COMPARATIVE STUDIES ON FLOTATION OF KASOLITE USING CATIONIC AND ANIONIC SURFACTANTS

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Abstract

Low grade uranium ore was obtained from Abu Rusheid area, South Eastern Desert of Egypt The present work is concerned with the flotation studies which were performed towards pre-concentration of uranium mineral. In the studied sample two chelating agents were used as collectors for flotation of uranium bearing mineral in the sample, those are sodium lauryl sulfate (sodium dodecyl sulfate) (SLS) and cupferron (ammonium N-nitroso phenyl hydroxyl amine). Variables such as grain size, effect of pH, collector concentration and conditioning time were studied. The tests were done using hallimond tube. When finely ground sample (less than 62 µm) was floated using both (1×10^{-3}M) of cupferron at pH 6.5 for 120 sec. and (1×10^{-4}M) of sodium lauryl sulfate at pH 7.8 for 120 sec., the recovery of uranium was 91.29% and 79.11% respectively.

***Keywords**: Uranium, Flotation, Cupferron, Sodium lauryl sulfate.*

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1. INTRODUCTION

Uranium is an important element in view of industry, especially in the nuclear energy production. Mining, nuclear fuel preparation and waste management require number of chemical methods for purification, enrichment and its determination. The preconcentration techniques, hitherto developed for uranium (VI) are based on the physical, physico-chemical and chemical principles. These include liquid–liquid extraction, ion exchange, extraction chromatography, flotation, electro deposition and solid phase extraction [1, 2]. A pre-concentration of the ore by physical beneficiation method is expected to reduce the high acid consumption in leaching of the low grade ore and would also be helpful in safe disposal of tailings [3]. This work is concerned with the applicability of froth flotation of uranium mineral (Kasolite) from low grade uranium ore obtained from Abu Rushied area, South Eastern Desert, Egypt. Froth flotation is a highly versatile method for physically separating particles based on differences in the ability of air bubbles to selectively adhere to specific mineral surfaces in mineral/water slurry [4]. The basis of froth flotation is the difference in wettability's of different minerals. Particles range from those that are easily wettable by water (hydrophilic) to those that are water-repellent (hydrophobic). If a mixture of hydrophobic and hydrophilic particles are suspended in water, and air is bubbled through the suspension, then the hydrophobic particles will tend to attach to the air bubbles and float to the surface. The froth layer that forms on the surface will then be heavily loaded with the hydrophobic mineral, and can be removed as a separated product. The hydrophilic particles will have much less tendency to attach to air bubbles, and so it will remain in suspension and be flushed away [5]. There are numerous researches performed on the recovery of uranium using flotation technique. For instance, Tripathi et al., (2013) create a novel floating polymeric-magnetite cryobead for the recovery of hexavalent uranium from the aqueous sub-surfaces. The alginate-agarose-magnetite cryobeads were synthesized by the process of cryotropic-gelation at subzero-temperature [6].

2. MATERIALS AND METHODS

2.1 Ore Sample

The sample under study was obtained from Abu Rusheid area; South Eastern Desert. The sample was ground to less than 500 µm and physically treated with heavy liquids. Grains of yellow colors were separated from the heavy fractions by hand picking. These grains were then subjected to X-ray diffraction analysis (XRD, using Phillips PW-3710). The mineralogical composition of the studied sample was detected by X-ray diffraction (XRD, using Phillips PW-3710)

2.2 Collectors

In the present study two chelating collectors were used in froth flotation of uranium bearing mineral. A chelating collector's molecule may be considered to comprise two parts, viz. the hydrocarbon part and the functional chelating part. The chelating part is hydrophilic while the hydrocarbon part is hydrophobic. It is the chelating group that bonds with the cations in a mineral present on the wet host (ore) particle and thus attaches the collector molecule to the mineral by chemisorption. Otherwise the hydrophobic part made up of the carbon skeleton of the molecule does not participate directly in bonding with the minerals [7].

2.3 Instruments

Widely used methods for uranium determination in environmental samples are inductively coupled plasma-mass spectrometry (ICP-MS), neutron activation analysis (NAA), gamma and alpha spectrometry [8-10] and electro analysis using ion selective electrode [11-14]. In this work the uranium is determined using an Inductively Coupled Plasma Optical Emission Spectrometer (Prism ICP, Teledyne technologies) due to its high sensitivity and low detection limit.

