

Preparation and Evaluation of Some Surface Active Sequestering Agents for Some Heavy Metals

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Abstract:

A novel series of chelating agents has been synthesized by the reaction of ethylenediaminetetraacetic acid, citric acid and polyethylene glycol with different molecular weights and different number of moles. The unique structural features of these surfactants have been confirmed by FTIR spectra, elemental analysis and ^1H NMR spectrum. These surfactants exhibit excellent properties in sequestering heavy metal such as copper, lead and mercury. They show good surfactant properties, including surface tension, effectiveness, efficiency and emulsifying power. Critical micelle concentration, maximum surface excess and minimum surface area have been studied. Free energy of micellization and adsorption have been calculated.

Key words:

Citric acid, EDTA, emulsifying power, polyethylene glycol, surface activity, multiple ionic chelates, anionic-nonionic hybrid series.

Introduction:

Toxic heavy metal in air, soil and water are global problems that are growing threat to the environment. There are hundreds of sources of heavy metal pollution, including the coal, natural gas, paper, mining, and chlor-alkali industries ^(1,2). Although there are many technologies for heavy metal remediation, the use of chemical chelates has only recently come under systematic investigation ⁽³⁾.

Mercury, lead and copper are three heavy metals that are abundant in the ecosystem of the earth and available largely due to anthropogenic sources. It has been estimated that mercury emissions globally exceed 3000 tons annually⁽⁴⁾. The element can be found in air, sediments, soils, sea water and fresh water. Effects of the high levels of mercury in blood and hair can be correlated with sensory disturbances (paresthesia, hypaesthesia), constriction of the visual field, hearing impairment, and neurological disturbance ⁽⁵⁾. Lead is toxic with various detrimental biological effects including inhibition of the synthesis of hemoglobin ⁽⁶⁾, dysfunction in the kidneys, reproductive system, liver, and the central and peripheral nervous systems^(7,8). Copper also can cause accute effects such as GL disturbances, damage to the liver and renal systems and anemia.

Accordingly, it has been the focus of our research to synthesize a series of chelate surfactants for heavy metals, which result soluble metal ligand complexes. These chelate surfactants appear to have high affinity for metals. These surfactants were synthesized by the reaction of ethylenediaminetetra acetic acid, (EDTA), citric acid and poly ethyleneglycol (400,600 or 1000) with different number of moles (1,2 or 3).

EXPERIMENTAL PROCEDURE:

Materials:

All the chemicals used throughout this investigation are submitted from international companies. All of them were in analytical grade as shown in Table 1.

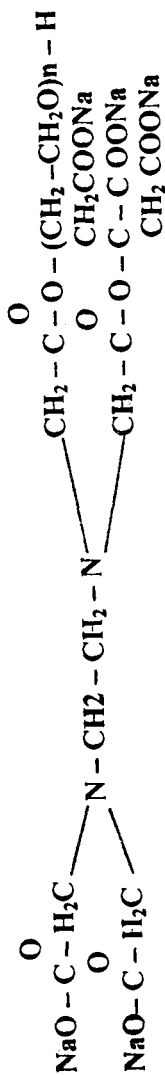
Table 1: Chemical used through out the investigation

Material	Molecular weight	m.p, °C	Source
EDTA	292	52	Hungary Co
Citric acid	192	152.154	Merck
Polyethelenglycol (PEG)*			
e.o. 9	400	6	Aldrich
e.o.13.6	600	22	Aldrich
e.o.22.7	1000	39	Aldrich

* where in, e.o = number of ethylene oxide units, which are coming from the corresponding M.wt of (PEG).

