



Preparation of amide oxime type surfactants and the chelation of copper and nickel cations

M. E Wasel^{1*}, M. G Abd El-Wahed¹, A. A El-Sawy², W. I. A EL-DougDoug², M. S El-Attar¹

¹Chemistry Department, Faculty of science, Zagazig University, P.O. Box: 44519, Egypt

²Chemistry Department, Faculty of science, Banha University, P.O. Box: 13518, Egypt



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Abstract

In this research, we present a novel approach for removal heavy metals from contaminated water using surfactants of the amide oxime type. The synthesis process consisted of several steps. Initially, we esterified the fatty acids with PEG (polyethylene glycol) molecules of varying molecular weights (600 and 1500) to produce fatty acid methyl esters (FAMEs). Subsequently, the FAMEs were subjected to cyano esterification by reacting them with acrylonitrile, resulting in the formation of cyano ester products. To convert the cyano esters into amide oximes, we carried out an amidation step. During this process, the cyano esters underwent a reaction with hydroxylamine, leading to the formation of the desired amide oxime products. To confirm the chemical structure of the amide oxime, we employed various analytical techniques, including nuclear magnetic resonance (NMR) spectroscopy and infrared spectroscopy. To evaluate the effectiveness of the amide oxime in binding metal ions, we conducted batch adsorption experiments. The quantity of heavy metals removed was determined using atomic absorption spectroscopy (AAS), a widely employed technique for such measurements. Our results indicate that the synthesized amide oxime exhibited a high affinity for heavy metals, such as nickel (43%) and copper (46.6%) . This proposed methodology offers a sustainable and environmentally friendly solution for the removal of heavy metals from industrial wastewater.

Keywords: Type your keywords here, separated by semicolons ;

Introduction

With the rapid development of global industry and the emergence of new technologies [1], environmental pollution poses a major threat to human health; especially with significant amounts of heavy metal ions in the wastewater[2]. Heavy metals are toxic pollutants that pose a serious threat to the environment and human health[3]. The removal of heavy metals from industrial effluents is essential to prevent contamination of water resources.[4],[5]. Various methods have been developed to remove heavy metals from wastewater [6],but adsorption is considered a promising method because of its high efficiency, low cost and ease of use[7].Surfactants are widely used in various fields[8]. Due to their unique properties, surfactants are used in a wide range of fields, including detergents, cosmetics and pharmaceuticals[9]. Recently, researchers have focused on the use of surfactants to remove heavy metals from wastewater[10]. Amide oxime surfactants have shown great potential as effective adsorbents for heavy metal removal due to their high adsorption capacity and

selectivity [11]. Vegetable oil [12][14], a by-product in the production of the fatty acids[15][19], an abundant and renewable resource that can be used as a precursor for surfactant synthesis[20][22]. The synthesis of amide oxime surfactants[23][25] from vegetable oil has been reported in the literature[26], and these surfactants have shown excellent surface-active properties. In this study, we aim to synthesize amide oxime surfactants from vegetable oil waste and evaluate their performance as adsorbents for heavy metal removal from industrial wastewater.This study will provide valuable insights into the use of renewable resources for the development of effective and sustainable adsorbents for heavy metal removal

Experimental

Materials

From Al-Gomhuria Co. in Egypt, Myristic acid, palmitic acid, stearic acid, oleic acid, and linoleic acid (known collectively as fatty acids) were purchased alongside a range of other chemicals, such as hydroxylamine hydrochloride, potassium hydroxide

*Corresponding author e-mail: eldougDoug1@gmail.com (W. I. A EL-DougDoug).

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(98%, from Winlab), and acrylonitrile from Alpha. The solvents - methylene chloride, methanol, and xylene - were all acquired from Al-Gomhuria Co. in Egypt and were duly cleansed of any impurities. Aldrich's paraffin oil, with a dynamic viscosity range of 110-230 mPas and a density of 0.827-0.890 g/ml at 20°C, was also included in the mix. Distilled water was utilized for the measurement of surface properties. Chemicals from around the world were sourced for this project, with copper sulfate and nickel sulfate coming from Merck Germany, and acetic acid and sodium acetate sourced from Al-Gomhuria Co

Synthesis of esterified fatty acid nonionic surfactants

The esterified fatty nonionic amide oxime surfactants were synthesized from vegetable oil waste by the following steps

Synthesis of fatty acids monoester.