2.4 Calculations

Uranium recovery (R_{U3O8}%): It is the percentage of uranium metal in the original feed that is recovered in the concentrate. This was calculated from the Eq. (1) using weights and assays as follows:

$$R_{U3O8}\ \% = (C_c)/(F_f) \cdot 100 \qquad (1)$$

Where F and C are the % weights of the feed, and concentrate respectively; and f and c are the assays of the feed and concentrate.

The Selectivity Index was calculated from the Eq. (2) as follows [15]:

$$\text{Selectivity Index } (\eta) = \sqrt{\frac{U_3O_8(\%)\, float}{U_3O_8(\%) tails}} \qquad (2)$$

2.5 Chemicals and Reagents

Extra pure sodium lauryl sulfate (SLS) obtained from MERCK in powder form and analytical grade cupferron obtained from SIGMA-ALDRICH were used as collectors for flotation of uranium ore. Hydrochloric acid obtained from BDH (37%), nitric acid (69-72%) obtained from Panreac, sulfuric acid (97-99%) obtained from ADWIC and hydrofluoric acid obtained from Ubichem were used for complete dissolution of froth and sink products , Laboratory grade sulphuric acid and sodium hydroxide were used for maintaining pH of the pulp.

2.6. Methods

The flotation process was performed under different conditions summarized in Table 1, to achieve the best recovery of uranium mineral. The bench scale flotation experiments were performed using hallimond tube. For this purpose 3 gram of the sample (less than 62 µm) was conditioned in 120 ml of collector solution for 10 min. and allowed to be floated .The froth was skimmed from the top of the hallimond tube to a beaker by the aid of a clean glass plate. At the end of the test, the froth and sink products were dried, weighed and analyzed using an Inductively Coupled Plasma Optical Emission Spectrometer (Prism ICP, Teledyne technologies).

Table -1: Studied factors affecting the flotation of uranium bearing minerals

Factors	Factors' values		Fixed conditions							
	Cupferron	SLS	Grain size(µm)		pH		Collator conc. (M)		Time (sec.)	
			Cupferron	SLS	Cupferron	SLS	Cupferron	SLS	Cupferron	SLS
Grain size	-62, -88, -149 µm	-62, -88, -149 µm	-	-	6.5	7.8	(1×10^{-3})	(1×10^{-4})	120	120
pH	2, 4.5, 6.5, 8	2.5, 5, 7.8, 10.5	-62	-62	-	-	(1×10^{-3})	(1×10^{-4})	120	120
Collector conc.	(1×10^{-2}), (1×10^{-3}), (1×10^{-4}) M	(1×10^{-2}), (1×10^{-3}), (1×10^{-4}) M	-62	-62	6.5	7.8	-	-	120	120
Time	30, 60, 90, 120 sec.	30, 60, 90, 120 sec.	-62	-62	6.5	7.8	(1×10^{-3})	(1×10^{-4})	-	-

3. RESULTS AND DISCUSSION

3.1. Mineralogy of the Sample

The presence of radioactive mineral kasolite $Pb(UO_2)[SiO_4].H_2O$_was indicated by X-ray spectra and compared with the ASTM card no. (8-0297) when yellow color grains were subjected to X-ray diffraction analysis. The X-ray spectrum of uranium bearing mineral is shown in fig. (1). The x-ray spectrum of the studied sample is shown in fig. (2). The sample contain many minerals in major portions, those are Montmorillonite ($Na_{0.2}Ca_{0.1}Al_2Si_4O_{10}(OH)_2(H_2O)_{10}$, quartz ($SiO_2$) and kaolinite ($Al_2Si_2O_5(OH)_4$).The chemical analyses of the studied sample (less than 500 µm) are given in table (2). The sample used for the studies was all passing below 500 microns. It assayed 0.0825 % U_3O_8. The granulometry of different size fractions of the sample is shown in fig. (3). Uranium concentration of different size fractions of the sample is shown in table (3). The distribution of uranium oxide in different size fraction is shown in fig. (4). From fig. (4), it appears that the greatest concentration of uranium is present in the fine size ranges.

