

# Synthesis evaluation and adsorption studies of anionic copolymeric surfactants based on fatty acrylate ester

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## Abstract

A series of anionic copolymeric surfactants based on *n*-dodecylacrylate ester ( $M_1$ ) as hydrophobe, and oxypropylated acrylate ester ( $MA_{4,6}$ ) as hydrophiles, were prepared by copolymerization of *n*-dodecylacrylate ( $M_1$ ) and oxypropylated acrylate ester ( $MA_{4,6}$ ) with molar ratio's (0.3:0.7, 0.4:0.6 and 0.5:0.5, respectively) in presence of benzoyl peroxide as initiator followed by sulfation and neutralization to afforded [(PAS<sub>4</sub>), and (PAS<sub>6</sub>)]<sub>a-c</sub>, as anionic copolymeric surfactant in suitable yield. These derivatives were purified and characterized by IR and <sup>1</sup>H NMR spectral studies. Surface activity, and biodegradability were evaluated. Adsorption of some copolymeric surfactant on salary sand was investigated to assess possibility of treating waste water streams for removal of Pb<sup>2+</sup> and Hg<sup>2+</sup> toxic minerals. The effect of several factors governing the adsorption such as initial concentration, temperature, pH, have been studied.

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**Keywords:** Dodecylacrylate; Oxypropylation; Copolymerization reaction; Salary sand and adsorption

## 1. Introduction

A surfactant is a substance that has a characteristic molecular structure consisting of a hydrophobic portion together with a polar or an ionic portion. When surfactant presents at low concentration in a system, it has the property of adsorbing onto the surface or interfaces of the system and altering to a marked degree the surface or interfacial properties. Polymer–surfactant interaction has been studied by various methods [1,2], all these studies indicate that polymers interact with surfactants by inducing micellization of surfactants on the polymer chain and after getting saturated with micelles the excess of surfactant forms free micelles [3]. Many workers investigated adsorption of phenol on different adsorbent by physico-chemical treatment processes as it is highly effective, the different adsorbent such as acetyl trimethyl ammonium bromide, treated silica [4,5], activated carbon obtained from dust of leather industry [6] and also absorption of chemically and biologically important com-

pound on the solid surfaces [7–9]. The authors aimed to prepare a novel series of anionic copolymeric surfactants (Scheme 1), and adsorption studies reveal that various adsorbents are used to remove Pb<sup>2+</sup> and Hg<sup>2+</sup> heavy metal ions from waste water.

## 2. Materials and methods

### 2.1. Materials

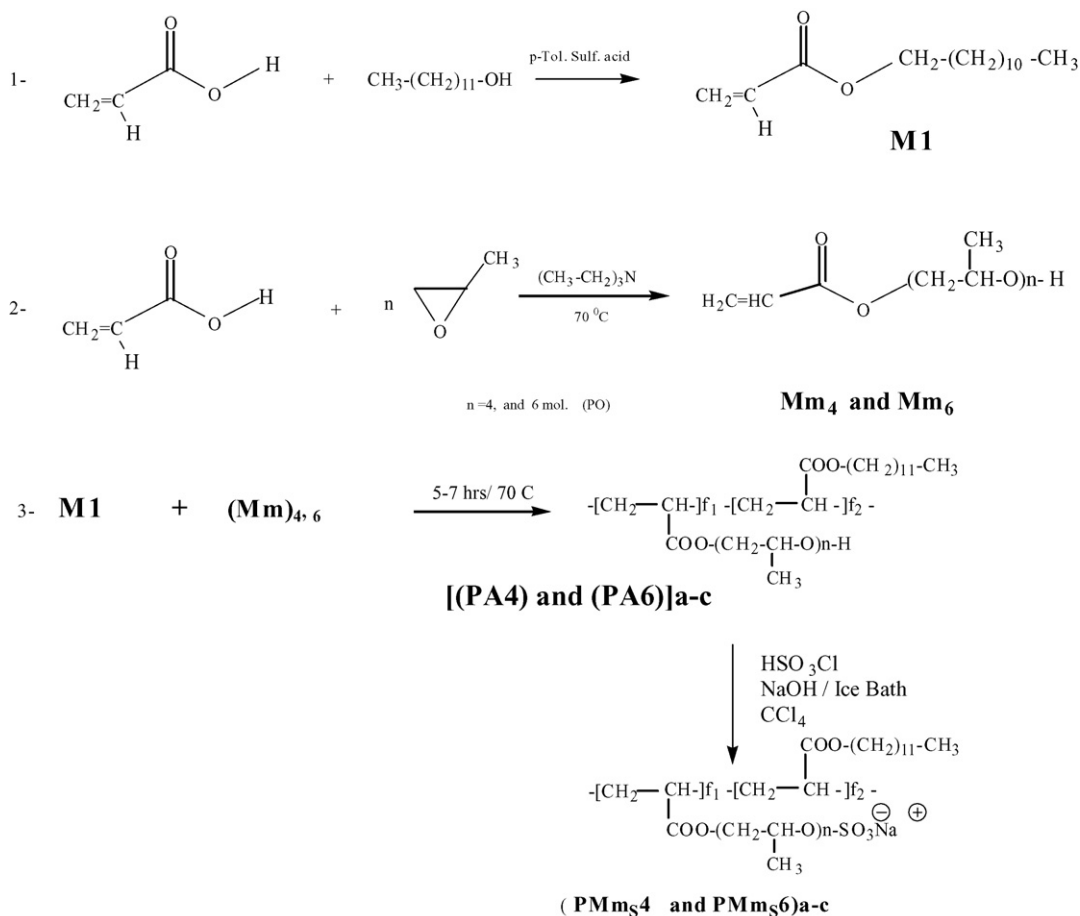
Acrylic acid, propylene oxide, *n*-dodecyl alcohol, *p*-toluenesulfonic acid, chlorosulfonic acid, sodium hydroxide and potassium hydroxide were Merck (Darmstadt) products. Benzoylperoxide was obtained from Aldrich (Steinheim, Germany).