Utilizing a concoction of fatty acids, including Oleic acid, Linoleic acid, Stearic acid, and Palmitic acid, with 0.1 mol of Polyethylene glycol, consisting of 1500 and 600, in the presence of a catalyst comprised of 0.01% p-toluene sulphonic acid, we initiated a reflux with dry xylene as the solvent using a Dean-Stark trap, until calculated amount of water (0.1 mol) was separated. The reaction mixture was neutralized with sodium carbonate, washed twice with distilled water, and dried with anhydrous sodium sulphate. Distilling off the solvent followed this process [27]. The chemical structures of the synthesized monoesters were confirmed using FTIR [28].

Synthesis of fatty acids monoester acrylonitrile

Typical procedure. After metallic potassium (0.2 g, 5 mmol) was dissolved in monoether ethoxylated fatty acid ester (50 mmol), acrylonitrile (3.0 g, 57mmol) was dropped carefully at 30 °C and the mixture was stirred at this temperature for 24 hr. After neutralization by hydrochloric acid, water (100 ml) was added and the reaction mixture was extracted with methylene chloride (100 ml, 4 times). After the extracts were combined and dried with MgSO₄, the evaporation of the solvent gave crude product (12.0 g; crude nitrile). The gas liquid chromatography (GLC)-pure 4,7,10,13,16-pentaoxahexacosanenitrile was obtained by the Kugelrohr distillation (b.p., 150 °C/0.05 Torr; 10.9 g, 64% yield) [29].

Synthesis of fatty acids monoester amide oxime

Hydroxylamine hydrochloride (3.5 g, 51 mmole) in methanol (30 ml) was added to sodium hydroxide (2.11 g, 50 mmole) in methanol (30 ml) at 40 °C, and insoluble solids were separated off by filtration. To this filtrate (20 mmole) of polymer containing nitrile

in methanol (40 ml) was dropped and the mixture was stirred at room temperature for 24 h. (The pH of this mixture was ranged from 5-7 under these conditions). After filtration and subsequent evaporation of the filtrate, methylene chloride (50 ml) was added to the residue. Insoluble solids were filtered off again, and the crude product was obtained by the evaporation of filtrate [23], [30].

Surface properties of amide oxime

Surface Tension

The Surface tension (γ) of the freshly prepared nonionic amidoxime solutions with different concentrations were measured by De-Noüy ring Tensiometer (Kruss-K9). Surface tension of the used distilled water was measured before preparing the surfactant's solutions. The platinum ring was immersed in water than rinsed with acetone followed by briefly flaming to remove the remained acetone [31].

Measurements of Foaming Properties [32]

In practice and laboratory research, this test is widely used to characterize the foaming power of surfactant solutions and foam stability in the presence or absence of antifoam agents. In our experiment [33], some nonionic surfactant (usually 0.1% wt aqueous solution of surfactant) was mixed on a magnetic stirrer for 10 min for pre-emulsification. The resulting emulsion was homogenized several more times by hand before being placed in a glass beaker and circulated for 20 seconds. The foaminess of the solution is determined, for example, by the initial foam volume, i.e., That is, after the circulation of the aqueous surfactant solution has stopped. Dynamic bubbles are formed during the liquid, for example after 30 seconds of circulation, the height/volume is almost constant. It measures. Dynamic Foam Height (DFH), which is characterized by the static height of the dynamic foam and is used as a quantitative measure of the foamability of the drug. The stability of the foam is calculated by the following formula [34].

$$\text{Foam stability (\%)} = \left(\frac{\text{Foam volume after 5 mins}}{\text{Foam volume after 0 mins}} \right) * (100) \dots \dots \dots (1)$$

The foam volume was measured in mL and the foam stability was measured by the time at 28 °C [35].

Stability to hydrolysis

10 mL of surfactant solutions (10 mmole) and 10 mL of sodium hydroxide 0.05 N or 10 mL of sulfuric acid 2 N in phenol tube placed in a thermostat system at 40 °C. The surfactant resistance to hydrolysis was determined by the time of sample solution takes to be clouded [36].

