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A Study on Household and Industrial Surfactants through Emulsion Stability Trials

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Abstract : Surfactants are a surface active agents, which plays a vital role in both Household and industrial applications. Surfactants happen to be a part of our daily life style, from morning tooth paste to night dish washing solutions. Domestic surfactants are a good cleaning agent, emulsifier, binders, corrosion inhibitors and bleaching agents. The household surfactant products performance makes our life in good health and cleanness. Surfactants applications in industries assist the production processes in more refined and hygienic way. The emulsifying ability determines the surfactants capacity to remove oil from clothes or combine the immiscible liquids to form creams. The household and industrial surfactants selected for the experimental studies were found to contain a good emulsifying property with various oils tested. The selected surfactants was capable of withstand a higher salt concentration and a stable emulsion formed up to 100°C.

Keywords : Surfactants, Emulsion, immiscible, binders, cleanness, inhibitors, bleaching, oils.

Introduction

Surfactant is an amphiphilic molecule, which contain both hydrophilic and hydrophobic domain. The nature of the hydrophilic part of surfactant is to get attached to the hydrophobic fluid and the hydrophobic part is fond of the hydrophilic fluid, in turn the two immiscible liquids are emulsified by the surfactant. The surfactant is a good emulsifying and foaming agent. Surfactants are classified as anionic, cationic and non-ionic. Unlike cationic surfactants, anionic and non- ionic surfactants are frequently in practice due to its trouble-free production and high efficiency. Surfactants are equally popular in household and industrial applications [1, 7, 8].

Surfactant plays a numerous number of roles in household mainly in cleaning and food products. Depending upon the specific surfactant product purchased from the market, the functions of surfactant in household are cleaning, foaming, bleaching, emulsification and anti-microbial activity. Some of the household surfactant products are soap, tooth paste, shampoo, dish washing liquid, detergents and hand sanitizer; personal care products such as face creams, face washing liquids and mouth fresher; food products, textile, painting, coating and oil products [4, 5, 10].

The surfactant is capable of reducing the surface tension of the liquids involved. The emulsion in return is achieved by reducing the interfacial tension between the two immiscible liquids; industries make use of these abilities of surfactant in various applications. In industries, the surfactants are implemented in oil production process, pulp and paper plants, tanneries and most of the food industries. Several of the industrially used surfactants are Ammonium Lauryl Sulphate, Sodium Lauryl Sulphate, Per Fluoro Octane Sulfonate, Alkyl Ether Phosphates, quaternary ammonium salts, Tween 80, Triton X-100 and Dimethyl Sulfoxide [6].

The cost of industrial surfactants is high due to the surfactants pure state to avoid the side reaction and contaminations, whereas the household surfactants contain only a specific quantity of pure surfactant (i.e. in low concentration) along with binders, bleaching agents and softening agents depending upon the vital purpose. The production of surfactants should be increased due to the uprising demand from household and industrial sectors. The use of laundry liquids are more purchased than bars or powder detergents in these days due to modernization in washing machines. The fashionable food products, clothing and effective cleaning agents in industry will increase the demand in upcoming years.

Materials and Methodology

A. Collection of surfactants and oils

Three different kind of household surfactant products were purchased from the supermarket, while the industrial surfactants such as Sodium Lauryl Sulphate (SDS) and Triton X-100 along with sodium chloride (NaCl) was purchased from a chemicals company. The Waste Lubricating Oil (WLO) was collected from the automobile shops and three different brands of oil were bought from the market.

B. Requirement of Equipment

Conductivity meter, pH meter, heating mantle and a Redwood viscometer for the determination of oil viscosity.

C. Determination of physical properties of oil

The waste lubricating oil and the three different band oils namely 90L, 90G and 320G were checked for their physical properties. Physical properties of oil such as color observation, density, API gravity, Kinematic viscosity and dynamic viscosity were determined before initializing the examination. The viscosities of the different kinds of oil were determined by using the Redwood viscometer. The kinematic viscosity of the oil was calculated from the formula [2,3]:

Kinematic viscosity = $A \cdot t - (B/t)$ in centistokes

If

Time of flow (t in seconds)	A	B
Up to 100	0.0026	1.72
Above 100	0.00247	0.5

The dynamic viscosity was calculated from the kinematic viscosity and density and their relation is given by
Dynamic viscosity = kinematic viscosity \times density in Kg/ms

D. Emulsion formation tests of surfactant products with Waste lubricating oil

Initially, 0.1% concentration of three different surfactant products was made at a constant salt concentration. High cleaning efficiency household surfactants were chosen for the experimental studies. Each of the chosen surfactant products was solid, powdery and liquid in their physical forms. Chosen domestic surfactants were named as solid surfactant product, powder surfactant product and liquid surfactant product for the convenience. Waste lubricating oil (WLO) was allowed to stand for 24 hours and then filtered in order to remove the impurities. The emulsion tests were conducted by choosing the 9 different ratios of surfactant and oil [1].

