ABSTRACT

This paper deals with the results of flotation studies carried towards pre-concentration of a low-grade uranium ore sample from Domiasiat, Meghalaya. A combination of potassium amyl xanthate, cupferron and light diesel oil was used as collector for flotation of uranium bearing minerals in the sample. The effects of various process parameters were studied. Under the optimum conditions the flotation concentrate weighed about 35 % with over 90 % recovery. Flotation with classified/deslimed feed also resulted in similar metallurgical results. Use of diethyl hexyl phosphoric acid, alamine 336 and tri-butyl phosphate in combination with potassium amyl xanthate and light diesel oil, as the alternate reagent scheme, also gave reasonably high uranium recovery.

INTRODUCTION

The uranium is the basic raw material for a nuclear energy programme. In India, uranium ore is processed essentially by hydrometallurgical route. The conventional process involves sulphuric acid leaching of uranium from the ore, ion-exchange concentration followed by precipitation of uranium as magnesium diuranate. 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 [1].

In Meghalaya occurrences of sandstone type uranium mineralisation is reported from Domiasiat. An economic exploitation of this deposit is desirable. At the instance of M/s Uranium Corporation of India Ltd., Jaduguda froth flotation studies were carried out on a low-grade uranium ore sample from Domiasiat, Meghalaya. It was envisaged that froth flotation would be helpful in pre-concentration of the ore reducing the load on downstream hydrometallurgical steps. This paper presents the salient results on the flotation of the low-grade uranium ore sample from Domiasiat, Meghalaya.

MATERIALS AND METHODS

Ore Sample

The sample used for the studies was all passing below 1680 microns. It assayed 0.052% U$_3$O$_8$. The granulometry and the uranium distribution in different size fractions of the sample is shown in Fig. 1.

Reagents

Commercial grade potassium amyl xanthate (KAX) and analytical grade cupferron from E. Merck (India) Ltd., Mumbai with light diesel oil (LDO) were used as collector for flotation of uranium ore. Laboratory grade methyl-iso-butyl carbinol (MIBC) was used as frother while commercial grade sodium silicate (SS) was used as depressant/dispersant for siliceous gangues. Laboratory grade sulphuric acid and sodium hydroxide were used for maintaining pH of the pulp.
Methods

The bench scale flotation experiments were carried in standard Wemco Fagergren Laboratory Flotation Cell. For this purpose 500 gram -1680 micron ore sample was wet ground in laboratory rod mill at a pulp density of 66% and floated after conditioning with the flotation reagents at a pulp density of 20%. All the products from the flotation experiments were assayed for %U₃O₈ by standard spectrophotometric method involving uranium extraction with tri-butyl phosphate.

RESULTS AND DISCUSSION

Chemical, Granulometric and Mineralogical Characteristics

As mentioned earlier the ore sample analysed 0.052 % U₃O₈. The particle size and uranium distribution in different size fractions of -1680 micron ore sample as give in Fig. 1, showed the preferential concentration of uranium values in the fine size ranges, particularly below 37 micron fraction contained 68.4 % of the uranium distribution. The X-ray diffraction studies carried out on the head sample indicated a broad picture of the various mineral phases. The sample was predominantly made up of quartz while other minerals like pyrophyllite, kaolinite, muscovite, paragonite, ferricolumbite and rutile occur in minor proportion to traces. The presence of radio-active mineral uraninite was indicated by X-ray spectra. The X-ray spectrum of the sample is shown in Fig. 2.

Figure 1: Particle size and uranium distribution in different size fractions of crushed sample.

Figure 2: X-ray diffraction pattern of uranium ore sample.
Effects of Granulometry on Flotation of Uranium Minerals

Experiments were carried out to study the effects of granulometry on the flotation behaviour of uranium bearing minerals. For flotation with potassium amyl xanthate pH of the pulp was maintained at 8.5 while for the combination of cuferron and light diesel oil, pH was 4.5. The results of these experiments are shown in Fig. 3. As we can see from the data shown in Fig. 3 that due to the better liberation an increase in fineness of the feed from 30.6 % to 35.1 % increased the uranium recovery from 85.6 % to 92.6 % respectively. Similarly the yield has also increased from 24.7 % to 35.6 %. A further increase in fineness of the feed and hence a decrease in the particle size shows a marginal improvement in recovery but at the cost of loss in selectivity as evident by the sharp increase in yield and a corresponding decline in grade of the concentrate [1]. An increase in the yield of the concentrate at finer size with increasing fineness of the feed is mainly due to increased gangue recovery by entrainment and entrapment phenomena and the mechanical carry over [2]. Based on these results a feed with 35.1 % -74 micron was considered suitable for uranium recovery from the sample.

![Figure 3: Effects of granulometry of feed on flotation of uranium bearing minerals.](image_url)

Flotation of Classified/Deslimed Feed

From the size-wise chemical analysis of crushed product and ground flotation feed it was found that the sample showed a preferential distribution of uranium values in the finer size fractions, particularly fraction below 37 micron contained uranium values more than 60 % [1]. So it was felt that there exists a possibility of separating uranium rich finer fraction and floating the sand against the direct flotation of the ground bulk feed. The classification/desliming of the flotation feed is also expected to remove the fine particles which are known for slow flotation rate and thereby improving the overall flotation efficiency [3,4]. In order to examine these facts the ground flotation feed was classified using 37 micron screen and sand was floated. Another flotation experiment was carried out under the identical conditions using a manually deslimed feed. The desliming of the feed was performed after suitable scrubbing in laboratory mechanical agitator. The uranium recovery from the classified and deslimed feed are shown in Fig. 4. As it can be observed from the data shown that classified and deslimed feed gave uranium recovery of 26.5 % and 22.7 % respectively. When the uranium rich fines (-37 micron fraction/slimes) are mixed with the flotation concentrate the resulting uranium recovery is ~92 % in both the cases which are similar to those obtained by direct flotation of the ground ore as shown in the same figure. So in the present case from the process point of view classification/desliming of the
feed is not essential but this offers an option for reducing the load on flotation by splitting the feed prior to flotation and thereby reducing cost on reagents etc.