Determination of the Cloud Point of the Prepared Nonionic Surfactants [37]

Nonionic surfactants Amide oxime are usually characterized by their cloud point. The cloud point (C.P.) is determined by heating of 1 wt% surfactant solution in deionized water until it becomes cloudy and then determining the temperature at which clear solution is obtained as the solution cools. These procedures were repeated at least three times, and the C.P. temperatures were determined with a reproducibility of ± 0.2 °C. Apparent cloud point (C.P.) values were given as a mean of three consecutive measurements [38]

Emulsifying Capability

To investigate the emulsifying capability, volumes of 5 mL with 2 % aqueous surfactant solution, respectively. The time of testing is from 0 to 24 h. Separation rate is calculated as the following formula: $R_s = V_s * 100/V_0$ (2)

where V_0 is the initial volume in mL of emulsion, R_s % is the separation rate percent, V_s is the volume of water in mL separated from the emulsion.

Hydrophilic–Lipophilic Balance (HLB) of Prepared Nonionic amide oxime Surfactants

To correlate the structure of surfactant molecules with their surface activity, Griffin developed the concept of hydrophilic–lipophilic balance (HLB)[39]. HLB values may be calculated for nonionic surfactants or may be determined experimentally. The experimental procedure is long and laborious and was described few years ago [40]. HLB values were calculated with the Griffin formula based on the ethylene oxide moiety in the molecule as follows

$$HLB = 20 * \frac{M_{WH}}{M_{WH} + M_{WL}} \dots \dots \dots (3)$$

where M_{WH} = Mol. W_t of the hydrophilic part and M_{WL} = Mol W_t of lipophilic part. Table 3 shows the HLB values calculated with the Griffin formula for the novel prepared surfactants. It is clear from Table 3 that HLB generally increases with increasing number of ethylene oxide units per surfactant molecule [41]

Gas-liquid chromatography (GLC)

Mixed fatty acid methyl ester GLC analysis was performed using Perkin Elmer Auto System XL utilized with a flame ionization detector (FID), a fused silica capillary column ZB-5 (60 m \times 0.32 mm I.D) and an oven which temperature was initially maintained at 150 °C with a pre-programmed gradual temperature increase from 150 to 240 °C by an increasing rate of 30 °C/min. The apparatus was operated with helium gas flow rate 1 mL/min and the temperatures of injector and detector were adjusted at 230 °C and 250 °C respectively. The split must be 1:10 and the size of the sample become 2 μ L. Identification of GLC peaks was carried out using chromatograms of standard fatty acid methyl esters (Sigma, USA), flavor

and odor lab (National Research Centre, Dokki, Cairo, Egypt)

Structural confirmation of the prepared compounds

FTIR Spectra

FTIR Spectra of the synthesized compounds was measured as liquid or solid in KBr disk on a thermo Nicolet iS10 FTIR spectrophotometer (Faculty of Science, Benha university, Benha, Egypt).

¹H NMR spectra

¹H NMR spectra was carried out by Bruker Avance (III) 400 MHz signal (Switzerland) with (128) scans at 298 °K in deuterated (DMSO-d₆) and/or in (CDCl₃-d) as a solvent and tetra methyl silane (TMS) as an internal reference

Preparation of Metal Ion Solutions

Solutions of Cu²⁺ and Ni²⁺ ions were prepared by dissolving an appropriate amount of each metal ion in certain volume of water to obtain .01M metal ion concentration. Initial and equilibrium concentrations of metal ions in the aqueous phase were determined

Batch adsorption

In case of removal experiments for a typical transition metal ions (Cu²⁺ and Ni²⁺), exactly 150 mg of the polymeric ligand was also immersed into metal ions (single metal) solution of 10 mL (0.01 M) to appropriate pH (6) using sodium acetate buffer (10 mL) and shaking for 2 h with the speed of 180 rpm. After equilibration, the ligand was separated by filtration and metal ions concentration were determined by atomic absorption. The initial and final readings (after adsorption) of the metal ion concentration were calculated according to eqn. (4).

$$q_e = (C_0 - C_e) V/L \dots \dots \dots (4)$$

Here, q_e is the equilibrium adsorption amount (mg g⁻¹), C_0 is the initial concentration of metal solution (mg L⁻¹), C_e is the equilibrium concentration of metal (mg/L) after adsorption determined using atomic absorption spectroscopy, V is the volume of metal solution (L), L is the mass of polymeric ligand (g)[42]

4. Result and discussion

Characterization

The FTIR spectra of Ethoxylated stearic acid ester.