Components	Ratios of the different surfactant and Waste Lubricating Oil (WLO)								
Surfactant	9	8	7	6	5	4	3	2	1
WLO	1	2	3	4	5	6	7	8	9

The phase behaviour of the different ratios of the surfactant and WLO was visually observed and noted the emulsion forming ratios of all the three kind of surfactants. The different concentrations of all the three

surfactants were prepared from 0.1% to 0.5% surfactant concentration and the phase behaviour was observed. The optimum surfactant concentration, which has the ability to form emulsion with the WLO in the minimum surfactant concentration of all the three surfactant products was noted and used for the further experimental studies. The same procedure was carried for the industrial surfactants and the optimum surfactant concentration was determined. WLO was the oil used throughout the research to conduct the test effect of salt concentration and temperature on surfactant products.

E. Identification of the emulsion nature

The emulsion formed during the experimental study may be of two types. They are

1. O/W emulsion

In O/W emulsion, the water is the continuous medium and oil is the disperse medium. This kind of emulsion is also called as water based emulsion, due to the higher contribution of aqueous phase than oil phase [9].

2. W/O emulsion

In W/O emulsion, the oil is the continuous medium and water is the disperse medium. This kind of emulsion is also termed as oil based emulsion, in which the water droplet is dispersed in the oil medium. The emulsifier used will be highly hydrophobic character [9].

In order to find the type of emulsion formed, here two kinds of tests were conducted.

1. Dilution tests

The dilution test was conducted in the emulsion formed, with either water or oil. If the excess oil is diluted with the emulsion and still the emulsion is stable, then it is a W/O emulsion. If the excess water is diluted with the emulsion and the emulsion is not disturbed, then it is an O/W emulsion.

2. Conductivity tests

The conductivity test was conducted in the conductivity meter. If the measured conductivity of emulsion is near the conductivity of oil, then it is a W/O emulsion; while the measured conductivity of emulsion is near the conductivity of water, then it is an O/W emulsion.

F. Effect of different oils on surfactant

The optimized surfactant concentration of all the surfactants (both household and industrial surfactants) and their emulsion formation ratios were taken into account and further used for the effect of surfactant studies on various parameters such as different oils, salt concentration and temperature. The 90L, 90G and 320G were taken in the different ratios as above mentioned and tested with the optimum surfactant concentration of all the surfactants. The emulsion formation ratios were observed and verified with the results of WLO, whether the same minimum amount of surfactant concentration has the ability to form emulsion with different kind of oils.

G. Observation of emulsion instability

Emulsion formed in various ratios of all the surfactant with different oils such as WLO, 90L, 90G and 320G were separated and the emulsion is allowed to stand for a period of 24 hours. The emulsion instability possibility such as cracking, creaming, sedimentation, flocculation, coalescence and phase inversion were checked out. Creaming and sedimentation are one of a kind of emulsion instability, which depends upon the movement of the particles to the apex or base of the container. Unlike other emulsion instabilities, sedimentation and creaming can be emulsified by simple shaking or stirring. Flocculation is an irreversible process, which occurs due to the van der Waals attraction. In flocculation, the emulsion formed droplets are formed as aggregates and increase the rate of creaming. In case of flocculation, the emulsion stability is maintained by coalescence which is leading the emulsion. The phase inversion is emulsion unsteadiness and commonly occurs in finer products. In phase inversion, the emulsifier is producing an O/W emulsion and then the phase changed from O/W emulsion to W/O emulsion [1, 9].

H. Effect of salt concentration on surfactants

The optimum surfactant concentration of all the surfactants were taken and prepared in the individual surfactant concentration with different salt concentration from 1% to 5%. The impact of different salt concentration on surfactants tests were conducted only to the emulsion forming ratios of surfactant and WLO. The emulsion results were noted and the nature of emulsion was verified.

I. Effect of temperature on surfactants

The experiment was conducted similarly to the effect of salt concentration on surfactants. The optimum surfactant concentration of all the surfactant were prepared and separated in different test tubes and placed at different temperature from 30°C to 100°C. The heated surfactants were tested in the emulsion forming ratios of WLO. The emulsion formation and its kind were observed and noted.