### 2.2. Methods

The IR spectrum was measured by SHIMADZU IR 2000 spectrophotometer as KBr disc in Benha University, Faculty of Science, Chemistry Department, and <sup>1</sup>H NMR was done in CDCl<sub>3</sub> and/or DMSO as solvent and tetramethylsilane (TMS) as internal standard on Varian Mercury operating at 300 MHz in

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Where: ( PMm<sub>4</sub> and PMm<sub>6</sub> )a-c : a: f1=0.7 f2=0.3 b: : f1 =0.4 ,f2=0.6  
c : : f1 =0.5, f2=0.5

Scheme 1.

Chemistry Department, Faculty of Science, Cairo University, to confirm the structure for some examples of the prepared copolymeric surfactants.

- Dodecylacrylate ester (M<sub>1</sub>). Dodecyl alcohol was esterified with acrylic acid using Dean Stark adapter in dry benzene as solvent and *p*-toluene sulfonic acid as catalyst to afford *n*-dodecylacrylate as hydrophobic monomer (M<sub>1</sub>) in good yield [10].
- Oxypropenoxylation of acrylic acid. The procedure described by El-Sawy et al. [11], was followed in which, interaction of acrylic acid, propylene oxide and pyridine as catalyst was conducted at 70 °C for about 7 h.
- Copolymerization reactions [12]. The copolymers of M<sub>1</sub> with (MA<sub>4,6</sub>) were obtained by the solution copolymerization technique. The predetermined amounts of the monomers M<sub>1</sub> and (MA<sub>4,6</sub>) were placed in polymerization tubes and diluted with dimethylformamide (DMF). The total monomers concentration was 2 mol/l. The polymerization was initiated by adding 1 mol% benzoyl peroxide as a free radical initiator. The tubes were flushed with nitrogen gas for about 20 min then sealed and thermostated at 65 °C for

about 15–30 h depending on the monomer pairs and composition.

The copolymers were obtained by reprecipitation from petroleum ether (40–60 °C) and finally dried under vacuum at 60 °C to gave [(PA<sub>4</sub>) and (PA<sub>6</sub>)]<sub>a-c</sub>, as the structure of the obtained copolymers was confirmed via spectral data.

- Sulfation. The typical procedure illustrated by El-Sawy et al. [11], was followed to afforded [(PAS<sub>4</sub>) and (PAS<sub>6</sub>)]<sub>a-c</sub> as anionic copolymeric surfactants.

### 2.3. Surface properties

- Surface and interfacial tensions, were measured using a Du-Nouy tensiometer [13] (Kruss, Type 8451), with 0.1% aqueous solution at room temperature (25 °C).
- Kraft point, was measured as the temperature at which 1% dispersion solution becomes clear on gradual heating [14].
- Wetting time, these was determined by immersing a sample of cotton fabric in 0.1% aqueous solution of the surfactants [15].

