Sono-chemical leaching of uranium

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Abstract

A fundamental study on the mechanism of uranium leaching in nitric acid and sulphuric acid media has been carried out to understand the effect of the ultrasound on leaching. The use of ultrasound clearly improves the leaching rate within the studied leach acid media. The enhancement in the leaching rate in the presence of ultrasound is higher with low leach acid concentration of nitric acid (HNO_3), and it is high at high leach acid concentration in the case of sulphuric acid (H_2SO_4) being used as a leachant, when compared to conventional mechanical agitation. The basic reason behind this observed variation is explained on the basis of the reaction mechanism involving the oxidative conversion of acid insoluble tetravalent uranium form to the soluble hexavalent form of uranium in the presence of ultrasound at a faster rate.
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1. Introduction

Uranium ore leaching process exemplifies the successful application of hydrometallurgical process for the uranium recovery, especially when treating with high grade ores. In lean grade uranium leaching process, conventional ore dressing technique have not been successful in achieving preconcentration of the uranium minerals [1]. A major problem in these existing hydrometallurgical leaching operations is the relatively long contact time required for the solubilization of metals from the ore materials. The mining industries are constantly seeking new and practical technologies to treat low grade ores to increase the profitability. Bioleaching is one of the biggest processes in use in the mining industry [2,3]. It involves the acid producing bacteria which are used for the solubilization of ores to recover valuable metals. Bioleaching provides possibility of metal recovery from low grade ores, however major challenge is to find a suitable match between an ore body and bioleaching technology, i.e., appropriate micro-organisms [4]. Inspite of slow recovery rates of the process, this technology is attractive as it is not energy intensive. Among various physical methods for speeding up the

leaching process, the application of ultrasound is proving to be of considerable interest for intensifying the performance of the process [5–7].

In seeking to shorten the leaching time with out a decrease or possible enhancement in the final uranium recovery values, a process intensification study has been carried out with the use of ultrasound. Ultrasound can rapidly extract metals from ores through a process of cavitation in the form of shock waves and micro-jet formation through the cavitating medium such as water [8,9] or dilute acids [10]. A large collection of the literature, about the application of ultrasound on the metal leaching operation has been reported in our earlier work [10]. The first attempt of intensifying the leaching of uranium using ultrasound has been successfully made and reported in our earlier work [10], where the leaching of uranium from MgF_2 particles of different size ranges under different leach acid concentrations of nitric acid (HNO_3) was studied. It was concluded that the improvement in the leaching rate stems from the enhancement of the convective diffusivity of the leach acid into the pores of MgF_2 particles and it also disintegrate the MgF_2 particles as a result of cavitational events (micro-jets leading to attrition of the solid particles).

Uranium ores which are of commercial importance in India are of comparatively low grade containing less than 0.1% of uranium. The disseminations in metamorphic rocks represent the

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most important uranium deposits in India. The economic recovery of uranium is a challenge for developing highly efficient chemical processes for the leaching, purification and concentration of uranium from such ores. In the present work, leaching of uranium from this low grade uranium ore material has been carried out in the presence of ultrasound, with different concentrations of nitric acid (HNO_3) and sulphuric acid (H_2SO_4) as leachants. Greater intensification in the leaching rate in the presence of ultrasound has been observed with high concentration of sulphuric acid (H_2SO_4), where as the greater enhancement in the leaching rate has been observed with low nitric acid concentration leach solution when compared to conventional mechanical agitation (stirring). This basic difference in the observed variation (higher ultrasound effect at high H_2SO_4 concentration and low ultrasound effect at higher HNO_3 concentration) has been explained in terms of the different reaction pathways of the conversion of acid insoluble tetravalent uranium form [U(IV)] to soluble hexavalent uranium [U(VI)] form with the two leach acids studied.

2. Materials and methods

2.1. Ore characteristics

The ore material was obtained from the Narwapahar uranium deposit (Jharkhand, India). The material was selected from a representative elemental and modal composition as well as of the uranium content. The average uranium content present in the ore was about 0.035% and uranium was present in the partly oxidized form with conversion of [U(IV)] to [U(VI)], the degree of oxidation ranges from 16 to 34%. The ore was also found to contains ∼9% of the Fe (iron) and remaining were acid insoluble silicates and soluble carbonates. The ore material was ground to a level of 68–75% below 200 mesh (i.e., ∼50 μm, approx.).