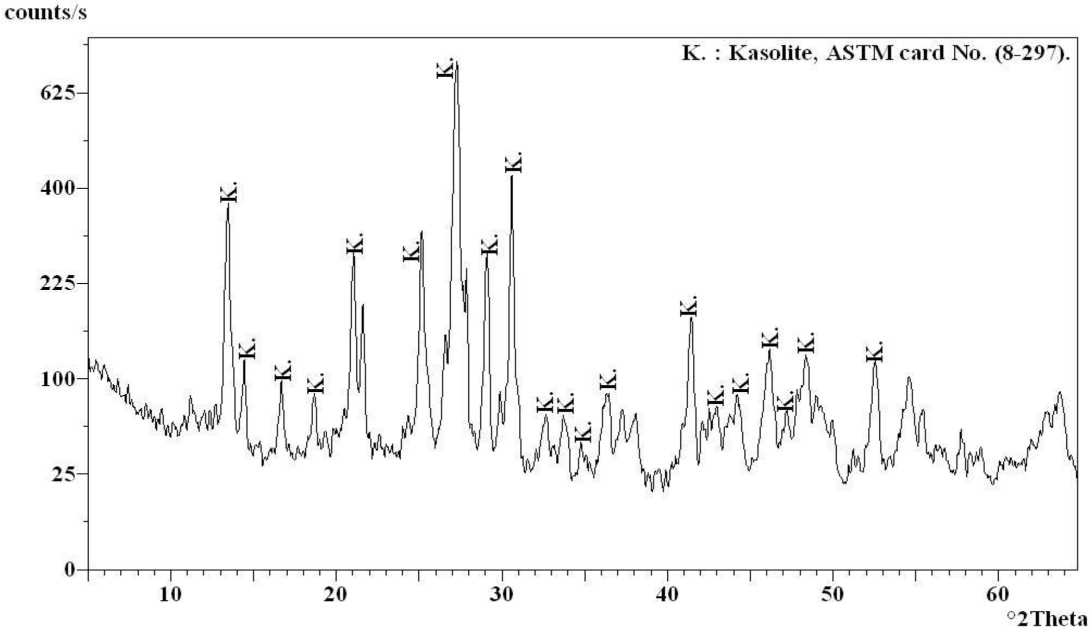

Fig -1: X-ray diffraction of uranium bearing mineral.

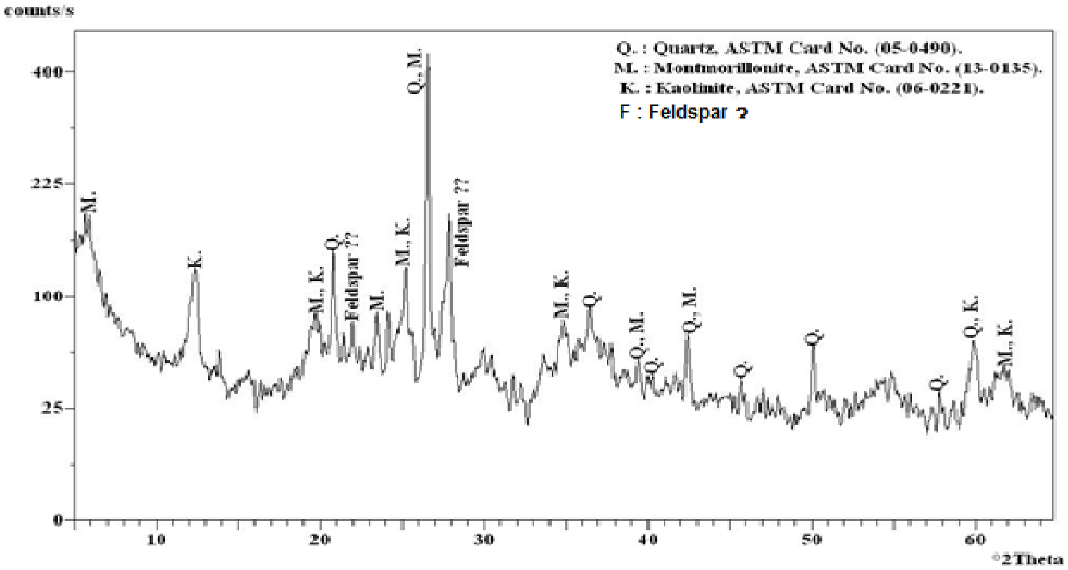

Fig -2: X-ray diffraction analysis of the studied sample.

