Selective Leaching of Steel Pollutant Element from Dilband Iron Ore, Pakistan

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ABSTRACT

Dilband Iron ore of Pakistan is lean grade ore which contains sufficient amount of phosphorous bearing mineral called fluorapatite. Since phosphorous is widely acknowledged as steel pollutant and disturbs the blast furnace chemistry, so it needs to be removed effectively from the ore before the smelting operation. Present paper, therefore, aims to provide the results of phosphorous leaching efficiency noted with HCl, HNO_3 and H_2SO_4 reagents. Results showed that H_2SO_4 leached the phosphorous quite selectively as compared to HCl and HNO_3. Henceforth, it was possible to eradicate 49.5, 55.6 and 82.2% P_2O_5 from -600+212, -212+106, and -106 μm particle size fractions respectively with 1% concentrated H_2SO_4.The gradual increasing trend of dephosphorization with decreasing the particle size implies that, in finer size fraction liberation of fluorapatite mineral was amplified. Perforation study using SEM (Scanning Electron Microscope) provided supporting evidence in this regard.

Key Words: Selective Leaching, Fluorapatite, Calcite, Kaolinite, Quartz, Hematite, Scanning Electron Microscope.

1. INTRODUCTION

PSM (Pakistan Steel Mill) is totally dependent on the imported ores because of the lacking of mineral processing activates in the country, concomitantly PSM in now a days is under major financial crises and is reached to the verge of collapse. It is, therefore, imperative to start the ore beneficiation activities so that nation can took full advantage from indigenous ore deposits. Among the iron ore deposits discovered so far in Pakistan, Dilband iron ore is more suitable than any other indigenous ores, because deposits are present in large quantity and of open mine type. Estimated amount of Dilband iron ore is 200 million tons and averagely it is composed of 46.27% hematite, 17.41% quartz, 14.47% calcite, 9.24% chlinochlore, 10.5% kaolinite and 1.75% fluorapatite [1-3]. The dilemma of Dilband iron ore is that it contains two major gangue minerals namely quartz and fluorapatite. Former is fuel consumer in smelting process and latter is major source of phosphorous which is generally believed to be steel pollutant deteriorating ductility of the steel products. Previous work done on this ore indicated that it is almost impossible to upgrade it by any physical separation method, since interlocking of these gangue minerals within the iron matrix is extended up to 5 micron mesh size [4]. Henceforth, the only way

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remains to upgrade this iron ore is acid leaching likewise most of the advanced countries where successful dephosphorization through chemical method has been achieved. The methods other than acid leaching like bio-leaching [5], roasting and melting separation [6], roasting followed by magnetic separation, and selective agglomeration plus reverse flotation [7] may be effective route to desulphurize the ore. Nevertheless, these methods are much costly, therefore for Dilband iron ore acid leaching [8-9] was selected. The selectivity and efficiency of the HCl, HNO_3 and H_2SO_4leachants was studied and effective particle size, leaching time and leachant dose was evaluated.

2. EXPERIMENTAL WORK

Dilband iron ore sample of different particle size fractions were received from PSM. The practical size and elemental analysis of the samples is given in Table 1.

For leaching experiments the iron ore of mass 5 gm was mixed with 50 ml of diluted acid in100 ml Pyrex cylinder. Then contents of cylinder were agitated using magnetic stirrer for time interval of 15, 30, 45 and 60 minutes at room temperature. Thereafter, leachates were separated using 5 µm Whitman filter paper. This was followed by rinsing of ore residue with distilled water and drying for overnight at 100^oC. Finally, Fe, Ca and Al were analyzed by using atomic PerkinElmer AA700 AAS (Absorption Spectrometer), whereas phosphorous content in leachate was determined by DR. 2000 spectrophotometer. SEM was used to study the perforations in the residue caused by leaching. The pH of the slurry before and after the addition of acids was 6.8 ± 0.5 and 2.5 ± 0.8.

3. RESULT AND DISCUSSIONS

3.1 Effect of Size on Dephosporization

To study the effective particle size, dephosphorization experiments were carried out in which -600+212, -212+106, and -106 µm size fraction were leached in 1% H_2SO_4 solution for 30 min at ambient temperature. The percent extraction of P, Fe, Ca and Al shown in **Fig. 1** indicates that dephosphorization achieved in -600+212, -212+106 and -106 µm particle size fractions was49.5%, 55.6 and 82.2%, respectively. The increasing trend of dephosphorization with decreasing the particles size can be attributed with increase in the liberation of flourapatite mineral. Thus, inadequate liberation of flourapatite in -600+212 and -212+106 µm particle size fractions reduced the reactivity of H_2SO_4 with phosphorous and resulted with sufficient extraction of Fe. The excess leaching of Ca in all size fractions further suggested that majority of flourapatite grains is distributed in the calcite matrix. This is in agreement with the findings of Abro et. al. [1] that fluoraptite is widely distributed in calcite rich biosparite iron stones.