2.2. Leach solutions and experimental setup

The leach solutions were prepared with nitric acid (HNO_3) and sulphuric acid (H_2SO_4) of different concentrations, and all the experiments were performed in a 200 mL three neck cylindrical shaped round bottom glass reactor (10 cm height and 5 cm internal diameter). 20 ± 1 kHz frequency of ultrasound irradiations (with delivered power of 36 W) were used in the ultrasonic cleaning bath type system having dimensions of height, width and breadth as 15 cm × 15 cm × 15 cm, respectively (Supersonics, Ultrasonic system, Mumbai). The distance between the cylindrical reactor bottom and the ultrasonic bath was maintained at 2 cm and the liquid pool was maintained up to a level of 11 cm in the ultrasonic bath, covering the entire acid slurry solution in the reactor as it was exposed to ultrasonic irradiation. Solid loading of 50% (50 g of ore material in 100 mL of leach acid solution of different concentrations) weight by volume has been used in all the reported experiments. In the conventional stirring based leaching process, the agitator used was 4 cm diameter, 45° down pumping 6 pitch bladed turbine impeller, which has good solid suspension characteristics for the solid particles. Impeller speed was maintained at 1000 rpm using
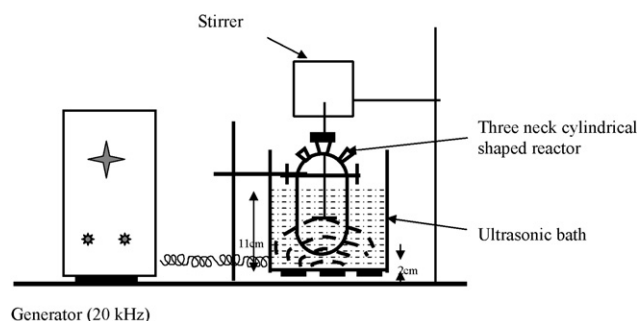

Fig. 1. Schematic diagram of experimental setup.

a variable speed drive motor with a display indicating impeller rotational speed (rpm). Schematic diagram of the experimental setup is shown in Fig. 1. The temperature was maintained at room temperature (30–32 °C), and the reaction mass was subjected to the ultrasound irradiation intermittently (gap of 10 min for every 30 min of continuous operation). Cooling water was used as a medium in the ultrasonic bath with a temperature controlled system. All the experiments were carried out in duplicate, and sometimes repeated again and the mean values have been reported. The maximum deviation observed between the reported experiments never exceeded 5%.

2.3. Analysis of leach liquor samples

The slurry samples were withdrawn at equal intervals of time. The fine solid particles are decanted by using high speed centrifuge and the supernatant liquid samples containing dissolved uranium (nitrate or sulfate) was analysed for the uranium content in the complex using UV–Vis spectro photometer (Chemito model-2500). Nitric acid leached samples (uranium nitrate, $UO_2(NO_3)_4$) were analysed at an absorbance wavelength of 375 nm, which is the characteristic wavelength of the uranium complex formed with the mixture of ethanolic ammonium thiocyanate and stannous chloride (9:1 ratio) with aluminum nitrate solution as a salting agent [10]. Sulphuric acid leached samples (uranium sulphate, UO_2SO_4) were analysed at an absorbance wavelength of 399 nm, which is the characteristic wavelength of the uranium complex formed with the mixture of 1% di-benzyle methane solution in pyridine (i.e., chromogenic solution) with aluminum nitrate solution as a salting agent (Thomas baker, analytical reagent). The extinction coefficient for this analytical method is 17,300 l/mol/cm. This high value indicates the high sensitivity of the analytical method used for the analysis. Similarly the extinction coefficient for the HNO_3 leach acid analysis is ≈4000 l/mol/cm (approx.).