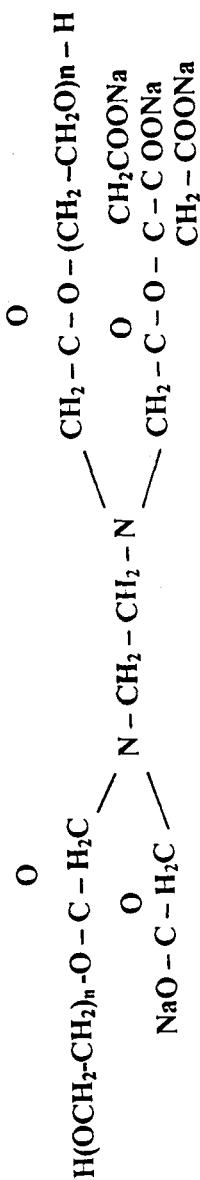
Synthetic procedure:

The chelate surfactants were prepared by stoichiometric amounts of reacting EDTA with citric acid in benzene in presence of hydrochloric acid as a catalyst (36.5%, 2mL) until the desired amount of water (0.1 mole, 1.8 ml) was removed. The removal of excess solvent was performed in rotary evaporator under reduced pressure. The compound obtained reacts with different molecular weight of PEG (400,600 or 1000) by different molar ratio (1:1, 1:2 or 1:3) followed by neutralization with NaOH to get three series of chelate surfactants. The purification of products was carried out by crystallization from ethanol. These ligands were designated by (I₁, I₂, I₃), (I₄, I₅, I₆) and (I₇, I₈, I₉).



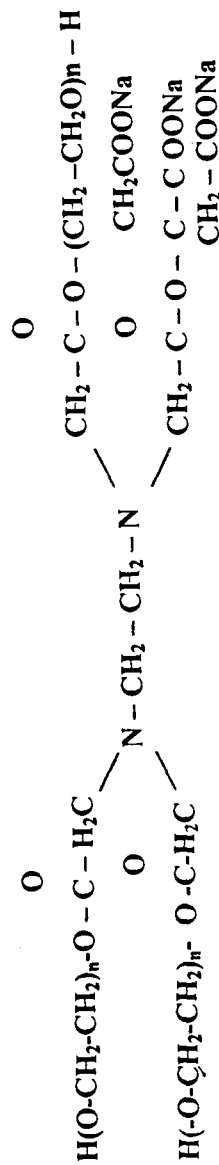
$n = 9, 13.6 \text{ or } 22.7$
(1:1 molar ratio)

I₁, I₄, I₇



$n = 9, 13.6 \text{ or } 22.7$
(1:2 molar ratio)

I₂, I₅, I₈



$n = 9, 13.6 \text{ or } 22.7$
(1:3 molar ratio)

I₃, I₆, I₉

Methods of Analysis and Instruments:

FTIR measurements:

Infrared spectra for prepared surfactants was measured using Avatar 230 FTIR spectrophotometer to measure intensity of absorption bands for the prepared surfactants. The measurements were carried out in Egyptian Petroleum Research Institute.

Elemental Analysis Measurements:

The elemental analysis for the obtained surfactants were carried out using Elemental Analyzer Model Varioelemenrar. The measurements were carried out in National Research Center.

Proton Nuclear Magnetic Resonance Measurements:

^1H NMR analyses were performed on a Varian-Gemini-200 instrument and the samples were run in deuterated chloroform (CDCl_3 , Cambridge Isotope Laboratories)

Atomic Absorption Spectrometer (AAS) measurements:

Copper, lead and mercury analyses were performed with ASS (Flame absorption) perkielmer, wave length 253.7nm and lamp current 6. The detection limits for these analysis are 0.02 mg/L for Cu, 0.007mg/L for lead and 0.0068mg/L for Hg.

Evaluation Methods of Surface Active Properties:

Surface and interfacial tension measurements:

Surface and interfacial tension measurements of the prepared surfactants were made at room temperature (25°C) with Du Nouy tensiometer (Kruss type 8451) using distilled water solution of 0.1% weight concentration⁽⁹⁾.