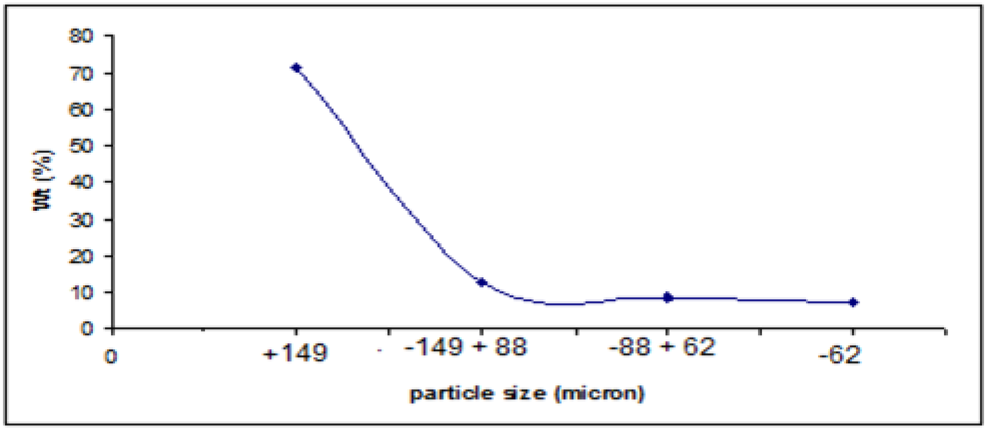

Fig -3: The granulometry of different size fractions of the sample.

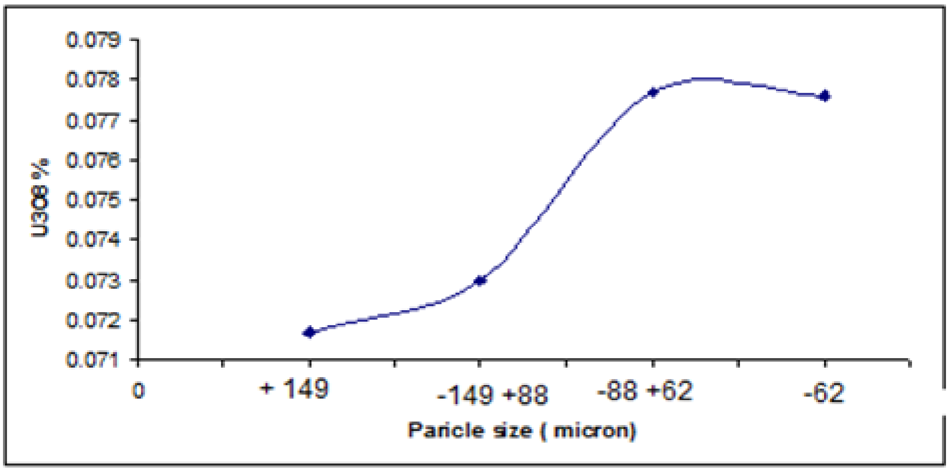

Fig -4: The distribution of uranium oxide in different size fraction.

Table -2: chemical composition of the selected ore sample

MAJOR OXIDES		TRACE ELEMENTS		REES	
OXIDE	WT. (%)	ELEMENT	CONC. (PPM)	ELEMENTS	CONC. (PPM)
SiO_2	48.3	Co	348.9	La	113.11
TiO_2	1.12	Zn	14415	Nd	3128.9
Al_2O_3	17.3	Zr	465.15	Sm	130.00
Fe_2O_3	14.8	Cu	823.00	Gd	82.070
CaO	2.50	Pb	2853.5	Dy	82.500
MgO	7.95	Ga	222.50	Ho	42.500
MnO	0.53	Ni	122.16	Er	45400
NaO_2	1.40	V	1617.0	Lu	88.360
K_2O	2.40	Cr	18.700		
P_2O_5	1.70	Nb	31.00		
L.O.I	1.39	Rb	115.00		
TOTAL	99.4	Sr	1.80		
		Y	36.00		
		Ba	1806		

Table -3: Grain size (Wt. %) and uranium concentration of 1461 gm. of the sample

Grain size	Wt. of grain size (gm.)	Wt. %	Conc. of U (ppm)
+149 µm	1041.2	71.26	608.15
-149 + 88 µm	186.8	12.79	619.85
-88 + 62 µm	125.4	8.58	659.75
-62 µm	107.6	7.37	658.15
Original sample	1461	100	700