3. Results and discussion

3.1. Experiments with nitric acid (HNO_3) as a leach acid

Experiments have been conducted using three different nitric acid (HNO_3) concentrations of 7.5, 10 and 15 volume percentage, equivalent to 0.88, 1.19 and 1.76 N, respectively. Results have shown improvement in the initial leaching rate with an
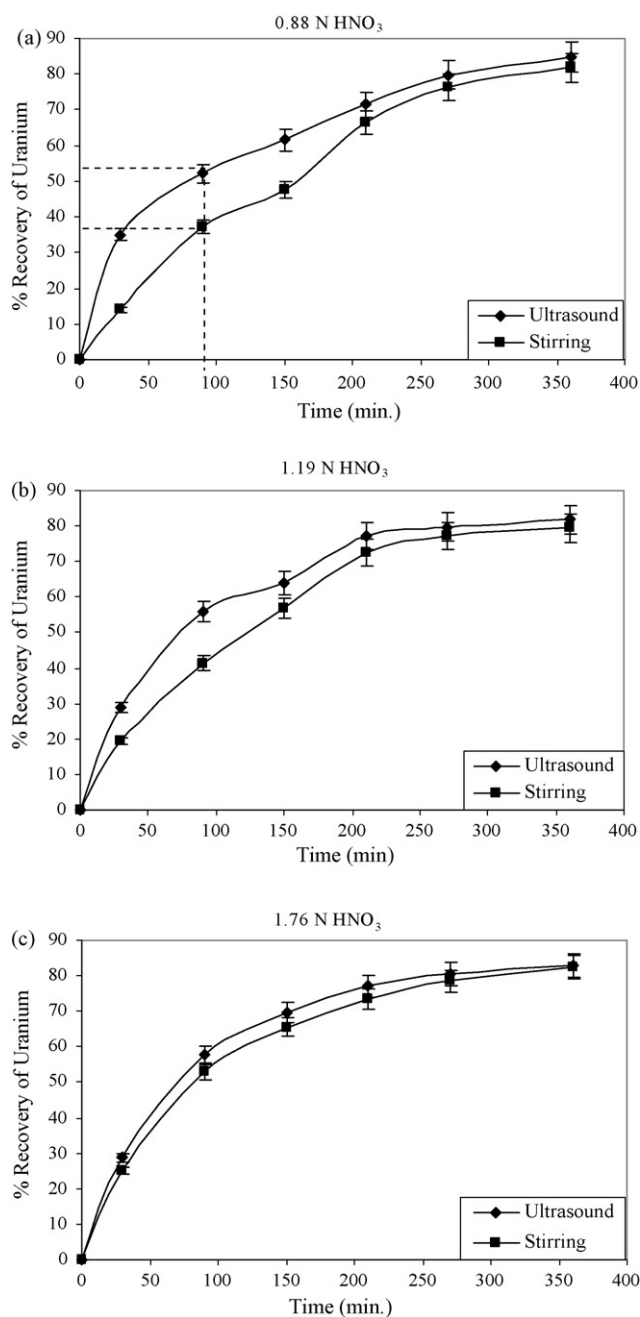
Fig. 2. Effect of ultrasound on uranium recovery with HNO_3 leach acid.

the overall extent of recovery was not much affected, remaining at ~80% for all the three leach acid concentrations studied over 6 h of leaching operation. Leaching rates of uranium were enhanced under ultrasound irradiation for all three acid concentrations (Fig. 2). As it can be observed from Table 1, under ultrasound the extent of enhancement was reduced from 42% to 8.7% with an increase in the leach acid concentration from 0.88 to 1.76 N of nitric acid. The process of transfer of leaching solvent to the inner particle ore is slower under mechanical stirring when carried out using low leach acid concentration possibly due to the slower dissolution of the gangue material exposing uranium to the leach acid. The physical effects of the cavitation such as the shock waves and the enhanced intra particle diffusion were found to increase the mass transfer rate, as a result of the asymmetric collapse of the cavity in the vicinity of solid particle [10]. It appears that the leaching solvent is penetrating into the solid particle mass and undergoing the reaction with the leaching solute (uranium) especially with low leach concentration. There is not much improvement in the leaching rate under ultrasound irradiation with higher concentration of the leach acid over mechanical agitation process. As the high concentration of leach acid diffuses more effectively due to higher concentration gradient of leach acid at solid particle surface. High leach acid concentrations are also possibly dissolving gangue material at a higher rate, exposing uranium for extraction and hence ultrasound irradiation is not as effective. These trends are similar to those observed for uranium leaching (from MgF_2 matrix) with nitric acid, reported in our earlier work [10]. The concentration of OH radicals produced in presence of ultrasound irradiation has been independently estimated in terms of iodine liberation (using KI decomposition studies [11]) to be 1.64×10^{-6} mol/l/s, where as the strength of HNO_3 used is 0.88, 1.19, 1.76 N, which is several orders of magnitude higher in concentration. Thus, it is expected that the contribution of OH radicals to the enhancement will be very low. The effect of micro-turbulence is expected to be independent of nitric acid concentration, as the cavity dynamics remains more or less the same under different acid concentrations.