The surface tension of the used distilled water was 73 mN/m and the interfacial tension between paraffin oil and distilled water was 56.2 mN/m. Surfactant solution were aged for 2h before any measurements were made. Three readings were made on each sample to determine any change with time and to obtain an average value⁽¹⁰⁾.

Emulsifying power:

In a measuring cylinder, surfactant solution (0.1g/L, 10ml) and paraffin oil (10ml) were mixed. After vigorous shaking (20 times), the tube was allowed to stand till any separation of the two phases appeared. Emulsifying power or emulsifying time (in sec) was determined according to the method described in (11,12).

Efficiency (PC₂₀):

PC₂₀ value measures the efficiency of adsorption of the surfactant at the interface⁽¹³⁾ and it is measured by negative logarithm of the surfactant molar concentration (mol/L) required to reduce the surface tension of the solvent by 20 dyne/cm⁽¹⁴⁾.

Effectiveness (Π_{cmc}):

The surface tension " γ_0 " values at CMC were used to calculate values of surface pressure (effectiveness) from the following equation

$$\Pi_{cmc} = \gamma_0 - \gamma_s$$

where γ_0 is the surface tension measured for the pure water at the appropriate temperature and γ_s is the surface tension at CMC. The effectiveness of adsorption is an important factor to determine such properties of surfactant as foaming, wetting and emulsification, since tightly packed coherent interfacial films have very different interfacial properties than loosely packed, non coherent films⁽¹⁴⁾.

Determination of Critical Micelle concentration (CMC):

CMC of the prepared surfactant was determined by the surface tension method⁽¹⁵⁾. In this method values of the surface tension obtained for various concentrations of aqueous solutions of the prepared surfactants were plotted vs. of the corresponding concentrations

Maximum surface excess Γ_{\max} :

The surface excess concentration = surface concentration at surface saturation, the maximum surface excess Γ_{\max} is a useful measure of the effectiveness of adsorption of the surfactant at the water-air interface, since it is the maximum value to which adsorption can attain.

$$\Gamma_{\max} = \frac{1}{2.303 RT} \left(\frac{\delta\gamma}{\delta \log c} \right)_T$$

where $R = 8.314 \text{ Jmol}^{-1}\text{K}^{-1}$, T is absolute temperature, $(\delta\gamma / \delta \log C)$ is the slope of the γ vs. $\log C$ plot at room temperature⁽¹⁶⁾.

A substance which lowers the surface tension is thus present excess at or near the surface, i.e. when the surface tension decreases with increasing the activity of the surfactant, Γ is positive.

Minimum surface Area (A_{\min}):

A_{\min} is the minimum area per molecule of the prepared compounds at the interface was calculated from the following equation

$$A_{\min} = \frac{10^{18}}{\Gamma_{\max} N}$$

Where N is Avogadro's number and Γ_{\max} is the maximum surface excess.

The standard free energies of micellization $\Delta G_{\text{mic}}^{\circ}$ and adsorption $\Delta G_{\text{ads}}^{\circ}$:

Understanding the process of micellization and adsorption are important for explanation of the effects of structural and environmental factors on the

value of the CMC and for predicting the effects on it of new structural and environmental variations standard free energy of micellization ΔG°_{mic} , and adsorption ΔG°_{ads} , have played an important role in such understanding. The standard free energy of micellization and adsorption are given by:

$$\Delta G^{\circ}_{mic} = RT \ln(CMC)$$
$$\Delta G^{\circ}_{ads} = \Delta G^{\circ}_{mic} - 0.6023 \times \Pi_{CMC} A_{min}$$

Analytical procedures for AAS analyses:

Copper, lead and mercury:

The reactions were carried out using a stoichiometric molar amount of the reagent with solutions of copper, lead and mercury. Each of the chelates were added separately to a series of 100ml 50.00ppm (part-per-million) Cu^{+2} , Pb^{+2} and Hg^{+2} solutions. Aliquots (10) ml were collected at intervals of 1,6 and 20h following the addition of the chelates (tables 6-8) and (Fig. 4-12).