The FT-IR spectra of polyethoxylated stearic acid confirm the expected functional groups in the synthesized ester. the broad characteristic absorption band appeared at about 3442 cm⁻¹ for O-H stretching vibration intermolecular hydrogen bonded due to the ethoxylated product. Two bands position at 2924, 2857 cm⁻¹ (ν C-H stretching aliphatic fatty chain) seemed to sharper this may be due to the increase in

Surface active properties

The study of the surface active properties of the ethoxylated compounds has been done in an aqueous solution (1wt %, pH = 7) at 25 °C[46]. The results are listed in Table 1.

Surface and interfacial tension

The surface and interfacial tension of the prepared of nonionic amide oxime surfactants are shown in (Table 1). It can be observed that the new nonionic amide oxime surfactants have pronounced surface activity. In general, the surface and interfacial tensions decrease with an increasing molecular weight of the hydrophilic moiety [47]. The data given in Table 2 shows that the values of surface and interfacial tension are decreased with the increasing number of polyethylene glycol added to the molecule. The results in Table 1.that indicate that increasing the number of ethylene oxide units increases hydrophilicity, which increases solubility of the surfactant in water[47]. Such improved solubility lowers the tendency for surfactants to migrate from the bulk to the interface.

Critical micelle concentration (CMC)

The relation between the surface tension and concentration of the synthesized surfactants at 30°C is represented in Fig. 6. The concentration ranges were chosen to cover wide range to describe all the expected changes in the behaviors of the surface tension of the surfactant solutions. It is clear that the surface tension concentration relations are characterized by distinguishable two regions, one at lower concentration range and characterized by a fast decrease in the surface tension value, the other at higher concentrations at which the surface tension value remain almost constant[23]. As polyoxyethylene content increase, anionic properties decrease and materials become more like nonionic in nature. From (Table 1) it is found that the CMC values increased by increasing the molecular weight polyethylene glycol (i.e. increase ethylene oxide units).

Effectiveness (π_{CMC})

Synthesized nonionic surfactants effectiveness can be estimated from the difference between the surface tension of the distilled water (γ_o) and the surface tension of the surfactant solution corresponding to the critical micelle concentration (γ_{CMC}) listed in (Table 1) based on the following equation.

$$\pi_{CMC} = \gamma_o - \gamma_{CMC} \dots\dots\dots (5)$$

Table 1 Surface properties of the synthesized surfactants

Property Surfactant	γ mN/m	π_{CMC} mN/m	Γ_{max} $\times 10^{-4}$ mol /cm ²	A_{min} $\times 10^{-7}$ (nm ²)	CMC	ΔG_{mic}
Fatty ethoxylated stearate (M.wt.1500)	41	31.86	7.8232	2.122	0.025	-20.69
Fatty ethoxylated oleate (M.wt.1500)	37.5	35.36	8.4378	1.967	0.0125	-24.58
ethoxylated fatty acid mixture (M.wt.1500)	40	32.86	5.9212	2.803	0.00625	-28.47

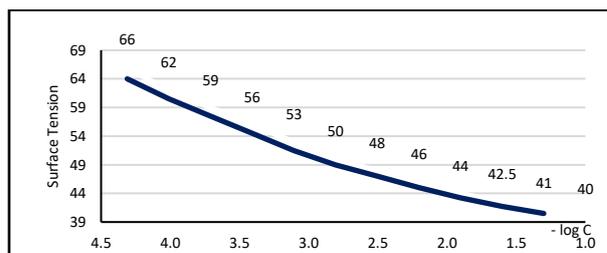


Fig. 6 Surface Tension V.S -log(conc.) of the synthesized surfactants (Mono stearate ester1500)