As it can be observed from Fig. 2, 37.0% of uranium leaching (up to first 90 min shown in Fig. 2(a)) has been obtained with 0.88 N nitric acid concentration and with 1.76 N leach acid concentration the initial leaching (i.e., 90 min) has been increased to 52.9% of uranium content. With the use of ultrasound using 0.88 N leach acid concentration the leaching recovery in the initial period has been increased from 37.0% to 52.2%, increasing the initial rate of leaching by ~1.4 times. The over all recovery however has not increased much (marginally increased by 3–4%) by the application of ultrasound with 0.88 N leach acid

increase in the nitric acid concentration. It is observed from Fig. 2, that the initial leaching rate (up to first 90 min) has been increased by 1.4 times as we increase the nitric acid concentration from 0.88 to 1.76 N, the initial leaching rates (estimated by method shown in Appendix I) are shown in Table 1. However,

Table 1
Initial leaching rates (g/s) under nitric acid (HNO_3) leaching

S.no.	Acid conc. (N)	Leaching rate during stirring ($\times 10^6$ g/s)	Leaching rate during ultrasound ($\times 10^6$ g/s)	% Enhancement in initial leaching rate
1	0.88	1.19	1.69	42.0
2	1.19	1.34	1.81	35.0
3	1.76	1.72	1.87	8.7

concentration over 6 h leaching operation. Thus, the advantage of low acid consumption to achieve the high leaching rates of uranium is possible with the use of ultrasonic irradiation. As discussed above Fig. 2(b) and (c) show a reducing influence of ultrasound with an increasing nitric acid concentration in the leach solution.

3.2. Experiments with sulphuric acid (H₂SO₄) as leach acid

3.2.1. Reaction mechanism of uranium leaching with H₂SO₄ leach acid

Most of the uranium mills use sulphuric acid to extract the uranium from its ore, as the use of HNO_3 is more expensive. Simple hexavalent uranium oxides and its compounds are readily soluble, but tetravalent uranium is insoluble in sulphuric acid and hence must be oxidized for its dissolution to occur. Uranium which exists in tetravalent form in natural uranium ore, require oxidizing conditions to convert uranium (uraninitte [U(IV)]) in the ore to the soluble uranyle [U(VI)]. Generally the oxidant used is pyrolusite (MnO_2) or sodium hypochlorate due to the cost and case of availability.

In a kinetic study of the dissolution of UO_2 in sulphuric acid Nicol et al. [12] found that the ferric ion (Fe^{3+}) oxidizes the tetravalent uranium by surface electro chemical mechanism in which the concentration of Fe^{3+} ions adsorbed on the surface determine the rate of conversion of U(IV) to U(VI). The ferric ions act more effectively in the form of ferric sulphate complex (in particular $Fe_2(SO_4)_3$), which exchanges the electrons more rapidly than the uncomplexed ion. Dissolution rate are also affected by the presence of Fe^{2+} ions, but the extent and the type of the dependence was mostly determined by the Fe^{3+}/Fe^{2+} ratio. During uranium leaching the ferric iron concentration in the leaching liquor is usually controlled by adjusting the redox potential of the leach liquor (by the addition of oxidant) within the range 400–500 mV (relative to saturated calomel electrode). The relationship between the redox potential and the iron concentrations (Fe^{3+}/Fe^{2+}) is given by the Nernst equation [13]

$$E_c = 397 + 0.1984 \cdot T \cdot \log_{10} \left(\frac{[Fe^{3+}]}{[Fe^{2+}]} \right) \qquad (1)$$

E_c is the solution potential relative to the saturated calomel electrode at 35 °C (mV), [ ] is the molar concentration, T is the temperature (K).