RESULTS AND DISCUSSION:

The prepared chelate surfactants were confirmed by different means. FTIR series spectrometer (Table 2), elemental analysis (Table 3), HNMR and mass spectroscopy. The results were found to be compatible with the supposed structure.

Results of FTIR data:

The chemical structures for surfactant samples were established according to FTIR (Table 2). The FTIR absorption spectra showed, generally absorption band around $3400cm^{-1}$ indicating for the presence of hydroxyl group, in addition to strong band at $1735cm^{-1}$ indicating for the presence of ester group in the structure of these surfactants. Also the spectra revealed absorption

bands at 1448 and 2800 cm^{-1} indicate the presence of carboxylate group and CH_2 respectively.

Results for elemental analysis data:

A further confirmation of the prepared surfactants is given by elemental analyses. The data of elemental analyses are presented in table 3. The data indicate that the calculated percentages of C, H and N are nearly close to the obtained measurements.

Results of proton nuclear magnetic resonance:

The ^1H NMR spectrum for some selective Chelate surfactants yielded:
 ^1H NMR (DMSO- d_6)

	$-\text{CH}_2-\text{COO}-$	$(\text{CH}_2-\text{CH}_2-\text{O}-)$	CH_2-OOC
I_3	2.5	3.5	3.9
I_5	2.5	3.5	4.3
I_6	2.5	3.5	4.2

Surface properties:

Surface and interfacial tensions:

Surface properties of the prepared chelate surfactants I_{1-9} were measured and tabulated in table 4.

The results in table (4) indicate that surface and interfacial tensions of the prepared chelate surfactants I_{1-3} , I_{4-6} and I_{7-9} decreased by increasing the number of moles of polyethylene glycol and decreasing the number of moles of anionic hydrophilic branch. This is because increasing of hydrophobic chain length and decreasing hydrophilic chain length leads to decrease of the aqueous solubility and migration of the surfactant from the bulk to the

interface⁽¹⁷⁾. The results in table (4) that indicate that (I₁,I₄, I₇), (I₂,I₅, I₈) and (I₃,I₆, I₉) at constant number of anionic hydrophilic branches, increasing the number of ethylene oxide units increases hydrophilicity, which increases solubility of the surfactant in water⁽¹⁸⁾. Such improved solubility lowers the tendency for surfactants to migrate from the bulk to the interface.

Emulsifying Power:

It is clear from table (4) that all prepared surfactants show adequate emulsifying power towards paraffin oil.

Critical micelle concentration:

At this concentration, the surfactant monomers begin to aggregate in the bulk phase forming a sort of cluster known as micelle. The amount of surface tension attained at this concentration is defined as γ_{cmc} . The values of both CMC and γ_{cmc} are collected in table 5 for anionic- nonionic hybrid series. These data indicate that the values of the prepared surfactants I₁₋₃, I₄₋₆ and I₇₋₉ decrease by replacement of anionic branch by nonionic branch, this is due to the fact that in aqueous medium ionic surfactants have much higher CMC than nonionic surfactant.

In polyoxyethylene chains with anionic characteristics of carboxylic group, as polyoxyethylene content increase, anionic properties decrease and materials become more like nonionics in nature. From table (5) (I_{1,4,7}, I_{2,5,8} and I_{3,6,9}), it is found that the CMC values increased by increasing the molecular weight polyethylene glycol (i.e. increase ethylene oxide units) at constant number of anionic branches, this is explained by The increasing number of ethylene oxide units increases the CMC.

Effectiveness " Γ_{cmc} ":

The most efficient surfactant is the one that gives the greatest lowering in surface tension for a critical micelle concentration (CMC). I₉ is the most found to be more efficient (Table 5) because it achieves the maximum reduction of the surface tension at CMC (Fig. 3).

