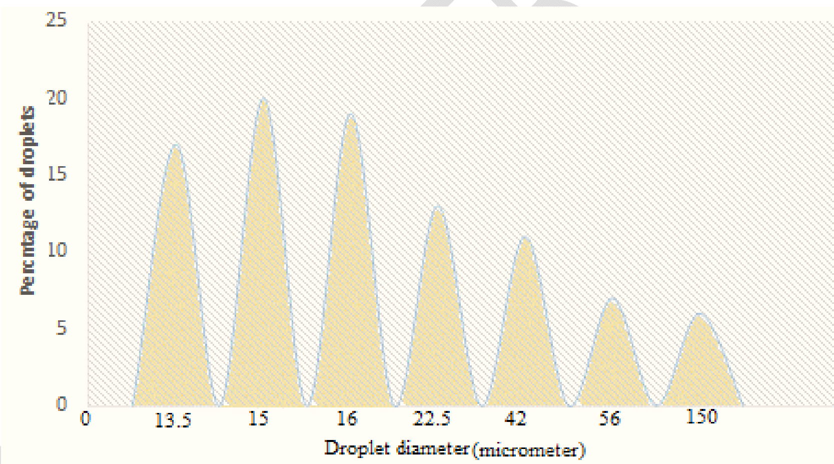
The microscopic view of the emulsions in ionic solutions significantly gives different size droplets. The monovalent Na^+ and K^+ ionic solutions shown maximum droplets of size 14 - 16 micrometers followed by droplets of size 21-22 micrometers. A few droplets of size ranges from 42 – 57micrometer and a very few droplets of size 150 micrometers were noticed. A similar trend was observed for the emulsions of Ba^{2+} and Mg^{2+} divalent ionic solution. But the number of droplets of size 14-16 micrometers were decreased and number of other droplets increased. As well as the droplet diameter increased as shown in the Table 6

Table 6 droplet diameter versus percentage droplets

Droplet	No.div. across droplet	1 div.om	d = No. div. across droplet X 1 div.om	Percentage
15	1.8	7.5	13.5	17
18	2.0	7.5	15.0	20
17	2.1	7.5	16.0	19
12	3.0	7.5	22.5	13
10	5.6	7.7	42.0	11
8	7.5	7.5	56	9
6	20	7.5	150	7

5	20.8	7.5	156	6

Average number of droplets under microscopic view was counted as reported in the Table 4 there by calculating the percentage of droplets in bulk of the emulsion. The percentage of dispersed oil droplets in water was conspicuously represented in the Figure 6.



The Figure 6 clearly explains the way how the dispersed droplets occupy the emulsion. Droplets of diameter ranging from 13.5 to 16 micrometers occupied the maximum of 56 percent of the total emulsion. Droplets with moderate diameter ranging from 22.5 to 56 micrometers covers about 33 percent and that of large diameter ranging from 150 to156 micrometers covers an area small (21%).The microscopic view clarifies that the numbers of larger droplets were increased significantly for Ba^{2+} and Mg^{2+} compared to Na^{+} and K^{+} ionic solutions showing increased coalesces with emulsion and the dispersion of the droplets is correlated with the observation by Johnson Kwame Efavi, et al [12]. The microscopic image of the emulsions in Al^{3+} and Fe^{3+} ionic solution couldn't be dispersed and no droplets were found under the microscope.

4 CONCLUSION

Stability of O/W emulsion depends on pH value, nature of the emulsifier, ionic strength and nature of the surfactant. This study very significantly presents the effect Debye-Hückel activity coefficient on ionic strength, P^H value, conductance and stability of the emulsion. Activity coefficient of polyvalent Lewis acid solution decreases from monovalent to trivalent ionic solution. Increased valence destabilizes the emulsion by decreasing the interfacial tension between oil and water. In turn, the binding ability of lecithin as emulsifier deteriorate and the emulsion starts coalesces. Because, the interfacial tension decreases with increasing ionic strength and activity coefficient of metal ions. This leads to break the emulsion through conspicuous separation of oil and water layer. Thus, segregation of oil droplets takes place, and large globules of oil was formed. This process took momentum with varying the valence of Lewis acid metal ions. Therefore, Al^{3+} and Fe^{3+} have lower activity coefficient and pH value couldn't form stable emulsion compare to divalent Ba^{2+}, Mg^{2+} and monovalent Na^+, K^+ ionic solutions.

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