Two collectors sodium lauryl sulfate (SLS) and cupferron were used for flotation of uranium bearing mineral in the present study. The effects of different factors on the recovery of uranium and the selectivity index were studied in details as follows:

3.2. Effect of Grain Size

Increasing the fineness of the feed and hence a decrease in the particle size was found to be effective in increasing the recovery of uranium bearing mineral and also give higher value of selectivity index using both cupferron and sodium lauryl sulfate (SLS). Increasing the fineness of the feed from (less than 149 µm) to (less than 62 µm) increased the recovery of uranium from 58.73% to 91.29% in case of cupferron and from 57.20 % to 79.11 % in case of sodium lauryl sulfate. It also increases the selectivity index from 0.99 to 1.43 in case of cupferron and from 1.01 to 1.19 in case of sodium lauryl sulfate. The increase in fineness of the feed gives higher recovery. This may be attributed to the increase in surface area of the particle and also the action of collector will be increased. As the fineness increased, the ability of the air bubbles to carry the particle on its surface will be increased. This is in agreement with Fuerstenau, (1980) [16] who found that the maximum recovery was noticed in the range 10-100 µm; he also suggested that chemisorption improved fines recovery for the following reasons:

a) Specific chemical interactions maintain selectivity.

b) The free energy of adsorption is large, resulting in lower reagent consumption;

c) The electrical charge on the mineral is lower, resulting in better recovery if collector adsorption occurs by exchange or neutral molecule adsorption; and

d) The reduced adsorption of collector on bubble surfaces contributes to better recovery of fines. The results are shown in Figs (5, 6). This set of experiments was conducted using collector concentration of (1×10^{-3})M and (1×10^{-4})M in case of cupferron and sodium lauryl sulfate respectively, for 120 sec. in case of both cupferron and sodium lauryl sulfate and at pH 6.5 and 7.8 in case of cupferron and sodium lauryl sulfate respectively.

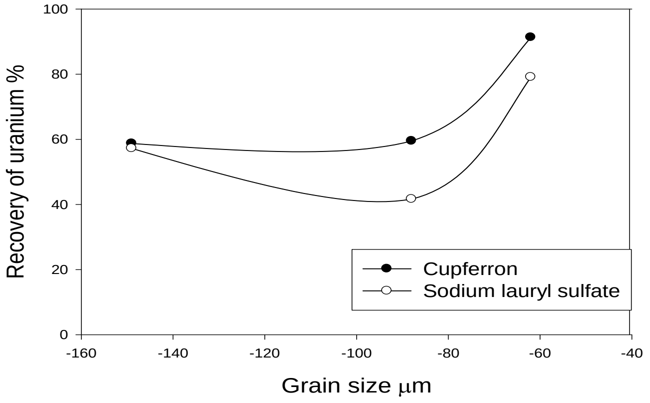

Fig -5: Relation between Grain size µm and recovery of uranium % using cupferron concentration (1×10^{-3}) M at pH 6.5 for 120 sec. and SLS concentration (1×10^{-4}) M at pH 7.8 for 120 sec.

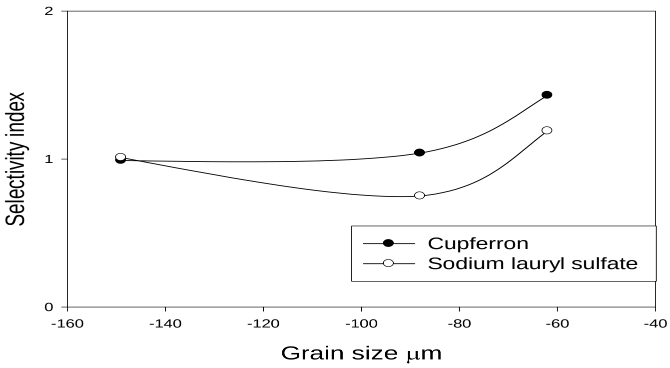

Fig -6: Relation between Grain size µm and the selectivity index using cupferron concentration (1×10^{-3}) M at pH 6.5 for 120 sec. and SLS concentration (1×10^{-4}) M at pH 7.8 for 120 sec.