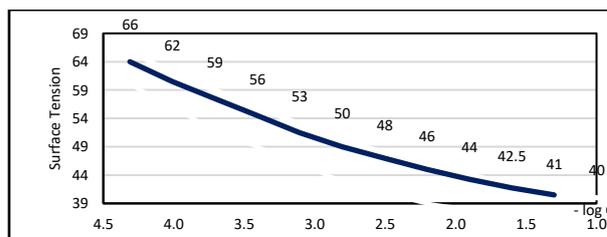


Fig. 7 Surface Tension V.S -log(conc.) of the synthesized surfactants (Mono oleate ester1500)

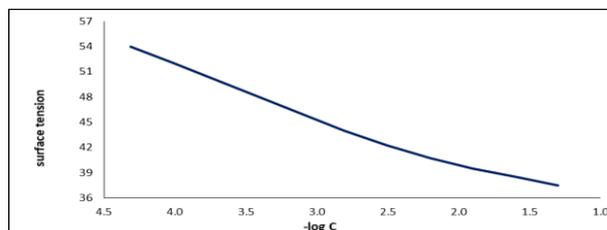


Fig. 8 Surface Tension V.S -log (conc.) of the synthesized surfactants (Mono fatty acid mixture)

Surface pressure (π_{cmc})

The surface pressure is adequate measuring criteria to evaluate the effectiveness of the surfactant with decreasing the surface tension of the water, π_{cmc} can be calculated as follows in (mN/m) based on the following equation:

$$\pi_{cmc} = \gamma_o - \gamma_{cmc} \dots\dots\dots (5)$$

Where γ_o is the distilled water surface tension and γ_{cmc} is the surfactant solution surface tension at CMC. The most efficient synthesized surfactant is one which gives the maximum decrease of the surface tension at the CMC [48].

Maximum surface excess (Γ_{\max}) and minimum surface area (A_{\min})

The maximum surface excess of the synthesized nonionic surfactant, Γ_{\max} , describes the accumulation of surfactant molecules at the air/water interface and can be calculated according to Gibb's equation [49].

$$\Gamma_{\max} = 1/2.303 nRT (\partial\gamma/\partial\log C) \dots\dots\dots (6)$$

Where, R= gas constant (8.314 J mol⁻¹ k⁻¹) and T= t+273 (°K); the value of n is the number of ionic species whose concentrated at the interface varies with the surfactant concentration in the solution. Γ_{\max} values of the synthesized nonionic surfactant were calculated at different temperatures and listed in Table (1). The average area occupied by each adsorbed molecule of a surfactant is given by the following Eq. (6) [50].

Emulsifying Power

It is clear from table (3) that all prepared surfactants show adequate emulsifying power towards paraffin oil explained. The increasing number of ethylene oxide units increases the CMC [51]. The synthesized nonionic surfactants showed moderated emulsification power for paraffin oil ranged between 234 minutes for (amidoxime ethoxylated stearic acid 600) and 55 minutes. for amide oxime ethoxylated stearic acid 1500 at room temperature (Table 2). As a general observation, increasing the number of the hydrophobic chains in the molecules increases the stability of the formed oil-in-water emulsion. The moderate emulsifying power is attributed to the polar nature of the nonionic surfactants with low hydrophobic chain contents[52]. On the other hand, increasing the hydrophobic chains increases the hydrophobicity of the molecules, which increases their tendency for migration from the aqueous medium to the oil medium and consequently increases their emulsifying power.

Foaming power

In general, the nonionic surfactants form unstable foams. The nonionic surfactants show foam stability smaller than the ionic and cationic surfactants in aqueous medium, due to large surface area per molecule and absence of highly charged films in these foams. The data of foaming power listed in (Table 2). In ethoxylated nonionic surfactants both foam stability and foam volume reach a maximum at a particular ethylene oxide [53][33]. This is due to maximum intermolecular cohesive forces in the adsorbed film as polyoxyethylene group increases.