The above equation indicates that at an oxidation potential of 400 mV about 50% of iron is in the form of the ferric state, while at 500 mV almost 98% is in the Ferric state. The use of increased concentrations of ferric ion (Fe^{3+}) to improve leaching rates has

also been suggested by Dutrizac and Mac Donald [14] intensifying the leaching operation. However, the full potential of this leaching can be realized only if the ferrous ion produced during the leaching can be reoxidised to ferric ion and recycled. Several methods of oxidation have been suggested [12], MnO_2 (pyrolusite) can be used as an oxidizing agent, however the cheapest and most promising for this application are either oxygen at ambient pressure or bacteria (*Thiobacillus ferrooxidans*) in the presence of oxygen [15,16].

Sulphuric acid used for the leaching is not directly consumed for the dissolution of UO_2, as it is used to maintain the required concentration of ferric sulphate [$Fe_2(SO_4)_3$] to facilitate the dissolution of hexavalent uranium.

$$UO_2 + 2Fe^{3+} \rightarrow UO_2^{2+} + 2Fe^{2+} \qquad (2)$$
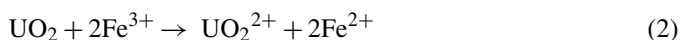
In the presence of MnO_2, the oxidation of ferrous ion to ferric ion occurs through the following mechanism

$$2Fe^{2+} + MnO_2 + 4H^+ \rightarrow 2Fe^{3+} + Mn^{2+} + 2H_2O \qquad (3)$$
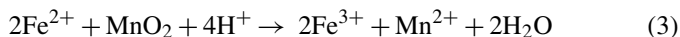
For most ores, reaction with gangue minerals account for the major fraction of acid consumption. In practice, ferric sulphate is produced by the dissolution of iron minerals present in the ore. Addition of sufficient oxidant, usually pyrolusite (MnO_2) ensures that iron is always present, predominantly in the ferric form (Fe^{3+}).

3.2.2. Experiments with H₂SO₄ as leach acid

Experiments have been conducted over 10 h of leaching period using three different leach acid concentrations of 5, 7.5 and 10 volume percentage equivalent to 1.87, 2.82 and 3.75 N of sulphuric acid (H_2SO_4) aqueous solutions. MnO_2 (85%, Thomas baker) is added to maintain the required oxidizing potential of the leach solution in the range of 480–510 mV and it is measured using Thermo-Orion (Pt) electrode (9678BN).

It can be observed from Fig. 3, that the initial leaching rate was increased with an increase in the leach acid concentration (similar to the observation with HNO_3 as a leach acid). As we increase the leach acid concentration from 1.87 to 3.75 N of sulphuric acid the initial leaching (up to first 210 min of operation) recovery has been increased from 62.7% to 72.1% with only mechanical agitation. The initial leaching rates (up to first 210 min) have been given in Table 2. With the supplement of ultrasound irradiation, the initial leaching rate is further increased, and this improvement in the initial leaching rate is higher at higher sulphuric acid concentration. With 3.75 N sulphuric acid the initial leaching recovery has been further increased to 85.6% from 72.1% by the application ultrasound compared to only mechanical agitation. As it can be observed from Table 2, under ultrasound the extent

