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Studying the Effect of Potassium Amyl Xanthate Surfactant on Fe, Cu and U Ions for the Pretreatment of Abu Zeneima Sulphate Leach Liquor

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Abstract

In this study, batch experiments were performed to study the behavior of potassium amyl xanthate surfactant on Fe, Cu and U ions for the pretreatment of Abu Zeneima sulphate leach liquor. Various parameters, such as amount of potassium amyl xanthate, pH, stirring time, sample volume to potassium amyl xanthate mass (v/m ratio) and temperature were tested to determine the optimum conditions. The numerical results obtained support the experimental data on decreasing the iron and copper values where 0.1 g potassium amyl xanthate could remove 86.8 mg Fe and 0.98 mg Cu from the studied sulphate solution with no effect on uranium content. FTIR spectroscopy and XRF analysis were used for investigation.

Keywords

potassium amyl xanthate (KAX), iron, copper, Abu Zeneima sulphate leach liquor

1 Introduction

Most common surfactants (surface-active agents) are classified mostly into three sorts; 1. cationic surfactants as hexadecyl trimethyl ammonium bromide (HTAB), 2. anionic surfactants, for instance potassium ethyl xanthate (KEX), potassium amyl xanthate (KAX), and sodium dodecyl sulfate (SDS), and 3. nonionic surfactants as Saponin (biosurfactant) [1–3].

As mentioned in literature, iron and copper ions can be eliminated from solutions by different separation methods. One of these methods is the using of potassium diphenylmethyl xanthate or potassium hexyl xanthate (PHX) which have been used to decrease the iron, cobalt, copper, nickel, and zinc content [4, 5]. Sodium isobutyl xanthate (SIBX) has been used to decrease cadmium and some of heavy metals from phosphoric acid solution [6]. Copper was successfully decreased from the Tunisian wet process phosphoric acid using bentonite by adsorption technique [7]. Aminotrimethylene phosphonic acid (ATMPA) has also been used for precipitating the iron from acidic solutions [5]. Several chemicals were used to decrease the iron content by precipitation, including NaOH and KOH, while sodium oxalate was found to be a poor precipitating agent [5]. Also, iron (Fe^{3+}) was eliminated from phosphoric acid solution by adsorption on charcoal [7] as well as it can be removed from the hydrochloric acid solution using methyl isobutyl ketone and acetophenone [8]. Furthermore, iron can be eliminated using poly (acrylamide-acrylic acid) - titanium silicate (P (AMAA)-TS) composite adsorbent [9]. Iron and manganese were successfully removed using carbon nanotube filters [10].

The results of interaction of xanthate with ions have received significant research consideration for decades, however are still not utterly revealed [11]. Various other reagents would necessitate an accurate consideration of the selectivity of any reagent. Potassium amyl xanthate (KAX) and sodium isobutyl xanthate (SIBX) are conventional thiol anionic surfactants utilized in the bulk and are selective for nickel, iron, and copper specially from sulfide mineral ores [12]. Any challenge at substitution xanthate by based on molecular recognition phenomena and in this context quantum chemical calculations offer a possibility.

Abu Zeneima sulphate leach liquor was selected as the working study sample. By analyzing such solution for Fe, Cu and U it was found to attain about 12500 ppm, 140 ppm

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and 490 ppm, respectively. The principle aim of the present work is to decrease iron and copper ions from such sulphate leach liquor without interrupting the uranium content using a simple applicable precipitation technique. So, for achieving such target, batch experiments tests were used to explore the effect of Potassium Amyl Xanthate (KAX) on Fe, Cu and U recovery through optimization of the effective removal process parameters.

2 Materials and methods

2.1 Structure and properties of potassium amyl xanthate

Potassium amyl xanthate is an organosulfur compound with the chemical formula $CH_3(CH_2)_4OCS_2K$. Its IUPAC name is potassium O-pentylcarbonodithioate. KAX molar mass is 202.37 $g \cdot mol^{-1}$, while the density is 1.073 g/cm^3 [13]. It is a pale yellow powder or pellet with a pungent odor, soluble in water. It is widely used in the mining industry for the separation of ores.