J. Operation of Sand Pack Column

A sand pack column was designed to determine the efficiency of produced surfactant enhanced oil recovery process. A glass column was made with 9 cm in length and 3.1 cm in diameter [11]. The column was packed with medium sand material and to achieve uniform packing, the sand was poured gently in the column.

The sand pack column technique was followed as described [12]. The column was flooded with brine (5% NaCl) and ensures that the column was 100% saturated. The pore volume of the column was calculated by measuring the volume of brine required to saturate the column (PV). Next the column was saturated with waste lubricating oil in the same way as brine, until residual brine saturation was reached. As oil entered into the column, brine was displaced and discharged to the bottom of the column. Initial oil saturation (S_{oi}) was calculated by measuring the volume of brine displaced by oil saturation, also called original oil in place (OOIP). Initial oil recovery was done by water flooding with brine, so the sand pack column was again flooded with 10-11 Pore volume of brine until there was no more oil found in the effluent, that is the residual oil saturation (S_{or}) was reached. The amount of crude oil retained in the sand pack was determined volumetrically. S_{or} was calculated by measuring the volume of displaced oil.

0.5 Pore volume of surfactant was passed through the column similar to brine and oil and incubated for 24 hours, then the column was again flooded with brine. Discharges from the column were collected to measure the amount of oil recovered using surfactant. The column was flooded with water (secondary flooding) followed by SDS and Tween 80 separately (chemical flooding).

The percentage of oil recovered was calculated as follows,

$$\text{Initial Water Saturation, } S_{wi} (\%) = ((PV - OOIP)/PV) \times 100$$

$$\text{Initial Oil Saturation, } S_{oi} (\%) = (OOIP/PV) \times 100$$

$$\text{Residual Oil Saturation, } S_{or} (\%) = ((OOIP - S_{orwf})/OOIP) \times 100$$

Additional Oil Recovery,

$$\text{AOR (\%)} = (\text{Oil recovered using surfactant} / \text{Oil in column after water flooding}) \times 100$$

$$\text{AOR (\%)} = (S_{orsf} / (OOIP - S_{orwf})) \times 100$$

Where,

Pore Volume (PV) (ml) = Volume of brine required to saturate the column

Original Oil in Place (OOIP) (ml) = Volume of brine displaced by oil saturation

S_{orwf} (ml) = Oil recovered after water flooding

S_{orsf} (ml) = Oil collected over residual oil saturation after Surfactant flooding

Results and Discussion

A. Properties of surfactants and oils

The properties of all different oils were determined and tabulated in turn to understand their physical properties respectively. The rheological property of oil was generally obeys Newton's law and said to be a Newtonian fluids. When the oil is mixed or contaminated with impurities such as wax, paraffin, clay and sand materials then the behaviour of oil was switched from Newtonian to non-Newtonian fluid behaviour.

Table 1. Physical properties of different oils at 50°C

Different type of oils	Density of oil (kg/m ³)	Kinematic viscosity (Centistokes)	Dynamic viscosity (kg/ms) ×10 ⁴
WLO	850	0.6824	5.8004
90L	930	1.1846	11.017
90G	850	1.2192	10.363
320G	890	2.1854	19.45

The emulsion stability assessments on household and industrial surfactants were conducted with the different sorts of oil, which was shown in the table 1. The table 1 exhibits the density and viscosity of various oils that has been used in the experimental studies. The WLO was found to be a low density and viscosity oil, whereas 320G was nearly having a high density and viscosity oil.

The property of surfactants such as pH and foaming nature was observed before the experimental studies. Apart from the property mentioned, the surfactant has a good emulsifying property, cleaning agent and anti-microbial compound. The table 2 showed the nature and properties of the selected surfactant products and chemical surfactants.

Table 2. Properties of household and industrial surfactants

Type of surfactant	Classes of surfactant	pH	Foaming property
Solid surfactant product	Anionic surfactant with binders	8-9	Less foaming due to the solid physical form
Powder surfactant product	Anionic surfactant with bleaching compounds and fragrance	7-9	Better foaming material
Liquid surfactant product	Anionic surfactant with softening agents and water	7-8	Good foaming agent
Sodium lauryl sulphate (SDS)	Purely an anionic surfactant	7-9	Good foaming agent
Triton X-100	Purely a non-ionic surfactant	6-8	Good foaming agent

B. Emulsion formation tests on the surfactants with WLO

Table 3 clearly shows the ability of 0.1% surfactant to form emulsion in varying ratios of WLO. The results for different concentration of surfactant were same as 0.1% surfactant concentration, which was likely the outcome for all different kinds of surfactants. The 0.1% surfactant concentration was taken as the optimum surfactant concentration for further analyses.