Figure 4: Flotation results with classified/deslimed feed against normal feed.

Effects of Xanthate Dosage on Flotation

Potassium amyl xanthate has proved useful in flotation recovery of uranium bearing minerals from the present ore sample. In order to study the effects of the variation of dosage of xanthate on the uranium recovery and the flotation selectivity, experiments were carried out varying the xanthate dosage from 0.075 kg/t to 1.2 kg/t. For these experiments flotation with the combination of cupferron and light diesel oil was discontinued. The results of the experiments on xanthate dosage variation are shown in Fig. 5. It was found that although there was a progressive increase in the yield of the flotation product with an increase in the xanthate dosage from 0.075 kg/t to 1.2 kg/t [1,5] but as it is evident from the Fig. 5 that almost there is no improvement in the uranium recovery. The increase in the yield was mainly due to loss in selectivity of separation leading to increased recovery of gangues with higher dosage of xanthate. So 0.075 kg/t xanthate (alongwith cupferron and light diesel oil) was considered suitable for flotation of uranium bearing minerals from the sample.

Figure 5: Uranium recovery under varying dosage of xanthate.
Variation of Dosage of Cupferron and Light Diesel Oil

As mentioned earlier cupferron and light diesel oil were used alongwith potassium amyl xanthate as collector for flotation of uranium bearing minerals. As discussed above xanthate was effective in recovering more than 60% of the uranium values from the sample. This was attributed to the association of pyrite and carbonaceous materials with uranium bearing minerals in the sample [1]. But for further improving the uranium recovery, cupferron, a uranium chelating agent, was used. Light diesel oil was used as a chain extender alongwith cupferron. Experiments were carried out to examine the effect of the variation of dosage of cupferron and light diesel oil on flotation of uranium minerals. The dosage of cupferron was varied from 0.312 kg/t to 1.25 kg/t while for light diesel oil the same was from 0.462 kg/t to 3.6 kg/t. The results are shown as the additional uranium recovery (over those already recovered by flotation with xanthate) against the dosage of the reagents in Fig. 6. As it can be observed from the results summarized in Fig 6 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. Again for light diesel oil, an increase in dosage from 0.462 kg/t to 1.8 kg/t, there is some improvement in recovery but further higher dosage does not lead to any improvement.

![Graph showing effects of cupferron and light diesel oil on uranium recovery.](image)

Figure 6: Effects of cupferron and light diesel oil on uranium recovery.

Effects of pH

pH governs the charge on the mineral surface and plays an important role in the adsorption of the flotation reagents and hence floatability of minerals. Normally xanthate is used in alkaline pH for flotation of most of the metallic sulphides but for pyrite slightly acid circuit is preferred. On the other hand cupferron forms complex with uranium ion in the reduced state (U⁴⁻) in a strongly acid medium. Good recovery of uranium is reported between pH 4-6 [6-8]. Marabini finds high recovery of pitchblende for Hallimond tube flotation over pH 0-4 [9]. But at the same time considering the presence and association of uranium phases and the varying degree of floatability in the present case the effects of pH was varied in a wide range. The results of pH variation is shown in Fig. 7. As it can be seen from the results that though a reasonably high recovery is possible in a wider range of pH but a slightly alkaline pH for flotation with xanthate and slightly acid circuit for flotation with cupferron is preferable. The best results obtained at pH 8.5 for flotation with xanthate and at pH 4.5 for cupferron flotation.
Alternate Reagents Schemes

Xanthate is a commercially established collector and is also known as an economical flotation reagent, which has shown its efficacy in the flotation of uranium bearing mineral species in the present ore sample.

On the other hand application of cupferron has been limited to laboratory flotation studies and it is considered costly for commercial applications. The reagents like diethyl hexyl phosphoric acid, alamine 336 and tributyl phosphate, because of their tendency to form complexes with uranium, are used in solvent extraction of the uranium [10]. So in order to develop alternate schemes, studies were undertaken using these reagents as flotation collector for uranium bearing minerals in combination with light diesel oil. It was interesting to note that a reasonably high uranium recovery could be achieved with above reagents along with potassium amyl xanthate [1,5].

CONCLUSIONS

Flotation studies were carried out on a low grade uranium ore sample from Domiasiat, Meghalaya. The sample assayed 0.052 % U3O8, and it showed a preferential concentration of uranium values in the fine size ranges. Use of potassium amyl xanthate in combination with cupferron and light diesel oil as collector for flotation of uranium bearing minerals resulted in a pre-concentrate with a yield of ~ 35 % and uranium recovery over 90 %. Flotation results with the alternate flotation reagent schemes involving diethyl hexyl phosphoric acid, alamine 336 and tri-butyl phosphate in combination with light diesel oil and potassium amyl xanthate were also found encouraging. Pre-concentration of the low grade sample by froth flotation can reduce the load on the hydrometallurgical extraction of uranium.

ACKNOWLEDGEMENTS

The authors sincere thanks are due to Prof. P. Ramachandra Rao, Director, National Metallurgical Laboratory, Jamshedpur for kindly permitting to publish this paper. They wish to thank to the management of M/s Uranium Corporation of India Ltd., Jaduguda for providing the financial support to this project. They also thank them for supplying the necessary uranium ore samples and carrying out the chemical analysis of the flotation products.
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