2.2 Metal removal mechanism

Potassium amyl xanthate (KAX) is an anionic type which dissociates into K+ and AX- ions (Fig. 1). It seemed that the xanthate (KAX) acts as an ion exchanger in the respect that the potassium ion substitutes by the heavy metal ions and precipitates [5, 6, 14].

$$nKAX_{(aq)} + M^{n+} \rightarrow MAX_{(s)} + nK^{+} \quad (1)$$

Xanthate compound forms a colored metal-xanthate precipitates [15–17]. The stability of xanthate was found to increase with the amount and degree of branching of the hydrocarbon chain part. Regarding the choice of xanthate, the alkali salts like sodium and potassium were found most suitable because of their excellent solubility [14]. The formed precipitates can be separated from the solution by flotation and subsequent filtration [6].

2.3 Geology of the ore

The geological material under study represents a sample from Abu Zeneima mineralized in Um Bogma Formation. Um Bogma area is located in the southwestern Sinai, Egypt between longitudes 33° 18' and 33° 27' E and latitudes 28° 55' and 29° 04' N. The thickness of Um Bogma formation at type locality (Gabal Nukhul and wadi El Khaboba plateau) is 40 m, but it varies from 1 m to 67 m at other localities around Gabal Um Bogma plateau and decreases toward the east [18]. Um Bogma formation has economic importance, where the most radioactive anomalies in this area are recognized. It also includes the manganese-iron ores and the secondary copper mineralization. So, this formation can be defined as a polymineralized rock unit. This sample represents one of the uranium occurrences related to the dolostone of Um Bogma formation. The main mineral constituents of the dolostone faces are calcite, ankerite, kaolinite, limonite, goethite besides arsenopyrite, chalcopyrite and framboidal pyrite.

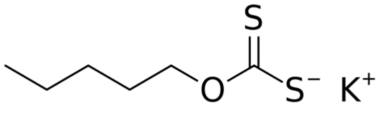

Fig. 1 Structure of potassium amyl xanthate (KAX)

2.4 Preparation of Abu Zeneima leach liquor uranium solution

A representative sample (60 g) of the provided ore material was treated with 180 mL of 30 % H2SO4 acid (Tedia, USA) and stirred in a glass beaker for about 8 hours at room temperature. Hydrogen peroxide (Poch, Poland) was also added to the mixture in order to oxidize all the divalent as Fe^{+2} and tetra valent ions as U(IV) found in the solution sample. The slurry was then left to settle and filtered.

2.5 Analytical procedures

2.5.1 Iron and copper analyses

Fe and Cu were determined through the experiments using Atomic Absorption Spectrometer model GBC 932-AAS. Features include: automatic wavelength and slit setting for maximum convenience and error-free. True double beam stability set-up. Protected optical system ensures long-term performance. Asymmetric modulation reduces noise. Flame atomization system. Specifications: Optics; single beam (933 Plus) or double beam (932 Plus) with background correction and flame emission capability. Monochromator: Ebert-Fastie design with 333 mm focal length and 185–900 nm wavelength range. Contin-uously adjustable slits with 0.2 to 2 nm spectral bandwidth. Lamp type: Plug-in mounting for hollow cathode lamp. Dimensions; 870 × 410 × 380 (WxDxH, mm). Weight: unpacked 48 kg, packed 75 kg. Electrical Requirements: 110/220/240 volts AC, 50/60 Hz, 300 VA.

2.5.2 Uranium analyses

Uranium analyses all over the study were carried out using a developed analytical method [19] by DX-500 Ion Chromatograph (Dionex Corporation, Sunnyvale, California, USA). The used DX-500 Ion Chromatograph is equipped with AD20 UV-Visible wavelength detector and GP40 gradient pump. Uranium is separated using an

IonPac CS5A analytical column and IonPac CG5A guard column and determined at wavelength 655 nm. The ion chromatography technique has indeed the ability to separate the ion before its determination and is then considered as indispensable tool in modern analytical chemistry [20].

All reagents used throughout were of analytical grade (AR). Solutions were prepared using nanopure water (18.2 MΩ-cm) obtained from a Thermo Scientific "Barnstead" lab water system (Thermo Fisher Scientific Inc., USA). The pH measurements were made on a digital Jenway pH meter (UK).