Table 3. Phase behaviour observation of household and industrial surfactants with WLO

Ratios of different surfactant and WLO		Solid surfactant product	Powder surfactant product	Liquid surfactant product	Sodium lauryl sulphate (SDS)	Triton X-100
0.1% Surfactant	WLO					
9	1	-	-	-	-	-
8	2	-	-	-	-	-
7	3	-	-	-	-	-
6	4	-	-	-	-	-
5	5	-	-	-	-	-
4	6	-	-	-	-	-
3	7	-	-	-	-	-

2	8	+	+	-	+	-
1	9	+	+	+	+	+

Symbols “-“ means phase separation and “+” means complete emulsion

C. Identification of the emulsion nature

The particular ratios of surfactant and WLO were able to form emulsion in the previous step. At the same moment, the type of emulsion formed was analysed by emulsion tests of dilution and conductivity. All the emulsion formed during the experimental studies of different surfactant concentration, a W/O emulsion was produced. W/O emulsion implies that the surfactants applied were highly soluble in oil compared to water. In dilution test, the addition of excess WLO does not seem to disturb the emulsion. In conductivity test, the formed emulsion was tested for the conductance and found to be having the same conductivity of WLO. The emulsion tests were carried out whenever the emulsion was formed during the investigation.

D. Emulsion instability

The formed emulsion is stable for a only a particular period of time in a single phase. As the time passes, the emulsion instability occurs. Sedimentation is the only emulsion instability arises during the course of the experimental studies. Sedimentation is a temporary state of phase behaviour as well a reversible process, happens when the density difference between the two immiscible liquids that forms the emulsion is greater with one another. Stokes law was applied to determine the rate of sedimentation [9].

$$\text{Rate of sedimentation} = \{2r^2 (\rho - \rho_0) g\} / (9\eta)$$

Where

r = Droplet radius

ρ = Density of the droplet

ρ_0 = Density of the dispersion medium

η = Viscosity of the dispersion medium

g = Acceleration due to gravity (9.8 m/s²)

Generally, the sedimentation type of emulsion instability occurs in the W/O emulsion and creaming occurs in the O/W emulsion. The term $\rho - \rho_0$ gave the idea whether the emulsion formed will undergoes sedimentation or creaming. If the $\rho - \rho_0$ is a positive value, then it is sedimentation emulsion instability and vice versa.

E. Effect of different oils on household and industrial surfactants

The optimized surfactant concentration of all the surfactant was tested with 90L, 90G and 320G. The result of emulsion forming ratios in WLO was varied in different oils. Depending upon the physical and chemical properties of the oils, the emulsion ratios were affected greatly. Even though the ratios of emulsion formation changes, the type of emulsion formed remains unchanged (i.e. W/O emulsion). The results were tabulated along with the results of WLO. Table 4 shows that the powder surfactant product was able to form emulsion in different oils in the ratio 1:9 of surfactant and oil, on the other hand the solid and liquid surfactant products would not have the ability to form emulsion in different oils except WLO. When all the surfactants were compared, the SDS has good efficiency in forming emulsion in various oils as experimented.

Table 4. Observation of Phase behaviour on household and industrial surfactants with different oils

Different salt conc.	Ratios of different surfactants and oils		Solid surfactant product	Powder surfactant product	Liquid surfactant product	SDS	Triton X-100
	Surfactant	Oil					
WLO	2	9	+	+	-	+	-
	1	8	+	+	+	+	+
90L	2	9	-	+	-	+	-
	1	8	-	+	-	+	-
90G	2	9	-	-	-	+	-
	1	8	-	+	-	+	-
320G	2	9	-	-	-	+	-
	1	8	-	+	-	+	-

Symbols “-” means phase separation, “+” means complete emulsion

F. Effect of salt concentration on surfactants

The optimized surfactant concentration of all the surfactant was tested with different salt concentrations. Table 5 shows that the solid surfactant product was stable up to 3% NaCl concentration, while the liquid surfactant product can withstand the salt concentration up to 4% NaCl in the particular emulsion ratios. The powder surfactant product emulsion ratios exhibited a deviation from other surfactant products, which showed a stable emulsion up to 5% NaCl only in the ratio of 1:9 of surfactant and WLO. The industrial surfactants SDS was stable up to 3% NaCl, in the meantime Triton X-100 was stable up to 5% NaCl concentration in the ratio 1:9 of surfactant and WLO. The nature of emulsion i.e. W/O emulsion was unaltered, even in higher salt concentration.