Efficiency " P_{C20} ":

Values of efficiency of the prepared surfactants are shown in table (5), the efficiency increase by increasing molar ratio of polyethylene glycol and decreasing the number of anionic branches which is due to the fact that the replacement of anionic hydrophilic group by non ionic one results in a large increase in P_{C20} ⁽¹⁹⁾ as shown in table (5).

Maximum surface excess Γ_{max} :

Regarding to the results listed in table (5) from I₁₋₃, I₄₋₆ and I₇₋₉ by increasing the number of moles of polyethylene glycol and decreasing the number of hydrophilic anionic moles, Γ_{max} increases, this is contributed to the tendency of molecules to migrate to the water-air interface causing a consequent increase in Γ_{max} values.

From the results listed in table (5) (I₁, I₄, I₇), (I₂, I₅, I₈), (I₃, I₆, I₉), it is found that at constant number of hydrophilic anionic moles, increasing the number of ethylene oxide units leads to increase in Γ_{max} . This may be attributed to the fact that the molecule at higher ethylene oxide units tend to coil again which decrease minimum surface area and hence increase Γ_{max} .

Minimum area per molecule A_{\min} :

Results given in table (5) indicate that by consequence replacement of anionic hydrophilic moles leads to decrease in A_{\min} values. This is due to the rowdiness occurred at the interface as a result of increasing Γ_{\max} .

The standard free energies of micellization $\Delta G^{\circ}_{\text{mic}}$ and adsorption $\Delta G^{\circ}_{\text{ads}}$:

From table (5) values of $\Delta G^{\circ}_{\text{mic}}$ and $\Delta G^{\circ}_{\text{ads}}$ are always negative indicating the spontaneous behaviors of these two processes, but there is more increase in negativity of $\Delta G^{\circ}_{\text{ads}}$ rather than those of $\Delta G^{\circ}_{\text{mic}}$ indicating the tendency of the molecules to be adsorbed at the interface.

Results of Cu^{+2} , pb^{+2} and Hg^{+2} from AAS data:

AAS indicates that by increasing the number of moles of poly ethylene glycol the concentrations of the Cu^{+2} , Pb^{+2} and Hg^{+2} decreased in the solution. This is due to the fact that the replacement of the active hydrogen atom of carboxylic acid groups by oxyethylene units results in an increase in affinity of the chelate surfactant for metal ion⁽²⁰⁾.

The resulting complexes are conveniently solubilized in the micelles of anionic-nonionic hybrid surfactant⁽²¹⁾ and are thus separated from the bulk of aqueous phase. This replacement caused the decrease in CMC, surfactant of lower CMC act as good chelating agent, which is due to the fact that the chelations is very slight at concentrations below the CMC of the surfactant but rises abruptly once the CMC has been reached. This indicates that chelation is micellar phenomenon.

Maximum results, for Cu^{+2} and Pb^{+2} removal with I_9 , (Tables 6-8), (Figs.4-12) were seen at 1 hour at a neutral condition from 50ppm up to

10ppm. However, at 20 hours the results of I₉ at a neutral condition, indicate a reasonably high removal of mercury with a final concentration 1.01 ppm.

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Table (2) FT-IR analysis results for chelate surfactants I₁₋₉.

Surfactant	O-H	C-O-	CH ₂	O C ester
	3600-3200	1030- 1450	2830-2975	1735- 1750
I ₁	+	+	+	+
I ₂	+	+	+	+
I ₃	+	+	+	+
I ₄	+	+	+	+
I ₅	+	+	+	+
I ₆	+	+	+	+
I ₇	+	+	+	+
I ₈	+	+	+	+
I ₉	+	+	+	+

Table (3) Elemental analysis for the prepared surfactants I₁₋₉.