- Foaming properties, these were measured according to Ross Miles method [16]. The foam production for 1.0% solution was measured by the foam height initially produced.
- Emulsion stability. The emulsion was prepared from 10 ml of a 20 mmole aqueous solution of surfactant and 5 ml of toluene at 40 °C. The emulsifying property was determined by the time it took for an aqueous volume separating from the emulsion layer to reach 9 ml counting from the moment of the cession shaking [17].
- Stability to hydrolysis. A mixture of 10 mmol polymeric surfactant and 10 ml 0.05N NaOH were placed in a thermostat at 40 °C. The time required for a sample solution to be clouded as a result of hydrolysis shows the stability of surfactant to hydrolysis [18].
- Ca<sup>2+</sup> stability [19]. Was determined by a modified Hart method. Where, the surfactant (10 mmole) solution was titrated against CaCl<sub>2</sub> (0.1N) solution. The end point was determined by visual observed of cloudiness the surfactant solution.
- Biodegradability. Biodegradability percentage was determined following the method of Eter et al. [20].

Batch adsorption experiments: were carried out by shaking fixed quantity of surfactants solution with known concentration containing 1 g of treated salary sand for about 2 h. The remaining amount of surfactant was estimated in supernatant solutions spectrophotometrically.

### 3. Results and discussion

Preparation of copolymeric surfactants containing oxypropenoxy sulfate groups was possible and could be isolated in suitable yield. Microanalytical data of the monomers and physical properties were tabulated in Table 1, infra red (IR) and proton nuclear magnetic resonance (<sup>1</sup>H NMR) spectroscopic analysis were conducted to confirm the structure of some representing examples of the monomers and copolymeric surfactants (Table 2).

#### 3.1. Surface properties

- Surface and interfacial tensions. The results recorded in Table 3, indicated that, the surface and interfacial tensions of the solutions are reduced relatively by addition of surfactants which might be due to the amphipathic structure causes of the surfactant molecules at the surface and reduced the surface and interfacial tensions [21]. On the other hand, increasing in the percentage of oxypropenoxy sulfate in the prepared copolymeric surfactants through each series [(PAS<sub>4</sub>), and/or (PAS<sub>6</sub>)]<sub>a-c</sub>, leads to decrease in the values of surface, interfacial tensions. This, phenomenon is due to the increasing in the hydrophilicity of the surfactant molecules that causes decrease of the concentration of the surfactants at the surface [21]. Also, the values of surface and interfacial tensions increase with increasing the percent of decylacrylate ester.

Table 1

Microanalytical data of the prepared dodecylacrylate (M<sub>1</sub>) and oxypropylated acrylate MA<sub>4</sub> and MA<sub>6</sub>

Compound no.	Molecular formula	Molecular weight	Yield (%)	Colour state	Microanalysis			
					C (%)		H (%)	
					Calc.	Fd.	Calc.	Fd.
M <sub>1</sub>	C <sub>15</sub> H <sub>28</sub> O <sub>2</sub>	240.39	98	Colourless liquid	74.95	74.90	11.74	11.70
MA <sub>4</sub>	C <sub>15</sub> H <sub>28</sub> O <sub>6</sub>	304.38	85	Colourless liquid	59.19	59.10	9.27	9.20
MA <sub>6</sub>	C <sub>21</sub> H <sub>40</sub> O <sub>8</sub>	420.54	83	Colourless liquid	59.98	59.80	9.59	9.40

Table 2

Spectral data of some representing examples for dodecylacrylate, oxypropylated acrylate