3.3. Effect of pH

pH has a great effect on the surface chemistry of most minerals. pH also plays an important role in the adsorption of the flotation reagents and hence floatability of minerals. Two sets of experiments were performed using the ground ore (less than 62 µm) in case of both cupferron and sodium lauryl sulfate, using collector concentration (1×10^{-3}) M and (1×10^{-4}) M in case of cupferron and sodium lauryl sulfate respectively and for 120 sec. in case of both cupferron and sodium lauryl sulfate.

Figs (7, 8) reveal that at pH 6.5, the recovery of uranium and the selectivity index give the highest values .The values of the recovery of uranium increased from 78.82 % at pH 4.5 to 91.29 % at pH 6.5, on the other hand that value decreased at pH 2 and 8 in case of cupferron while in case of sodium lauryl sulfate at pH 7.8, the recovery of uranium and the selectivity index give the highest values. The values of the recovery of uranium increased from 64.21 % at pH 2.5 to 79.11 % at pH 7.8, on the other hand that value decreased at pH 5. As shown in figs (7, 8) the values of the selectivity index increased from 1.14 at pH 4.5 to 1.43 at pH 6.5, on the other hand that value decreased at pH 2 and pH 8, the best recovery of uranium was found to be between pH 4.5 and 6.5 in case of cupferron and this is similar to that observed by Muthuswami, et. al. (1983) who found that good recovery of uranium is between pH 4 and 6 and referred to the formation of complex between cupferron and uranium ion in the reduced state (U^{4+}) in a strongly acid medium [17-19], while in case of sodium lauryl sulfate the values of the selectivity index increased from 1.03 at pH 2.5 to 1.19 at pH 7.8, on the other hand that value decreased at pH 5. This means that the best recovery of uranium was found to be between pH 7.8 and 10.5. This may be attributed to the chemical composition of kasolite mineral, which is containing lead. It may be suggested that sodium lauryl sulfate attaches to kasolite mineral through lead and brings it to the froth. This is in agreement with Alessandra Gorette de Morais, et.al (2013) who found that the great recovery of lead was obtained by flotation at pH 10 with only (1×10^{-3}) M sodium lauryl sulfate and poor selectivity was observed under alkaline conditions resulting in low grade concentrate [20].

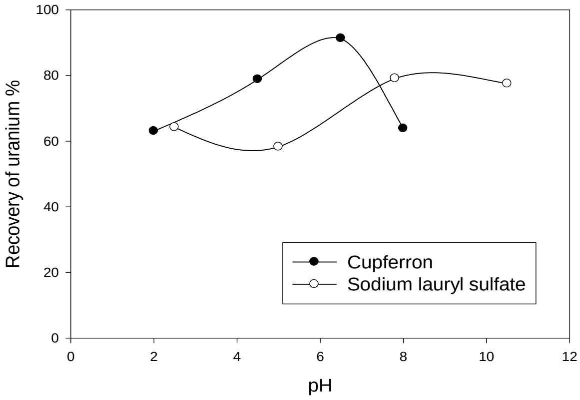

Fig -7: Relation between pH and recovery of uranium % using cupferron concentration (1×10^{-3}) M, on the ground ore (less than 62 µm) for 120 sec. and SLS concentration of (1×10^{-4}) M, on the ground ore (less than 62 µm) for 120 sec.

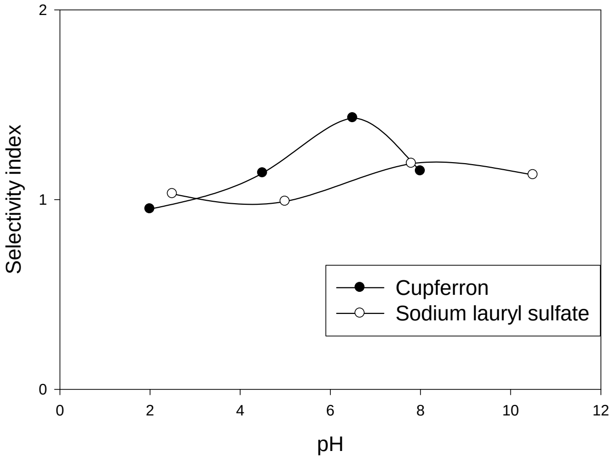

Fig – 8: Relation between pH and the selectivity index using cupferron concentration (1×10^{-3}) M, on the ground ore (less than 62 µm) for 120 sec. and SLS concentration of (1×10^{-4}) M, on the ground ore (less than 62 µm) for 120 sec.