Surfactant	M.wt	C%		H%		N%	
		Calc.	Found	Calc.	Found	Calc.	Found
I ₁	958	42.59	42.74	5.53	5.64	2.91	2.94
I ₂	1318	47.34	47.54	6.82	6.92	2.11	2.14
I ₃	1678	50.06	50.12	7.57	7.68	1.67	1.69
I ₄	1158	44.77	44.92	6.17	6.20	2.41	2.45
I ₅	1718	49.17	49.38	7.38	7.50	1.63	1.68
I ₆	2278	51.41	51.51	7.99	8.31	1.23	1.26
I ₇	1558	47.29	47.70	6.91	7.22	1.79	1.84
I ₈	2518	50.89	50.12	7.92	8.11	1.11	1.15
I ₉	3478	52.50	52.70	8.38	8.50	0.80	1.12

Table (4) Surface properties of chelate surfactants I₁₋₉.

Surfactant	Ethylene oxide units (n)	Surface tension (mN/m)	Interfacial tension (mN/m)	Emulsifying power (min)
I ₁	9	42	20	24
I ₂	18	36	17	22
I ₃	27	32	18	23
I ₄	13.6	44	21	21
I ₅	27.2	38	20	18
I ₆	40.8	40	19	20
I ₇	22.7	44	22	18
I ₈	54.4	42	21	15
I ₉	68.1	41	20	16

Table (5): The critical micelle concentration (CMC) and surface parameters of the prepared surfactants I₁₋₉ at 25°C.

Surfactant	CMC x10 ⁻² (mol /L)	γ_{CMC} (dyne cm ⁻¹)	Π_{CMC} (dyne cm ⁻¹)	P _{C20} (mol/L)	$\Gamma_{max} \times 10^{-10}$ (mol/cm ²)	A _{min} (nm ²)	ΔG°_{mic} (KJ mol ⁻¹)	ΔG°_{ads} (KJ mol ⁻¹)
I ₁	0.1	49	22.8	3.0	0.060	2.60	-34.20	-70.70
I ₂	0.07	40	31.8	3.6	0.066	2.50	-35.60	-83.50
I ₃	0.05	44	27.8	3.5	0.069	2.40	-37.60	-77.78
I ₄	1.0	38	33.8	2.6	0.086	1.93	-22.79	-62.08
I ₅	0.8	31	40.8	3.3	0.100	1.59	-23.89	-62.22
I ₆	0.5	29	42.8	4.5	0.140	1.28	-26.22	-59.21
I ₇	1.2	30	41.8	3.4	0.110	1.40	-22.79	-60.30
I ₈	0.9	26.3	45.5	3.6	0.120	1.37	-23.31	-60.84
I ₉	0.6	25	46.8	4.3	0.146	1.27	-25.32	-61.11