2.5.3 Characterization of the worked KAX surfactant

Fourier Transform Infrared Spectrometer (FTIR)

Fourier Transform Infrared Spectrometer model Thermo Scientific Nicolet IS10 (Germany) is used for investigating the functional groups reactivity of the KAX surfactant and KAX complexed with Fe and Cu ions.

EDXRF spectrometer

The Rigaku EDXRF spectrometer model NEX CG was used for investigating the worked Abu Zeneima leach liquor solution for Fe, Cu and U content. It is a non-destructive elemental analysis with specifications include: high resolution silicon drift detector (SDD) with core dimensions of 600 (W) × 400 (H) × 600 (D) mm which attributes 50 W, 50 kV X-ray tube (Pd target, air cooled), autosampler, He-flush. Power requirements: 1Ø, 100/220 VAC 50/60 Hz, 15/7 A. Analysis could occur in air, helium, or vacuum. Polarized excitation delivers lower detection limits.

2.6 Analysis of Abu Zeneima uraniferrous leach liquor

A representative sample of the provided Abu Zeneima uraniferrous ore material and the prepared sulfate leach liquor were analyzed for the major oxides, as well as for uranium and heavy metals (Table 1). For the former, the rapid analysis procedure of the silicate of Shapiro and Brannock [21] was used while for uranium and heavy metals, developed ion chromatography methods (which was found necessary to overcome the interference of the complex matrix) by Ahmed [19, 22] were utilized using Dionex (DX-500) Ion Chromatograph.

2.7 Experimental procedure and results evaluation

Several batch experiments were performed to study the different relevant removal factors. Except otherwise cited, each experiment was performed by stirring a certain amount of the KAX with certain of the prepared Abu Zeniema sulphate leach liquor at 25°C±1 for 60 minutes. The solution was then filtered, and the concentration of Fe and Cu were determined after equilibration.

The iron and copper removal efficiency (%) was calculated as the formula below [23]:

$$\text{Fe/Cu removal efficiency} = \left(C_i - C_t\right) / C_t \times 100\,, \qquad (2)$$

where C_i is the initial Fe/Cu concentration (mg L^{-1}), C_t is the residual Fe/Cu concentration (mg L^{-1})

3 Results and discussion

3.1 Characteristics of the worked KAX surfactant by FTIR

The removal efficiency of xanthate (KAX) depends on its functional groups reactivity. IR spectra of KAX surfactant and KAX complexed with Fe, Cu ions were shown in Fig. 2 and their interpretations were illustrated in Table 2.

Table 1 Chemical analyses of the major oxides together with the heavy metals and uranium in working Abu Zeneima sulfate leach liquor sample

Major elements (Oxides)	SiO_2	Al_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
Leach liquor sample (g/L)	59.20	23.70	4.70	0.25	2.07	2.10	2.35
Heavy metals & U	Fe	Cu	Ni	Zn	Cd	Mn	U
Leach liquor sample (ppm)	12.5 g/L	140	150	70	345	220	490

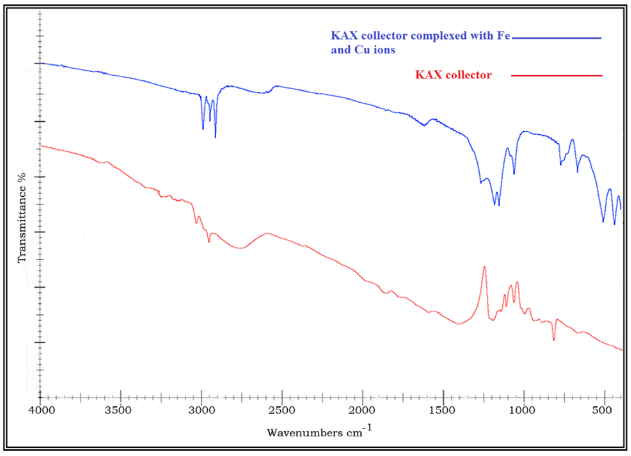

Fig. 2 FTIR spectra of KAX surfactant and KAX complexed with Fe and Cu ions

Table 2 IR stretching bands for KAX and [Fe(AX)3], [Cu(AX)2]