3.4. Effect of Collector Concentration

Collector concentration is important from the view of commercial application. As the collector concentration decreased, the amount of reagent needed will be decreased and this is commercially desirable. Two sets of experiments were performed using the ground ore (less than 62 µm) in case of both cupferron and sodium lauryl sulfate, at pH 6.5 and 7.8 in case of cupferron and sodium lauryl sulfate respectively and for 120 sec. in case of both cupferron and sodium lauryl sulfate.

As shown in Figs (9, 10) the recovery of uranium and the selectivity index give the highest values at cupferron concentration of (1×10^{-3}) M. On the other hand, the recovery of uranium decreased at concentration of (1×10^{-2})M and (1×10^{-4}) M in case of cupferron while in case of sodium lauryl sulfate the recovery of uranium and the selectivity index give the highest values at sodium lauryl sulfate concentration of (1×10^{-4}) M.

From Figs (9, 10), it can be observed that at lower as well as at higher concentrations, the selectivity index, an indicator of separation efficiency, varied between 1.16 and 0.73 in case of cupferron. This may be illustrated as following:

At lower concentration, the amount of cupferron adsorbed on uranium bearing mineral would have been insufficient to impart flotation effect and at higher concentrations, the selectivity would have been lost due to masking of uranium bearing minerals by excessive cupferron concentration than what was necessary for floating uranium bearing mineral. The optimum concentration was found to be (1 × 10^{-3}) M which would have been just sufficient enough to preferentially form a coating / layer to float uranium bearing mineral and to give better selectivity as proven by the selectivity index and the recovery values. Singh et.al., found that 0.312 kg/t of cupferron is not sufficient and an increase dosage to 0.625 kg/t improves uranium recovery but a further increase in cupferron dosage showed a decline in uranium recovery [21]. On the other hand, the values of the recovery of uranium and the selectivity index decreased at higher concentrations to be 61.83 % and 1.03 respectively in case of sodium lauryl sulfate. This decreasing in the values of the recovery of uranium and the selectivity index exhibited at higher concentration of collector may be attributed to the nature of the froth as was observed during the performance of the experiments; bubbles were very small and not very persistent to keep the heavier particles of uranium bearing mineral at its surface.

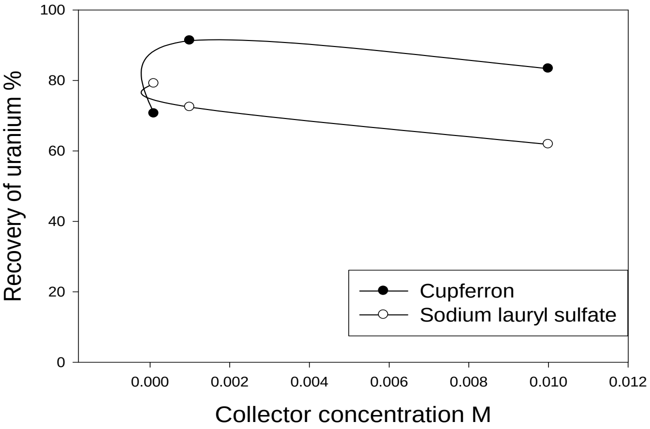

Fig -9: Relation between collector concentration(M) and recovery of uranium % using cupferron at pH 6.5, on the ground ore (less than 62 µm) for 120 sec. and SLS at pH 7.8, on the ground ore (less than 62 µm) for 120 sec.

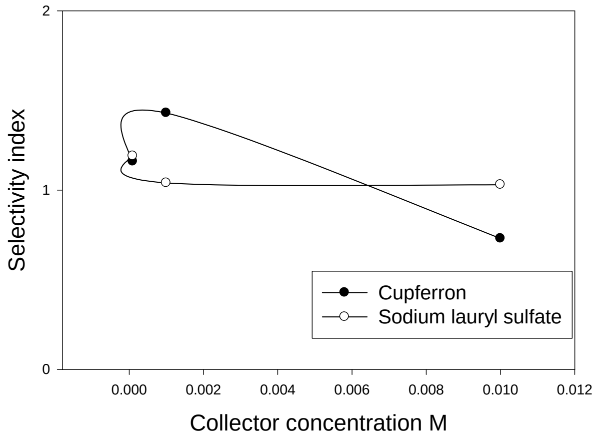

Fig -10: Relation between collector concentration (M) and selectivity index using cupferron at pH 6.5, on the ground ore (less than 62 µm) for 120 sec. and SLS at pH 7.8, on the ground ore (less than 62 µm) for 120 sec.