Compounds	<sup>1</sup> H NMR (δ = ppm)	IR (cm <sup>-1</sup> )
M <sub>1</sub>	δ 0.80 (t, 3H, term CH <sub>3</sub> ); δ 0.95–.50 (br.s.; 20H, CH <sub>2</sub> chain); δ 4.10 (t, 2H, –COO–CH <sub>2</sub> –CH <sub>2</sub> –); δ 5.90 (d, 1Ha, one proton Ha(Hb)C=CH–COOCH <sub>2</sub> –); δ 6.10 (t, 1H, CH <sub>2</sub> =CH–COOCH <sub>2</sub> –) and δ 6.30 (d, 1Hb, the second proton of Ha(Hb)C=CH–COOCH <sub>2</sub> –)	The IR spectra decylacrylate show the following data: 3130 cm <sup>-1</sup> ν <sub>C–H</sub> olefinic; 2990, 2920 cm <sup>-1</sup> ν <sub>C–H</sub> aliphatic, 1735 cm <sup>-1</sup> , ν <sub>C=O</sub> of ester; 1640 cm <sup>-1</sup> , ν <sub>C=C</sub> and 1450, 1260, 1150 cm <sup>-1</sup> , ν <sub>C–O</sub> respectively
MA <sub>6</sub>	δ 1.3 (d, 18H, term CH <sub>3</sub> in propylene oxide(6) unit.); δ 3.3–3.8 (br. m, 16H, CH <sub>2</sub> –CH(CH <sub>3</sub> )–) δ 4.20 (t, 2H, COO–CH <sub>2</sub> –CH(CH <sub>3</sub> )–OH); δ 4.90 (s, 1H, COO–CH <sub>2</sub> –CH(CH <sub>3</sub> )–OH); δ 5.80 (m., 1H, H <sub>2</sub> C=CH–COO–), δ 6.1 (d, 2H, CH <sub>2</sub> =CH–COO–) and δ 6.30 (d, 1Hb, the second proton of Ha(Hb)C=CH–COO) respectively	The IR spectra of the prepared monomers show the following data: 3480 cm <sup>-1</sup> ν <sub>C–H</sub> in propylene oxide, 3120 cm <sup>-1</sup> ν <sub>C–H</sub> olefinic; 2960, 2920, 2870 cm <sup>-1</sup> ν <sub>C–H</sub> aliphatic; 1450, 1240, 1100 cm <sup>-1</sup> ν <sub>C–O–C</sub> (with high intensity in M <sub>6</sub> , respectively
PSA <sub>4b</sub>	δ 0.80 (t, 3H, term CH <sub>3</sub> ); δ 0.9–1.2 (br.s.; 20H, CH <sub>2</sub> chain); δ 1.4 (d, 12H, term.CH <sub>3</sub> in propylene oxide unit); δ 3.1–3.8 (m, 12H, –CH <sub>2</sub> –CH(CH <sub>3</sub> )–); δ 2.8 (m, 1H, –CH <sub>2</sub> –CH(CH <sub>3</sub> )–OSO <sub>3</sub> <sup>-</sup> Na <sup>+</sup> ) and δ 4.20 (t, 2H, –COO–CH <sub>2</sub> –CH <sub>2</sub> –), with discharge of olefinic protons	The IR spectra of the prepared copolymeric surfactants show the following data: 2980, 2950 cm <sup>-1</sup> ν <sub>C–H</sub> aliphatic; 1735 cm <sup>-1</sup> ν <sub>C=O</sub> of ester; 1450, 1240, 1150, 1100 cm <sup>-1</sup> ν <sub>C–O–C</sub> and 1170, 1070, 680 cm <sup>-1</sup> characteristics for ν <sub>SO<sub>2</sub></sub> , ν <sub>S–O</sub> , respectively

And some examples of anionic copolymeric surfactants.

Table 3  
Surface properties of anionic copolymeric surfactants [(PAS<sub>4</sub>) and (PAS<sub>6</sub>)]<sub>a-c</sub>

Compound no.	S.T., 0.1% (dyne/cm)	I.F.T., 0.1% (dyne/cm)	Kraft P., 1.0% (°C)	Wetting t., 0.1% (s)	Foam h., 1.0% (mm)	Emul. stab. (s)	Ca <sup>2+</sup> stability (ppm)	Stability to hydr.	
								Base (min:s)	Acid (min:s)
PAS <sub>4a</sub>	31.50	08.5	2	32	230	412	2100	32:02	111:22
PAS <sub>4b</sub>	31.00	08.0	0	34	250	430	2200	33:52	112:31
PAS <sub>4c</sub>	32.50	09.5	<0	36	320	450	2400	35:20	123:00
PAS <sub>6a</sub>	33.0	08.0	0	39	220	380	2200	35:33	118:41
PAS <sub>6b</sub>	33.0	07.0	<0	41	240	474	2300	38:45	123:42
PAS <sub>6c</sub>	35.5	10.5	<0	42	290	490	>2400	39:22	128:32