Cloud point

The cloud point is a measure of the inverse solubility characteristic of nonionic surfactant. The energy of the hydrogen bond is insufficient to retain the surfactant molecules and the dehydration takes place when the

surfactant solution is heated at elevated temperature, and the most of surfactants molecules separate out with little amount of water. The surfactant solubility in water at certain temperature shows turbidity by heating. This turbidity is reversible, so that the solution clears again on cooling [54],[55]. The data presented in Table 2 reveal that, the cloud point increases with increasing polyoxyethylene groups[55].

Stability to Hydrolysis

All the prepared nonionic polymeric surfactants exhibited good stability as listed in Table 3, especially in an acidic medium but have lightly lower stability in a basic medium. The increase in lengths of both the saturated fatty alkyl chain and the polyoxyethylene chain lead to an increase in stability in both acidic and basic media [56][57].

Hydrophilic –Lipophilic Balance

The HLB values were calculated using the general formula of nonionic surfactants.

$$HLB = 20 * \frac{MWH}{MWH+MWL} \dots\dots\dots (3)$$

Where:

MW_H: formula weight of the hydrophilic portion of the surfactant molecule, MW_L: formula weight of the hydrophobic portion of the surfactant molecule. The hydrophilic-lipophilic balance (HLB) of a surfactant is a measure of degree to which it is hydrophilic or lipophilic. Griffin's method [43] for nonionic surfactants was used. The HLB value can be used to predict the surfactant properties of molecule as follow: a value 10: lipid soluble (water insoluble); and a value 10: water soluble. HLB values of the synthesized surfactants using Eq. 3 range between 7 and 16 which describe these compounds as water in oil emulsifiers.

Table 2 Surface active parameters of amide oxime nonionic surfactants at 30°C

Property	Surface tension (mN/m)		cloud point (°C)	Foam height (mm)	Foam stability (mm) 1.0 wt%			Emulsion stability (min: Sec) 1 wt%	Stability to hydrolysis (min: sec)		HLB
	0.1 wt.% at 30°C	0.1 wt.% at 30°C			0 min	3 min	5 min		Acidic (2.0N)	Alkaline (0.05N)	
	Surfactant		wt.%	wt.%							
			at 30°C	at 30°C							
amidoxime Ethoxylated Myristic acid 600	29.4	8.8	63	260	260	120	100	01:35	6:23 hr	10:35 hr	14.9
amidoxime Ethoxylated Palmitic acid 600	32.3	17.6	60	80	80	20	10	01:13	7:10 hr	10:30 hr	14.5
amidoxime Ethoxylated Stearic acid 600	38.2	8.8	55	100	100	20	15	03:54	7:25 hr	11:05 hr	14
amidoxime Ethoxylated Oleic acid 600	35.3	11.8	63	155	155	65	60	01:32	6:26 hr	10:34 hr	14.1
amidoxime Ethoxylated Linoleic acid 600	32.3	11.8	65	240	240	170	150	01:45	6:11 hr	10:22	14.1
amidoxime Ethoxylated Myristic acid 1500	23.5	8.8	>90	260	260	200	150	01:13	23:02 hr	48:22 hr	17.5
amidoxime Ethoxylated Palmitic acid 1500	38.2	5.9	>90	100	100	50	40	00:50	25:33 hr	60:25 hr	17.2
amidoxime Ethoxylated Stearic acid 1500	26.5	8.8	>90	250	250	190	170	00:55	26:10 hr	61:20 hr	16.9
amidoxime Ethoxylated Oleic acid 1500	33.8	5.9	>90	240	240	140	130	01:30	25:09 hr	55:34 hr	17
amidoxime Ethoxylated Linoleic acid 1500	26.5	8.8	>90	600	600	360	340	01:15	24:42 hr	50:15 hr	17