Complexes/Stretching bands	(C-H), cm^{-1}	(C-O-C), cm^{-1}	(C=S), cm^{-1}	(C-S), cm^{-1}
KAX	2952	1200-1050	997	827
$[Fe(AX)_3]$, $[Cu(AX)_2]$	2954	1259-1117	1030	808

In the spectrum of KAX surfactant, the characteristic absorption bands at 1200-1050, 997 and 827 cm^{-1} are assigned to the C−O−C stretching vibration, C=S stretching vibration, C−S stretching vibration respectively while the peak at 2952 cm^{-1} is attributed to the stretching vibration of C−H in the amyl group of KAX surfactant [24–26]. After the reaction of KAX with iron and copper ions presented in the leach liquor solution, the C−H, C−O−C, C=S and C−S peaks are shifted to 2954, 1259-1117, 1030 and 808 cm^{-1} respectively [26]. The C=S stretching vibration's position is related to the stability constant of the complex: the greater the stability constant, the lower the frequency [26]. The carbon-sulfur bonds therefore may change from C=S to C−S. This shift indicates that the sulfur C−S- is bonded chemically with Fe, Cu ions. Therefore, our hypothesis of the KAX's removal mechanism is reasonable which is further proved by XRF investigations as will be shown below.

3.2 Potassium amyl xanthate behavior

The behavior of the potassium amyl xanthate (KAX) surfactant on the removal of iron, copper and uranium from Abu Zeinema sulphate leach liquor was investigated. The studied parameters involved different factors, such as amount of potassium amyl xanthate, pH, stirring time, sample volume to KAX mass (v/m ratio) and temperature. The sample under study, Abu Zeinema leach liquor, contains about 12500 mg/L Fe, 140 mg/L Cu and 490 mg/L U.

3.2.1 Effect of potassium amyl xanthate amount

The effect of amount of potassium amyl xanthate on its removal of iron, copper and uranium was studied by stirring a fixed sample volume of 10 mL (125 mg Fe, 1.4 mg Cu and 4.9 mg U) at room temperature (25°C ± 1) and at pH of −1.2 ± 0.01 for 60 minutes using a range of amount of potassium amyl xanthate varying from 0.0125 to 1.0 g. From the obtained results shown in Fig. 3, it was observed that by increasing the potassium amyl xanthate amount, the efficiency of iron and copper removal has increased and reached a maximum of about 34.28 and 50.6 % for Fe and Cu which is equivalent to 42.85 and 0.708 mg at 0.1 g of KAX, respectively. Thereafter, the removal efficiency for both Fe and Cu has remained constant expressing that the working KAX has reached to its maximum capacity under such conditions. On the other hand, KAX recorded zero % removal efficiency for uranium ions present in the solution.

3.2.2 Effect of pH

One of the most important parameters that has a great effect on controlling the removal process of metal is the pH. In order to obtain the most favorable pH value for Abu Zeinema sulfate leach liquor, a number of several batch experiments tests were performed at different pH values ranging from −1.2 ± 0.01 to 1.6 ± 0.01 remaining the other conditions constant (potassium amyl xanthate amount of 0.1 g, stirring time of 60 minutes and sample volume of 10 mL attaining 125 mg Fe, 1.4 mg Cu and 4.9 mg U, with v/m ratio of 100 at a room temperature of 25°C ± 1). The pH was adjusted to the required value with NaOH solution. Fig. 4 shows the influence of the pH on the removal of Fe, Cu and U by KAX surfactant. It is clear from the latter that the removal efficiency increased by increasing the pH value till reached a value of 1.3 in case of Cu and 1.6 in case of Fe. After pH of 1.6 Fe and Cu ions precipitation occurs. Thus, at pH 1.6, the iron removal efficiency was 69.40 % (86.8 mg/0.1 g KAX) while that of copper at pH 1.3 was 70.5 % (0.98 mg/0.1 g KAX). For uranium ions, it is obvious from the figure that KAX behavior on the removal of such ions was zero efficiency.