Table 2
Initial leaching rates (g/s) under sulphuric acid (H₂SO₄) leaching

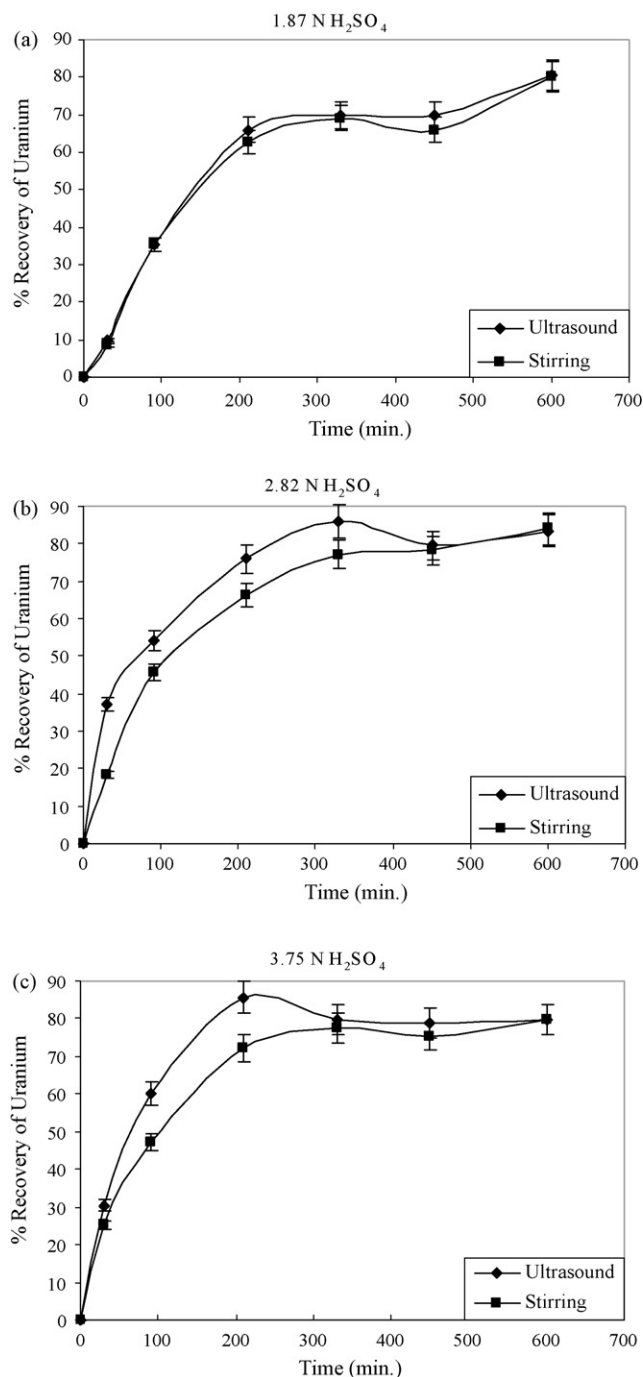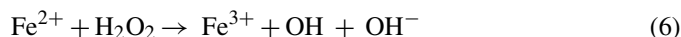| S.no.	Acid conc. (N)	Leaching rate during stirring ($\times 10^6$ g/s)	Leaching rate during ultrasound ($\times 10^6$ g/s)	% Enhancement in initial leaching rate
1	1.87	0.85	0.90	5.9
2	2.82	0.90	1.05	16.7
3	3.75	0.99	1.19	20.2

Fig. 3. Effect of ultrasound on uranium recovery with H_2SO_4 leach acid.

of enhancement in the leaching rate was increased from 5.9% to 20.2% with an increase in the leach acid concentration from 1.87 to 3.75 N of sulphuric acid. Thus, the presence of ultrasound was found to be more effective at higher leach acid concentration than at low leach acid concentration. This observed effect due to ultrasound on the leaching rate enhancement was exactly opposite to that observed with nitric acid leaching process. It seems that mechanism of leaching under ultrasound irradiation with two studied leach acid media appears to be different.

The observed variation in the leaching rates with sulphuric acid and the possible reason for the enhancement in leaching rates under ultrasound irradiation is explained on the basis of oxidative conversion of U(IV) to U(VI). The extreme conditions (such as high temperature in the range of 5000 K and pressure of 1000 atm) generated during the catastrophic collapse of the cavities known to produce OH radicals (strong oxidizing agents) under ultrasound irradiation. Under acid medium these in turn facilitates the oxidative conversion of Fe^{2+} to Fe^{3+}. This oxidation, is however not only due to OH radicals but also, HO_2 radicals, H_2O_2 and H atoms formed during the ultrasound irradiation initiated cavitating conditions (shown in Eqs. (4)–(6)) [17,18]

$$Fe^{2+} + OH \rightarrow Fe^{3+} + OH^- \tag{4}$$

$$Fe^{2+} + HO_2 \rightarrow Fe^{3+} + HO_2^- \tag{5}$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH + OH^- \tag{6}$$