The removal of heavy metals via amidoxime surfactant

The analysis of samples of pure copper sulfate 0.01M and the other 10 ml copper sulfate 0.01M Containing amide oxime 0.03 gm dissolved in 10 ml of buffer sodium acetate showed the efficient of amide oxime nonionic surfactant toward Cu²⁺ and Ni²⁺ ions different from PEG 600 and 1500. And from fatty chain length and saturated and unsaturated. In case of PEG 600 for Cu²⁺ ions it was noted that the highest uptake processes amide oxime ethoxylated stearic acid 600 arrived 45.39% the saturated fatty chain more effective than unsaturated for Cu²⁺ ions complexation. The long saturated fatty chain (fatty ethoxylated (M.wt.600) Stearate amidoxime (45.39%) is more effective than short saturated fatty chain (Fatty Ethoxylated (M.wt.600) Myristate amidoxime (27.60%) and Fatty Ethoxylated (M.wt.600) palmitate amide oxime (36.03%). The mixture of saturated amide oxime (39.70%) (amide oxime ethoxylated myristic acid 600 & fatty ethoxylated (M.wt.600) palmitate amide oxime & fatty ethoxylated

(M.wt.600) stearate amide oxime) is more effective than the mixture of unsaturated amide oxime surfactant (33.50%) (fatty ethoxylated (M.wt.600) oleate amide oxime & fatty ethoxylated (M.wt.600) linoleate amide oxime) and mixture of all amide oximes surfactant (32.80%)

In case of PEG 1500 for Cu²⁺ ions it was noted that the highest uptake processes amide oxime ethoxylated Stearic acid 1500 arrived 46.60% the saturated fatty chain more effective than unsaturated for heavy metals complexation. The long saturated fatty chain (amide oxime ethoxylated stearic acid 600 45.39%) is more effective than short saturated fatty chain (amide oxime ethoxylated myristic acid 1500 25.40% and amide oxime ethoxylated palmitic acid 1500 19.03%). The chain which have more number of double bonds (amide oxime ethoxylated stearic acid 1500 (40.90%)) is more effective than that has less number of double bonds amide oxime ethoxylated oleic acid 1500 (30.90%). The mixture of saturated amide oxime 28.40% (amide oxime ethoxylated myristic acid 1500 & amide oxime ethoxylated palmitic acid 1500 & amide oxime ethoxylated stearic acid 1500) is more effective than the mixture of unsaturated amide oxime surfactant 19.60% (fatty ethoxylated (M.wt.1500) oleate amide oxime & fatty ethoxylated (M.wt.1500) linoleate amide oxime) and mixture of all amide oximes surfactant 19.30. As shown in (Table 3).

Table 3 The amide oxime (0.03 gm) compounds with copper sulfate solution 0.01M

Sample Name	Cu ²⁺ (Mg/L)	Perc. %	Sample Name	Cu ²⁺ (Mg/L)	Perc. %
Cu+2	630	100%		-	-
Fatty Ethoxylated (M.wt.600) Myristate amidoxime	456	27.60%	Fatty Ethoxylated (M.wt.1500) Myristate amidoxime	470	25.4%
Fatty Ethoxylated (M.wt.600) Palmitate amidoxime	403	36.03%	Fatty Ethoxylated (M.wt.1500) Palmitate amidoxime	510	19%
Fatty Ethoxylated (M.wt.600) Stearate amidoxime	344	45.39%	Fatty Ethoxylated (M.wt.1500) Stearate amidoxime	336	46.6%
Fatty Ethoxylated (M.wt.600) Oleate amidoxime	412	34.60%	Fatty Ethoxylated (M.wt.1500) Oleate amidoxime	435	30.9%
Fatty Ethoxylated (M.wt.600) Linoleate amidoxime	457	27.50%	Fatty Ethoxylated (M.wt.1500) Linoleate amidoxime	372	40.9%
Fatty Ethoxylated (M.wt.600) Unsaturated amidoxime	419	33.50%	Fatty Ethoxylated (M.wt.1500) Unsaturated amidoxime	506	19.6%

Fatty Ethoxylated (M.wt.600) Saturated amidoxime	380	39.70%	Fatty Ethoxylated (M.wt.1500) Saturated amidoxime	451	28.4%
Fatty Ethoxylated (M.wt.600) mixture amidoxime	423	32.80%	Fatty Ethoxylated (M.wt.1500) mixture amidoxime	508	19.3%

The analysis of samples of pure Nickel sulfate 0.01M and the other 10 ml Nickel sulfate 0.01M Containing amide oxime 0.03 gm dissolved in 10 ml of buffer sodium acetate showed in the (table 9) the efficient of amidoxime nonionic surfactant toward Ni²⁺ ions different from PEG 600 and 1500. And from fatty chain length and saturated and unsaturated.