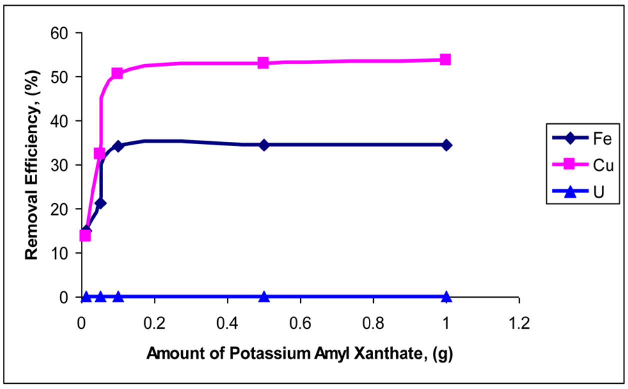

Fig. 3 Effect of KAX amount on its removal efficiency of iron, copper and uranium at pH = −1.2

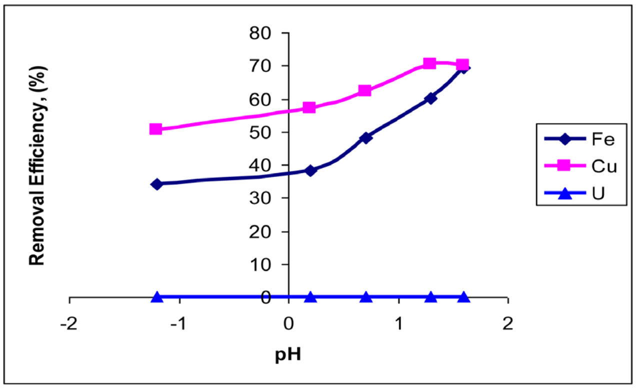

Fig. 4 Effect of sample pH on the removal efficiency of iron, copper and uranium

By overviewing the previous studies that explain the effect of pH on the removal process of iron and copper from solutions using xanthate, it was found that Haraldsen has stated in his US patent [6] that the solution is preferably neutralized to pH 1.6–1.8 before precipitation of the heavy metals from the phosphoric acid solution by flotation and subsequent filtration. Also, Naseeruddin [27] has been proved that iron (III) ions were precipitated using ethyl xanthate at pH 2.5 to 3.5. In addition, Sarvaramini et al. [28] have been proved that the removal process of iron and copper ions decreased with increasing the pH, at high pH value (in alkaline medium), the concentration of OH– group increased and hence the OH– group is reacted with $Fe(OH)_3$ and $Cu(OH)_2$ through formation of a covalent bond between its oxygen atom and iron or copper metals leading to the formation of $Fe(OH)_{-4}$ and $Cu(OH)_{-3}$ respectively, while xanthate is excluded from having an interaction with the metal hydroxide [28], it may be concluded that the interactions of $Fe(OH)_3$ or $Cu(OH)_2$ with OH– groups are more favorable than those with the xanthate group [28]. From all the latter studies, it can be concluded that the removal process of iron and copper ions from the Abu Zeneima sulphate leach liquor is favorable at low pH (in acidic medium) and the obtained data in the present study are in agreement with those obtained by the previous studies reported in the literature survey.

3.2.3 Effect of stirring time

Fig. 5 shows a plot of the removal efficiency of Fe, Cu and U versus the stirring time ranging from 10 to 120 min at a constant amount of KAX of 0.1g using two sample volumes of 10 mL (125 mg Fe, 1.4 mg Cu and 4.9 mg U) with pH of 1.6 ± 0.01 for Fe and 1.3 ± 0.01 for Cu at room temperature of 25°C ± 1 with v/m of 100. From these findings, it was found that the time required for maximum Fe and Cu removal is achieved within 60 min. and then become nearly constant. The maximum removal efficiency reached at that time was 69.4 and 70.5 corresponding to 86.8 mg Fe and 0.98 mg Cu respectively with nil removal efficiency for uranium ions.