To confirm this mechanism of oxidative conversion of Fe^{2+} to Fe^{3+} experiments were conducted with and without ultrasound irradiation using 3.75 N sulphuric acid leach solution without the (externally added) oxidizing agent (MnO_2) and these leaching curves are shown in Fig. 4 over a 10 h period. As it can be observed from Fig. 4, 41% and 31.8% leaching recoveries have been obtained with and without ultrasound irradiation respectively without the externally added oxidizing agents. Hence, 29% improvement in the leaching recovery value obtained (over 10 h of operation) indicate a clear influence of the in situ formed OH radicals under ultrasound irradiation, facilitating the conversion of Fe^{2+} to Fe^{3+}. The variation in the measured oxidizing potential (ORP reading) of the slurry i.e., ratio of (Fe^{3+}/Fe^{2+}) during the leaching operation with respect to the time is shown in Fig. 5. The higher values of the ratio Fe^{3+}/Fe^{2+} during the initial period (210 min) of the ultrasound irradiation assisted operation, responsible for the observed high initial leaching rates of the uranium (over agitation) leaching, is readily evident from Fig. 4. However, this ratio was found to be decrease as the time of leaching operation progress indicating possibly the consumption of
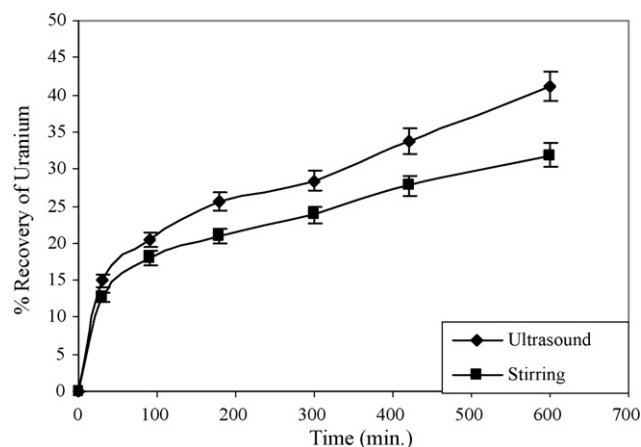

Fig. 4. Uranium recovery with and without ultrasound irradiation with out any addition of external oxidizing agent (MnO_2).
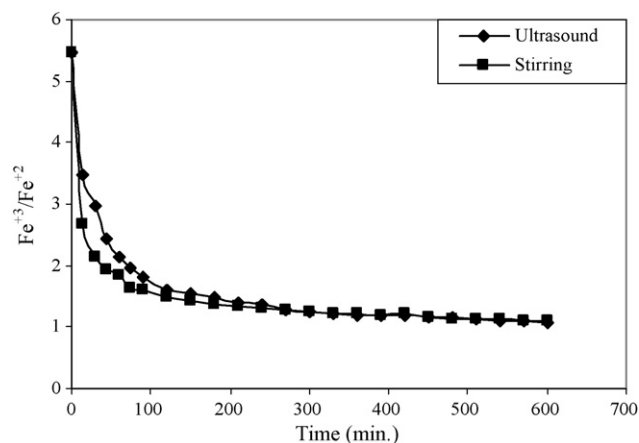
Fig. 5. Variation of Fe^{3+}/Fe^{2+} ratio during leaching operation with and without ultrasound irradiation.

the Fe^{3+} ions in the conversion of tetravalent form of uranium to hexavalent form.

The oxidizing conditions are readily present under ultrasound without MnO_2 but are not efficient enough for achieving the higher leaching rates as well as the recoveries (∼75–80%), as observed by the externally added MnO_2 as shown in Fig. 3. The combination of the ultrasound with external addition of the MnO_2 seems to be more effective for the conversion of Fe^{2+} to Fe^{3+}. This oxidative conversion rate under ultrasound is expected to be more under high (3.75 N) sulphuric acid concentration [19]. This appears to be the main reason why higher concentration of sulphuric acid show a better leaching rates than low acid concentration under ultrasonic irradiation than compared to agitation leaching observed in Fig. 3. The variation in the initial leaching rates observed with different acid concentration under ultrasound is not possibly due to the particle size reduction occurring during the ultrasound irradiation in this case and can be attributed to microscopic turbulence, enhancing the leaching rate. Our earlier work [10] with higher starting particle size (≈230 μm) showed a clear reduction in the particle size in the presence of ultrasound and the observed enhancement in the leaching rate was much more than observed in this work. As explained earlier [10], enhancement in the leaching rate is lower for lower starting particle size as is observed in the present work. The lower starting particle size (≈50 μm) in this work does not result into significant particle size disintegration and hence possibly is not the rate controlling phenomena for the observed enhancement in the leaching rates with different leach acid concentrations under ultrasound.