- **Kraft point.** The results of Kraft point of copolymeric surfactants [(PAS<sub>4</sub>) and (PAS<sub>6</sub>)]<sub>a-c</sub>, are shown in Table 3. From the data recorded, the polymeric surfactants with higher percent of hydrophilic monomers which processes propenoxy units and –SO<sub>3</sub><sup>−</sup> Na<sup>+</sup> groups satisfy lower values of Kraft point, thus, might be lead to the widely used in industrial applications.
- **Wetting time.** The measured data of the wetting time for the prepared polymeric surfactants are recorded in Table 3, where, the wetting time decreases as the percent of hydrophilic monomer in the copolymeric chain decreases. On the other hand, wetting time increases slightly as the percent of hydrophobic monomers increased [22]. In general, the poor wetting times of copolymeric surfactants are recorded for PAS<sub>6c</sub> with lower propylene oxide ratio's (1 mol).
- **Foaming height.** The prepared anionic copolymeric surfactants exhibit relatively low foaming height. The low-foaming tendency of surfactants is recently considered as an important property in some applications such as dyeing auxiliaries in modern textile dyeing industry. The relative low-foaming properties of the copolymeric surfactants containing oxpropoxysulfate groups are probably due to the presence of multihydrophilic groups causing a considerable increase in the area per molecule and producing less cohesive forces at the surface, moreover, the repeating –SO<sub>3</sub><sup>−</sup> Na<sup>+</sup> of oxypropylated groups, are believed to coil in the aqueous phase and decreasing the cohesive force due to intra-intermolecular bond [26] (Table 3). From the data recorded copolymeric surfactants PAS<sub>4b</sub>, PAS<sub>6c</sub> revealed higher foam height than PAS<sub>4a,c</sub> and/or PAS<sub>6a,b</sub> respectively.
- **Stability to hydrolysis.** Copolymeric surfactants PAS<sub>6c</sub> exhibited excellent stability in acidic medium than in basic

media, this might be due to the easily hydrolysis of the ester groups cooperated with the—[CH(CH<sub>3</sub>)–CH<sub>2</sub>O]<sub>n</sub>–SO<sub>3</sub><sup>−</sup> Na<sup>+</sup> groups [22].

- **Emulsification stability.** From the data recorded in Table 3, the prepared compounds exhibited an excellent degree to form emulsions either O/W or W/O for those with high percent of hydrophobicity. So, it was reported that, better emulsifying properties were obtained with derivatives containing propylene oxide incorporated with –SO<sub>3</sub><sup>−</sup> Na<sup>+</sup> into their structure [22].
- **Ca<sup>2+</sup> stability.** From the data recorded in Table 3, it is found that, copolymeric surfactants with high percentage of propylene oxide revealed more tolerance towards the very hard water.
- **Biodegradability.** The results of biodegradability as shown in Table 4, reflect the fact that, it decreased with increasing number of repeating units of propenoxy group as well as, the repeating alkyl groups in hydrophobe unit [23]. Moreover, PAS<sub>4a</sub> recorded higher degree of degradation than the other prepared copolymeric surfactants, this gave rise to the conclusion that, a longer propenoxy group retains the diffusion of the molecule through the cell membrane, and thus the degradation would be more difficult.

### 3.2. Adsorption studies

The Freundlich adsorption isotherm

$$\log(x/m) = \log k + 1/n \log C$$

where *C* is the equilibrium concentration of surfactant (*x/m*) is the amount adsorbed. The plot of log(*x/m*) versus log *C* was obtained to be linear, it shows that the adsorption follows Freundlich isotherm. The values of *n* obtained were between

Table 4  
Biodegradability of anionic copolymeric surfactants [(PAS<sub>4</sub>) and (PAS<sub>6</sub>)]<sub>a-c</sub>

Compounds	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day
PAS <sub>4a</sub>	51.00	59.50	69.50	76.50	86.00	95.50	–
PAS <sub>4b</sub>	49.50	59.00	68.50	76.00	85.50	91.50	98.00
PAS <sub>4c</sub>	48.50	57.50	65.50	75.50	83.50	90.50	97.00
PAS <sub>6a</sub>	53.00	58.00	70.00	78.50	89.50	97.00	–
PAS <sub>6b</sub>	49.50	54.50	68.00	75.50	87.00	94.50	95.50
PAS <sub>6c</sub>	51.50	59.00	67.00	74.50	84.50	91.00	96.50

Table 5  
Variation of contact time temperature:  $25 \pm 0.5$  °C and initial concentration 50 ppm