Table 5. Examination of Phase behaviour on household and industrial surfactants with different salt concentration

Different salt conc.	Different ratio of surfactant and WLO		Solid surfactant product	Powder surfactant product	Liquid surfactant product	SDS	Triton X-100
	Surfactant	WLO					
1% NaCl	2	9	+	+	Nil	+	Nil
	1	8	+	+	+	+	+
2% NaCl	2	9	+	-	Nil	+	Nil
	1	8	+	+	+	+	+
3% NaCl	2	9	+	-	Nil	+	Nil
	1	8	+	+	+	+	+
4% NaCl	2	9	-	-	Nil	-	Nil
	1	8	-	+	+	-	+
5% NaCl	2	9	-	-	Nil	-	Nil
	1	8	-	+	-	-	+

Symbols “-” means phase separation, “+” means complete emulsion and

“Nil” indicates the specific ratio is not checked due to its phase separation in initial stage.

G. Effect of temperature on surfactants

The surfactant liquids heated in different temperatures were mixed in the emulsion ratios with the WLO and observed the results. All the surfactant products were capable of forming emulsion in all the temperature range from 30°C to 100°C without any phase transformation of W/O emulsion.

H. Operation of Sand Pack Column

Surfactant products were tested for its application in *ex-situ* enhanced oil recovery using sand- pack column technique. Table 6 showed that, various surfactant products and chemical surfactant (such as SDS and Triton X-100) have the ability to enhance oil recovery with the sand-pack column. Initial, pore volume of the sand pack volume was found to be 13.5-14.5 ml. The Original Oil In Place (OOIP) of the column was in the range of 12-13.5 ml. After waterflooding, the residual oil saturation was reached in the range of 53.6-62.5% in the column with medium sand particle. The powder surfactant product is nearly capable of additional oil recovery compared with the SDS and Triton X-100. The Additional oil recovery of various surfactant products and the chemical surfactants were plotted in the graph that helps to clearly understand the recovery efficiency of the household and industrial surfactants individually and comparatively.

Table 6. Enhanced oil recovery of various surfactant products and chemical surfactant in sand pack column by using a medium sand particles

	Solid surfactant product	Powder surfactant product	Liquid surfactant product	SDS	Triton X-100
PV (ml)	13.5	14.5	14	14.1	13.8
OOIP (ml)	12	13	12.5	13.5	12.5
S_{orwf} (ml)	4.5	5.5	5	6	5.8
S_{orsf} (ml)	3	5	3.5	5.5	4.5
S_{oi} (%)	88.88	89.66	89.28	96.43	90.58
S_{wi} (%)	11.11	10.34	10.71	4.25	9.420
S_{or} (%)	62.5	57.69	60	55.55	53.6
AOR (%)	40	66.66	46.66	73.33	67.16

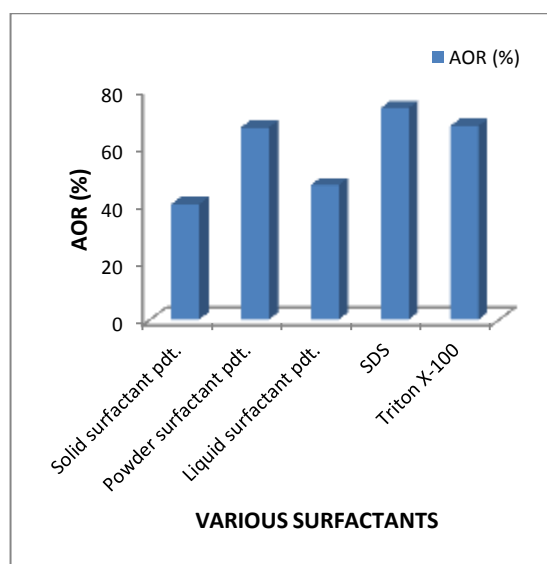


Figure 1 Percentage of additional oil recovered by various surfactant products and chemical surfactants in sand pack column

Conclusion

The household and industrial surfactants are differentiated mainly by their indispensable purposes. The cost of industrial surfactants is high compared to the household surfactants, although when the purity of surfactants is considered than the industrial surfactants are unmatched.

The emulsion stability investigation showed the household surfactants have the ability to form emulsion nearly in the same way as that of the industrial surfactants. A very minimum amount of surfactant was able to form emulsion with a variety of oil. The household surfactants are capable of withstanding a higher salt concentration and temperature till 100°C.

The commercial household surfactants have good emulsification ability with different kinds of oil. The production cost of pure surfactants cannot be reducing by these investigations, whereas the consumption of the pure surfactant in industries can be replaced by means of household surfactants wherever if possible.

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