As it can be observed from Fig. 3(c), 78% uranium recovery has been achieved using 3.75 N leach acid concentration in 10 h of agitation leaching. Where as 85% uranium recovery has been achieved using the same leach acid concentration, supplemented with ultrasound irradiation within 3.5 h of leaching operation. The overall recovery has been increased by only ∼9% but a substantial reduction in the leaching period (3.5 h with ultrasound against 10 h with only stirring) has been achieved. This clearly shows a significant enhancement in the leaching rate. Further continuation of leaching, uranium content in the leach

liquor has been found to decrease to 78% (6.5 h of further ultrasound irradiation), indicating the loss of soluble uranium. It has been reported in the literature [20], sufficient acid is required to maintain pH less than 2.5 and preferably below 2.0 to prevent precipitation of the ferric ion as the hydroxide. The reason for this observed reduction in the uranium recovery could be after 3.5 h of operation, acid has started reacting with the gangue material present in the ore and hence the consumption of acid has resulted into an increase in the pH, (possibly above 2.0) leading to the precipitation of some ferric ion as hydroxide. Similar decreasing trend in leaching recovery as a function of time has also been reported in conventional leaching process of metals such a copper, nickel and cobalt from lean grade oxides and silicates ores [21,22]. The losses encounted during the leaching of such metals from ores have been attributed to the adsorption of dissolved species of interest metal on the residual soils having hydroxide/oxide surfaces [23]. At this stage, it is only speculated and further work is required to ascertain these speculations.

4. Conclusion

Enhancement in the leaching rate of uranium with nitric acid under ultrasound irradiation seems to be only due to physical effects of cavitation phenomena. Nitric acid is a strong oxidizing agent, it oxidizes the U(IV) to U(VI) at a much faster rate, and hence the leaching rate is controlled by convective diffusivity of the leach acid into the micro-pores of ore particle which is facilitated by cavitating conditions. Where as in the case of sulphuric acid leaching, MnO_2 (pyrolusite) is used as an oxidant, since the sulphuric acid is not a strong oxidizing agent. The oxidative formation of Fe^{3+} [$Fe_2(SO_4)_3$] appears to be the rate controlling phenomena, and this step is speeded by the oxidizing conditions (OH radicals) produced during ultrasound cavitation under high acid medium conditions, i.e., chemical effects of ultrasound cavitation causing the enhancement in the leaching rate of uranium.

Acknowledgement

Appendix I

The leaching rates shown in the tables have been calculated based on the initial leaching rates. In the case of nitric acid leaching, experiments have been conducted for 6 h of operation, so the initial leaching rate has been considered up to first 90 min of operation, since the leaching is occurring with constant rate up to first 90 min of operation.

For typical sample calculation, the leaching recovery of uranium up to first 90 min of operation under ultrasonic irradiation and only mechanical agitation with 0.88 N leach acid shown in

Fig. 2(a) is observed to be 52.2 and 37.0%, respectively, and the corresponding uranium leached out (recovered) is 9.1 and 6.5 mg, respectively (uranium percentage in the ore is 0.035% by wt. and 50 g of sample used in each experimental run).

Initial leaching rate under ultrasonic irradiation

$$= \left(\frac{\text{uranium recovered}}{\text{time of leaching}} \right) = 1.69 \times 10^{-6}\,\text{g/s}$$

Similarly, initial leaching rate under mechanical agitation = 1.19×10^{-6} g/s

%Enhancement in the initial leaching rate with ultrasonic

$$\text{irradiation} = \left(\frac{\text{increase in the leaching recovery}}{\text{recovery due to only agitation}} \right) = 42.0\%$$

Similarly for other leach acid concentrations the initial leaching rates and the enhancement due to ultrasonic irradiation are shown in Table 1.

With sulphuric acid leaching, the experiments have been conducted for 10 h of operation, the initial constant leaching rate has been considered to be up to first 210 min of operation. The initial leaching rates and the % enhancement due to ultrasonic irradiation have been calculated and shown in Table 2.

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