Table (6) AAS Results of I₁₋₉ with Cu⁺²

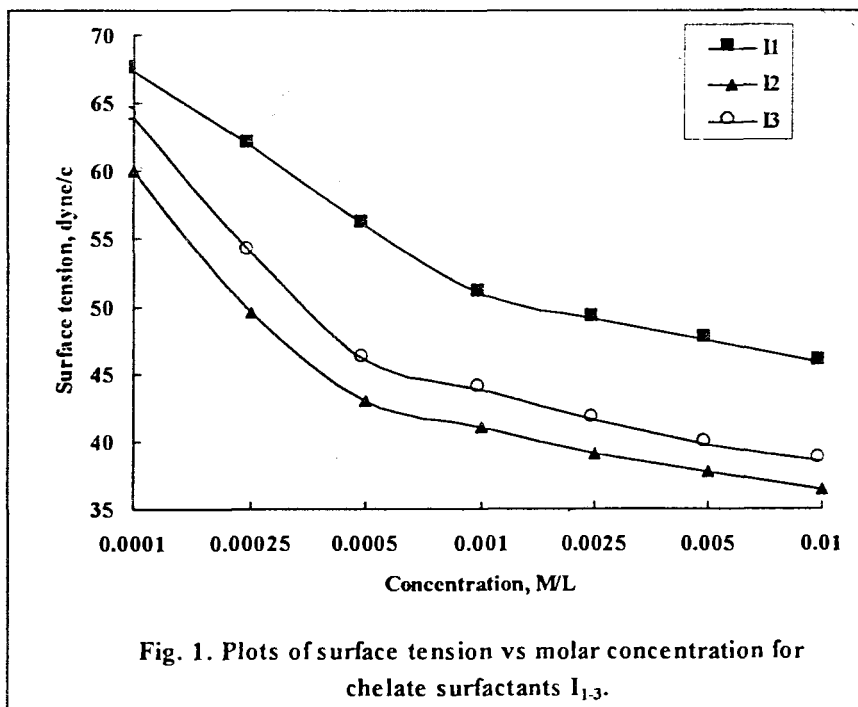
Chelating agent	Metal	Original metal conc. (PPM)	Ligand dosage (g)	Metal dose	Time the sample removal (h)	Final metal concentration (ppm)
I ₁	Copper	50	0.068	Stoichiometric	1 6 20	24 25 26
I ₂	Copper	50	0.098	Stoichiometric	1 6 20	13 16 17
I ₃	Copper	50	0.129	Stoichiometric	1 6 20	11 12 14
I ₄	Copper	50	0.083	Stoichiometric	1 6 20	21 25 29
I ₅	Copper	50	0.130	Stoichiometric	1 6 20	18 19 22
I ₆	Copper	50	0.177	Stoichiometric	1 6 20	9.5 11.5 14.0
I ₇	Copper	50	0.116	Stoichiometric	1 6 20	18.5 19.0 20.0
I ₈	Copper	50	0.160	Stoichiometric	1 6 20	13.6 14.0 15.0
I ₉	Copper	50	0.270	Stoichiometric	1 6 20	10.0 13.0 13.6

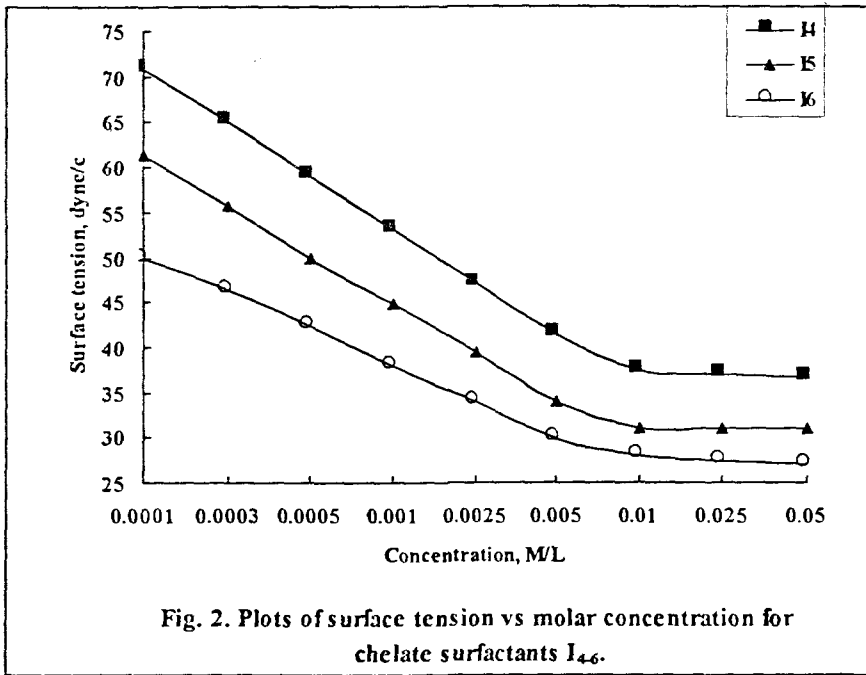
Table (7) AAS Results of I_{L_9} with pb^{+2}