3.2.4 Effect of sample volume to potassium amyl xanthate mass (v/m ratio)

The effect of sample volume to potassium amyl xanthate mass ratio (v/m) on removal efficiency of Fe, Cu and U from Abu Zeinema sulphate leach liquor was then studied. For this purpose, a series of experiments was performed using different volumes of two sample solutions 10–80 mL (corresponding to v/m ratio range of 100–800) assaying 12500 mg/L Fe at 1.6 ± 0.01 pH and 140 mg/L Cu at 1.3 ± 0.01 pH and 25°C ± 1 room temperature. Each of these solutions was stirred with 0.1 g KAX for 60 minutes. As illustrated in Fig. 6, the removal efficiency of both Fe and Cu ions decreased as the v/m ratio increased this may be interpreted that by increasing the solution volume the metal ion content increased while the amount of used KAX was kept constant at 0.1 g, therefore the metal ion removal efficiency decreased by increasing the metal ion content (by increasing the solution volume), so in order to increase the metal ion removal efficiency, KAX amount has to be increased to remove most of the metal ions present in the solution. 10 mL of the sample solution volume represents the optimum sample volume which expressed a minimizing efficiency of 69.4 % for Fe and 70.5 % for Cu. Accordingly, it can be concluded that the chelation capacity of the working KAX attains 86.8 mg/0.1 g KAX for Fe and 0.98 mg/0.1 g KAX for Cu. At the same time, it has been reported that by observing the removal efficiency of KAX for uranium ions as a factor of v/m ratio a straight line was obtained coincide with the x-axis recording 0 % result.

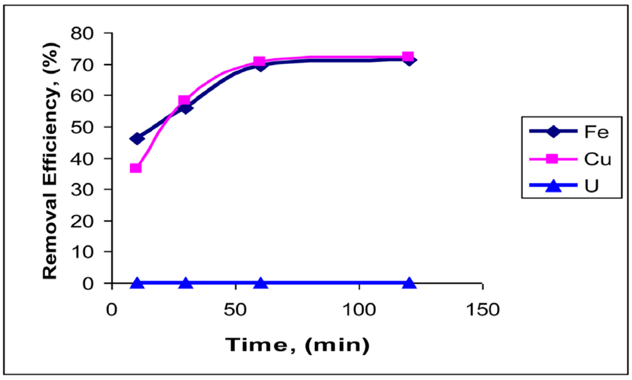

Fig. 5 Effect of stirring time on the removal efficiency of iron, copper and uranium

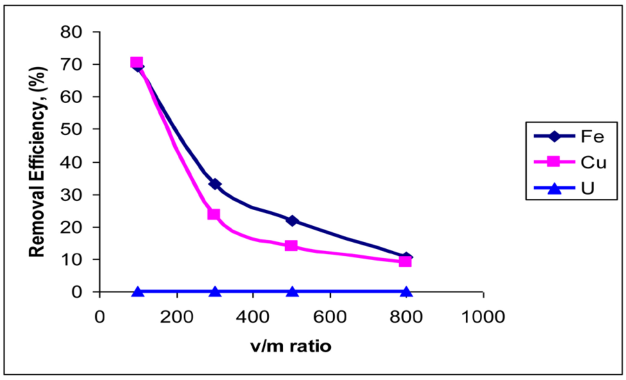

Fig. 6 Effect of sample volume to KAX mass (v/m ratio) on the removal efficiency of iron, copper, and uranium

3.2.5 Effect of temperature

The effect of temperature on the removal of Fe, Cu and U was studied from 25 to 80◦C ± 1. The operating conditions used involved a v/m ratio of 100, 1.6 and 1.3 ± 0.01 as solution pH for Fe and Cu respectively and for a stirring time of 60 minutes. From the obtained results reported in Fig. 7, it can be observed that the Fe and Cu removal efficiency has been decreased with the increase of temperature. Accordingly, the maximum efficiency uptake by potassium amyl xanthate is 69.4 % for Fe, 70.5 % for Cu and nil percent for U and the removal process is clearly favored at room temperature (25°C ± 1).

Summing up, it can be mentioned from the above data that stirring a volume of 10 mL Abu Zeinema leach liquor of 12500 mg/L concentration (125 mg) Fe and 140 mg/L concentration (1.4 mg) Cu at 1.6 and 1.3 ± 0.01 pH value respectively with 0.1 g potassium amyl xanthate for 60 minutes at the room temperature of 25◦C ± 1 will result in about 86.8 mg Fe and 0.98 mg Cu removal; a value which would be considered as an optimum value under these operating conditions. By analyzing this sample, it was found that the uranium concentration assaying 490 mg/L from which it was obvious that the potassium amyl xanthate surfactant has no effect on the removal of uranium ions keeping its content unchanged.