Sl. no	Contact time (min)	Adsorption of $Y_2$ (%)		Adsorption of $Z_2$ (%)		Fraction removal of ions	
		$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$
1	5	26	24	33	25	0.30	0.25
2	10	28	26	36	30	0.32	0.28
3	15	30	28	40	33	0.35	0.31
4	20	33	35	44	34	0.38	0.35
5	30	35	42	46	35	0.41	0.39
6	40	38	45	47	37	0.42	0.39
7	60	45	47	48	43	0.46	0.45
8	120	55	57	58	54	0.56	0.55

2 and 10, which indicates favourable adsorption. The adsorption kinetics were studied by using Lagergren equation:

$$\log(q_e - q) = \log q_e - K_{ad}t/2.303$$

where  $q$  is the amount adsorbed at  $t$ ,  $q_e$  is the amount adsorbed at equilibrium time. Linear plots of  $\log(q_e - q)$  versus  $t$  indicate the applicability of Lagergren, further it confirms adsorption process as first-order kinetics, the value of  $K_{ad}$  determined from the slope. The rate constant of intra-particle diffusion  $K_p$  were determined using Weber and Moris relationship by plotting amount of surfactant adsorbed versus square root of time ( $q = K_p t^{1/2}$ ). The results reveal that the initial part of these plots is straight line not passing through the origin shows that intra-particle diffusion is not the only rate controlling step. The surface complex so formed at adsorbate–adsorbent interface attributed to anionic surfactant.

*Effect of contact time.* In adsorption studies, effect of contact time plays vital role irrespective of the other experimental parameters effecting adsorption kinetics. The adsorption studies were carried out at different contact time as constant initial concentration of adsorbate with fixed dose of adsorbent (Table 5). It is observed that at initial stage, adsorption is rapid and becomes slow and gets stagnated with increase in time. Thus the first-order kinetic nature of adsorbate–adsorbent system is attributed to exponential absorption.

*Effect of initial concentration.* The effect in initial concentration of surfactant solution and amount of surfactant adsorbed on salary sand was undertaken with a fixed doses of adsorbent and contact time by varying the initial concentration

Table 6  
Variation of contact time

Concentration of solution, $C_0$ (ppm)	Amount $Y_2$ adsorbed $Q_e$		Equilibrium concentration, $C_e = C_0 - Q_e$ (ppm)		Amount $Z_2$ adsorbed $Q_e$		Equilibrium concentration, $C_e = C_0 - Q_e$ (ppm)	
	$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$
50	30	35	15	18	29	36	21	16
100	60	71	16	20	76	73	42	62
150	80	93	33	49	94	78	48	35
200	95	135	54	65	145	97	87	57
250	132	180	58	90	192	124	94	62
300	145	193	63	95	197	152	96	65

Table 7  
Variation of adsorbent—initial concentration: 300 ppm

Weight of adsorbent (g)	Amount $Y_2$ adsorbed, $Q_e$ (ppm)		Amount $Z_2$ adsorbed, $Q_e$ (ppm)	
	$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$
1	65	74	75	58
2	132	154	142	96
3	145	186	195	125
4	195	221	237	198
5	201	245	247	215

Table 8  
Variation of pH—initial concentration: 100 ppm

pH	Amount $Y_2$ adsorbed, $Q_e$ (ppm)		Amount $Z_2$ adsorbed, $Q_e$ (ppm)	
	$Hg^{2+}$	$Pb^{2+}$	$Hg^{2+}$	$Pb^{2+}$
2.80	22	30	32	24
3.06	24	33	34	26
4.04	25	36	35	28
5.12	28	38	37	30
5.88	32	42	43	32
6.90	35	46	47	34

of surfactant. This study reveals that amount of surfactant adsorbed exponentially increases while the percentage removal decreases with increase in initial concentration of surfactant, Table 6. This attributed to lack of available active sites on the adsorbent surface compared to relatively large number of active sites required for the high initial concentration of surfactant.

*Variation of adsorbent.* The variation of adsorbent and at fixed dose of adsorbate, with constant concentration of surfactant with optimum contact time, as the amount of adsorbent increased (Table 7). Also, adsorption increased may be attributed to increase in availability of the active sites due to this increase in surface area.

*Effect of pH.* This study was carried in the surfactant solution of 100 ppm, pH range of 2.8–6.9 adsorption of surfactant increases (Table 8). The increased adsorption of surfactant may be attributed to binding of surfactant molecules to the adsorbent (salary sand), whereas lower adsorption at lower pH may be attributed to the presence of excess hydrogen ion which lowers binding of surfactant molecules on sand.

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