Chelating agent	Metal	Original metal conc. (PPM)	Ligand dosage (g)	Metal dose	Time of sample removal	Final metal concentration (ppm)
I ₁	Lead	50	0.020	Stoichiometric	1	27
					6	29
					20	29
I ₂	Lead	50	0.029	Stoichiometric	1	25
					6	26
					20	28
I ₃	Lead	50	0.039	Stoichiometric	1	19.6
					6	20.8
					20	24.0
I ₄	Lead	50	0.025	Stoichiometric	1	21
					6	23
					20	23
I ₅	Lead	50	0.039	Stoichiometric	1	18.8
					6	20.9
					20	21.0
I ₆	Lead	50	0.053	Stoichiometric	1	16.4
					6	19.2
					20	19.8
I ₇	Lead	50	0.035	Stoichiometric	1	14.0
					6	16.8
					20	17.0
I ₈	Lead	50	0.049	Stoichiometric	1	12.0
					6	16.2
					20	16.2
I ₉	Lead	50	0.082	Stoichiometric	1	16.3
					6	10.9
					20	15.9

Table (8) AAS Results of I_{1.9} with Hg⁺²

Chelate agent	Metal	Original metal conc. (PPM)	Ligand dosage (g)	Metal dose	Time of sample removal	Final metal concentration (ppm)
I ₁	Mercury	50	0.02	Stoichiometric	1 6 20	21.09 19.79 17.38
I ₂	Mercury	50	0.031	Stoichiometric	1 6 20	16.87 13.62 11.40
I ₃	Mercury	50	0.04	Stoichiometric	1 6 20	10.52 8.03 7.01
I ₄	Mercury	50	0.026	Stoichiometric	1 6 20	17.14 13.49 11.27
I ₅	Mercury	50	0.091	Stoichiometric	1 6 20	13.20 10.70 8.55
I ₆	Mercury	50	0.059	Stoichiometric	1 6 20	6.18 4.03 2.41
I ₇	Mercury	50	0.036	Stoichiometric	1 6 20	5.94 4.93 3.90
I ₈	Mercury	50	0.06	Stoichiometric	1 6 20	4.31 2.97 2.03
I ₉	Mercury	50	0.085	Stoichiometric	1 6 20	3.20 1.90 1.01





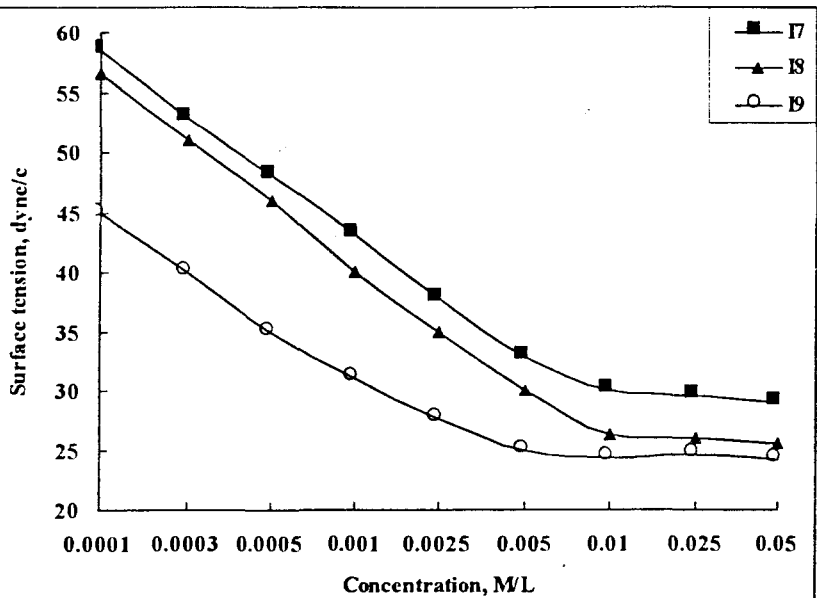


Fig. 3. Plots of surface tension vs molar concentration for chelate surfactants I_{7,9}.