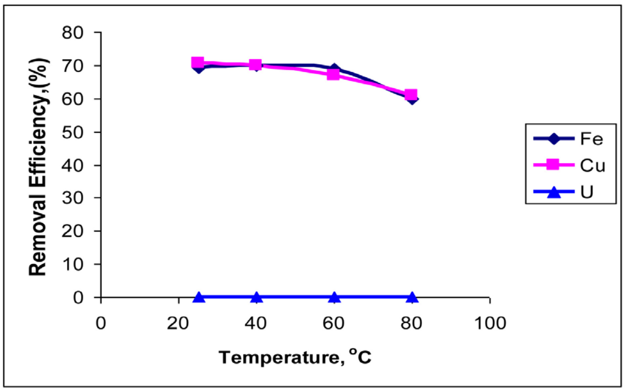

Fig. 7 Effect of temperature on the removal efficiency of iron, copper, and uranium

3.3 Characteristics of the worked KAX surfactant by EDXRF spectrometer

Fig. 8 shows the XRF spectra of potassium amyl xanthate (KAX) surfactant. As observed from Fig. 8 two huge peaks were appeared at about 2.3 keV and 3.3 keV representing sulphur (S) and potassium (K) which are the main constituents of the worked KAX surfactant.

Fig. 9 shows the XRF spectra intensity of the effect of KAX surfactant on Fe, Cu and U after pretreatment of Abu Zeniema sulphate leach liquor. As observed from the RX9 energy range, sulplur (S) huge peak still appeared as in Fig. 3 at 2.3 keV because it is not involved in complexation reaction of KAX with the sample ions. Concerning Cu energy range, the intensity of potassium (K) peak at 3.3 keV was observed to be decreased due to its substitution with Fe and Cu ions, as illustrated in peaks in both Cu and Mo energy ranges at nearly 6.4 keV for Fe ions and 8.1 keV for Cu ions.

Looking precisely at the four spectra, covering the RX9, Cu, Mo and Al energy ranges, it was found that there is no peak intensity observed representing U which proved that KAX surfactant has no effect on U ions present in the worked sulphate leach liquor, the matter which interpret why the worked potassium amyl xanthate surfactant removed Fe and Cu ions and not U ions from the sample solution.

Furthermore, it can be interpreted chemically according to Eq. (1) where KAX is an anionic group in the form $[n\,(AX)^{-}]$ so behaves as ion exchanger for cations present in the solution (the study Fe and Cu ions). For uranium ions present in the worked sulphate leach liquor solution, they are found as negatively charged uranyl sulfate complex in the form $[UO_2(SO_4)_3]^{-4}$. So, it is impossible that the anionic KAX be complexed with negatively charged uranyl sulphate complex.