In case of PEG 600 for Ni²⁺ ions. It was noted that the highest uptake processes Fatty ethoxylated (M.wt.600) palmitate amide oxime (49.2%) it is obvious that the saturated fatty chain of amide oxime surfactant more effective than unsaturated fatty chain of amide oxime surfactant for Ni²⁺ ions complexation. The chain which have a greater number of double bonds (Fatty Ethoxylated (M.wt.600) linoleate amide oxime (43.9 %) is more effective than that has a smaller number of double bonds amide oxime (fatty ethoxylated (M.wt.600) oleate amide oxime (31.3%)). The mixture of saturated amide oxime surfactant 600 (38.8%) and mixture of unsaturated amide oxime surfactant 600 (37.2%) nearly similar more than the mixture of all amide oxime surfactant 600(34.4%).

The analysis of amide oxime surfactant 1500 gave less values than corresponding amide oxime surfactant 600 with Ni²⁺ ions at all. It was noted that the highest uptake processes fatty ethoxylated (M.wt.1500) palmitate (43.3%) this mean the saturated amide oxime surfactant 1500 more effective than unsaturated fatty chain of amide oxime surfactant for Ni²⁺ ions complexation. The chain which have more number of double bonds (fatty ethoxylated (M.wt.1500) linoleate amide oxime (43.9 %) is more effective than that has less number of double bonds amide oxime (fatty ethoxylated (M.wt.1500) Oleate (31.3%)). The mixture of saturated amide oxime surfactant 1500 (31.14%) is more effective for chelation of Ni²⁺ ions than mixture of unsaturated amide oxime surfactant 1500(21.3%) and the mixture of all amidoxime surfactant 1500 (26.2%) as shown in (Table 4)

Table 3 The amide oxime (0.03 gm) compounds with copper sulfate solution 0.01M

Sample Name	Ni+2 (mg/l)	Percentage %	Sample Name	Ni+2 (Mg/L)	Percentage %
Ni+2	610	100%	-	-	-
Fatty Ethoxylated (M.wt.600) Myristate amidoxime	374.11	38.7	Fatty Ethoxylated (M.wt.1500) Myristate amidoxime	426	30.16
Fatty Ethoxylated (M.wt.600) Palmitate amidoxime	310	49.2	Fatty Ethoxylated (M.wt.1500) Palmitate amidoxime	346	43.3
Fatty Ethoxylated (M.wt.600) Stearate amidoxime	371.11	39.16	Fatty Ethoxylated (M.wt.1500) Stearate amidoxime	386	36.7
Fatty Ethoxylated (M.wt.600) Oleate amidoxime	419.1	31.3	Fatty Ethoxylated (M.wt.1500) Oleate amidoxime	420	31.1
Fatty Ethoxylated (M.wt.600) Linoleate amidoxime	342	43.9	Fatty Ethoxylated (M.wt.1500) Linoleate amidoxime	389.3	36.22
Fatty Ethoxylated (M.wt.600) Unsaturated amidoxime	373	38.8	Fatty Ethoxylated (M.wt.1500) Unsaturated amidoxime	480	21.3
Fatty Ethoxylated (M.wt.600) Saturated amidoxime	383	37.2	Fatty Ethoxylated (M.wt.1500) Saturated amidoxime	420	31.14
Fatty Ethoxylated (M.wt.600) mixture amidoxime	400	34.4	Fatty Ethoxylated (M.wt.1500) mixture amidoxime	450	26.2

Conclusion

Through the work about the surfactants of the amide oxime type, it may conclude that:

- 1-Preparation of amide oxime from vegetable oil refining waste.
- 2-The synthesized amide oxime surfactants have excellent surface-active properties and can effectively chelate copper and nickel cations, making them attractive candidates for various applications in fields such as catalysis and metal extraction.
- 3- the proposed methodology using amide oxime surfactants offers a sustainable and environmentally friendly solution for the removal of heavy metals from industrial wastewater.

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Declarations

Conflict of interest: All authors declare that, there is no competing of interests

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