3.5. Effect of Time

Two sets of experiments were performed using the ground ore (less than 62 µm) in case of both cupferron and sodium lauryl sulfate, at concentration of (1×10^{-3}) M and (1×10^{-4}) M in case of cupferron and sodium lauryl sulfate respectively, at pH 6.5 and 7.8 in case of cupferron and sodium lauryl sulfate respectively. As shown in Figs (11, 12), the recovery of uranium and the selectivity index give the highest values at time 120 sec. for both cupferron and sodium lauryl sulfate. This may be attributed to the fact that increasing time will increase the chance of the particles to react with the reagent and hence increase the floatability of uranium bearing mineral.

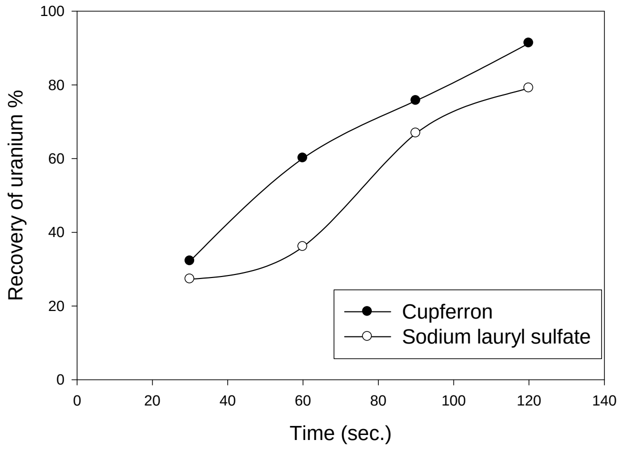

Fig -11: Relation between time (sec.) and recovery of uranium % using cupferron concentration (1×10^{-3}) M at pH 6.5, on the ground ore (less than 62 µm) and SLS concentration (1×10^{-4}) M at pH 7.8, on the ground ore (less than 62 µm).

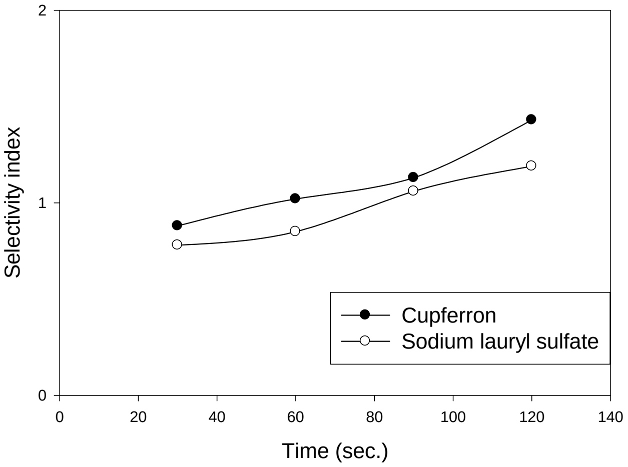

Fig -12: Relation between time (sec.) and the selectivity index using cupferron concentration (1×10$^{-3)}$ M at pH 6.5, on the ground ore (less than 62 µm) and SLS concentration (1×10^{-4}) M at pH 7.8, on the ground ore (less than 62 µm).

4. CONCLUSIONS

Flotation studies were performed on the ore sample to obtain the highest recovery of uranium bearing mineral. The sample contains 0.0825 % U_3O_8.The flotation study was applied using two kinds of collector, one of them is cationic and the other is anionic. The fine size (less than 62 µm) was found to be the desirable size of uranium recovery for both cupferron and sodium lauryl sulfate. The slightly acidic and slightly alkaline media was found to be the desirable media for cupferron and for sodium lauryl sulfate respectively. The concentration of (1×10^{-3}) M and (1×10^{-4}) M was found to be the desirable concentration of cupferron and sodium lauryl sulfate respectively. The preferable time for the process was 120 sec. for both cupferron and sodium lauryl sulfate. Under these optimum conditions the recovery of uranium reaches 91.29 % and 79.11 % for cupferron and sodium lauryl sulfate respectively. For commercial applications sodium lauryl sulfate will be preferable due to its low price compared with cupferron which is not economical flotation reagent.

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