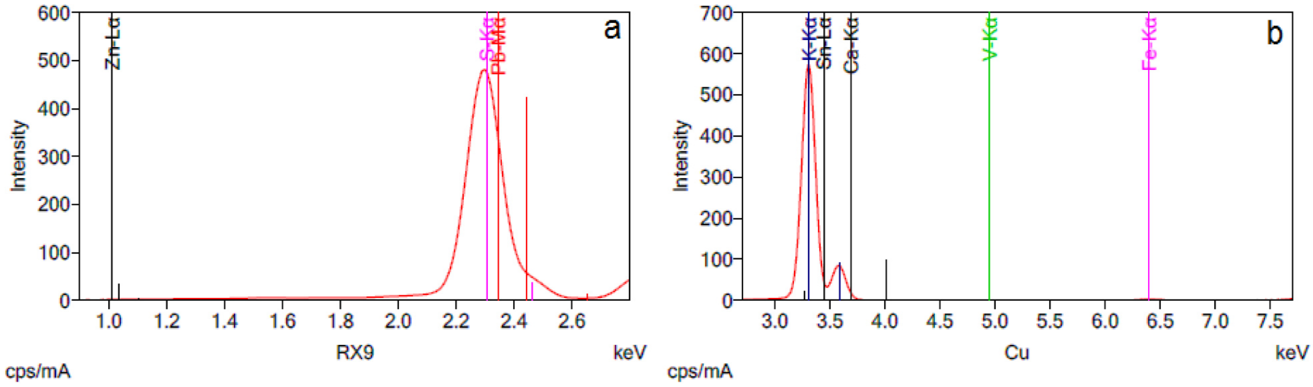

Fig. 8 XRF spectra of KAX before the treatment process

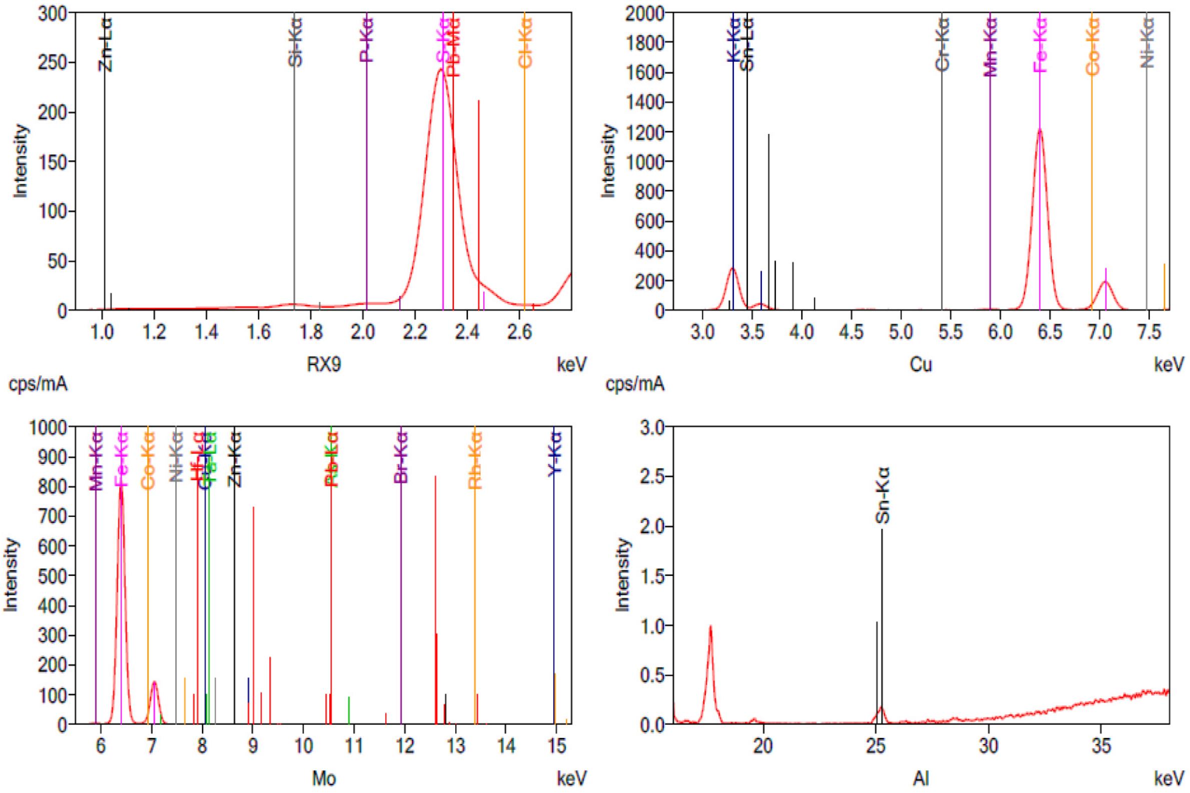

Fig. 9 XRF spectra of KAX after the treatment process

By this step, we finally reached our goal for this paper which deals with the pretreatment of Abu Zeinema sulphate leach liquor via removal of the main interfering ions in the studied solution (iron and copper) so the present uranium ions could be readily recovered.

4 Conclusion

A batch studies were conducted to determine optimum conditions for pretreatment of Abu Zeinema sulphate leach liquor. A variety of parameters was tested to establish the optimum conditions where the anionic potassium amyl xanthate (KAX) was used as a surfactant. It was observed that it is possible to separate iron and copper ions from the studied sulphate solution at pH 1.6 ± 0.01 for Fe and 1.3 ± 0.01 for Cu at room temperature of 25°C ± 1. The iron and copper removal efficiency were found as 69.4 % and 70.5 % respectively. Considering the ease of application and very high capacities achievable in the process, such results are very promising.

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