3.2 Effect of Leachant Type and Concentration

The leaching behavior of -106 µm size fraction at ambient conditions for 30 minutes was further studied in HCl, HNO_3 and H_2SO_4. The P, Fe, Ca, and Al leaching tendency of H_2SO_4, HNO_3 and HCl at different concentration is plotted in **Figs. 2-5.** Results shown in Fig. 2 indicates that at 1% concentration of HCl, HNO_3 and H_2SO_4 the phosphorous removal was about 7, 29,

TABLE 1. PRACTICAL SIZE AND ELEMENTAL ANALYSIS OF SAMPLES

Sample Size (µm)	P (%)	Fe (%)	Si (%)	Al (%)	Ca (%)
-600+212	0.38	39.23	19.75	3.61	4.08
-212+106	0.27	38.12	19.76	3.36	7.93
-106	0.45	38.15	22.84	3.19	9.36

and 82.2% respectively. By increasing the dose of the acids up to 3% the desulphurization in case of HCl and HNO_3 was reached to 68.9 and 77.8% respectively, whereas in case of H_2SO_4 marginal variation in % removal of phosphorous was noted. The desulphurization trend represented by Fig. 2 clearly demonstrate that H_2SO_4 was more effective than other acids. In order to know whether H_2SO_4 is also selective as it has been reported in the literature [8-9], the leaching rate of Ca, Fe and Al was evaluated. Fig. 3 indicates that leaching rate of Ca was appreciably low in case of H_2SO_4 as compared to HCl and HNO_3, whereas leaching of Fe and Al, shown in Figs. 4-5 respectively, was almost equivalent in all acids. The excess leaching of Ca with HCl and HNO_3 as compared to H_2SO_4 is the additional witness from which it was substantiated that H_2SO_4 is also more selective to extract phosphorous as compared to counterpart HCl and HNO_3 acids.

The above data make it obvious that H_2SO_4 has responded ideally for phosphorus extraction from iron ore. The selective extraction of phosphorous by H_2SO_4 can be explained by considering the number and nature of negative and positive ions (e.g. SO_4^{-2}, NO_3-Cl- and H^+) produced by the acids and their affinity for Ca^{+2} cations present in flourapatite mineral [$Ca_5(PO_4)_3F$]. It is reported that SO_4^{-2} released from H_2SO_4 possess higher affinity with Ca^{+2} cations as compared to Cl^-, and NO_3^- species released from HCl and HNO_3. Yong-Shi et. al. [8] and Cheng et. al. [10] have also observed that H_2SO_4 is preferentially reacting with phosphorous bearing mineral. Keeping in view the ideal response of H_2SO_4, an attempt was made to assess the leachability of leachant obtained from first set of experiment. It was noted that

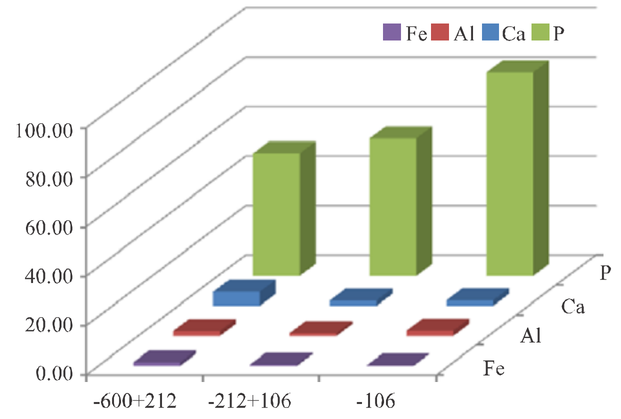

FIG. 1. EFFECT OF PARTICLE SIZE ON % LEACHING OF P, Fe, Ca, AND Al USING 1% H_2SO_4 FOR 30 MIN AT ROOM TEMPERATURE

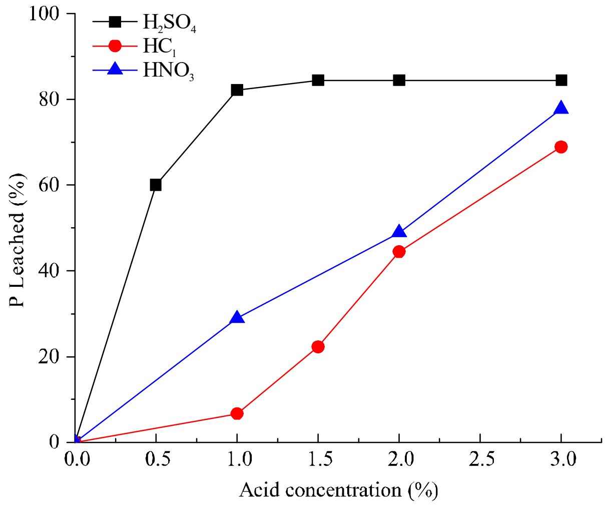

FIG. 2. EFFECT OF ACID TYPE AND CONCENTRATION ON LEACHING OF P PRESENT IN DILBAND IRON ORE

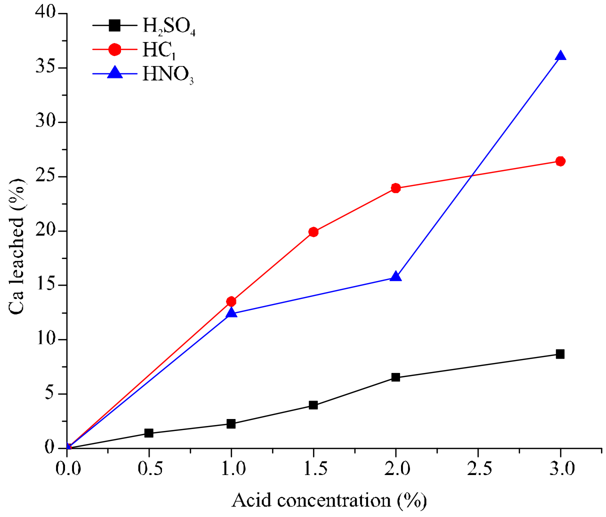

FIG. 3. EFFECT OF ACID TYPE AND CONCENTRATION ON LEACHING OF Ca PRESENT IN DILBAND IRON ORE

by recycling the leachant of H_2SO_4 hardely 20% desulphurization was achieved.

3.3 Effect of Leaching Time

Effects of the leaching time on dephosphorization isshown in **Fig. 6** which indicates that the percent extraction of phosphorous in case of H_2SO_4 remained dominate irrespective of leaching time. Moreover, from the results shown in Fig. 6 it can be substantiated that 30 min time is sufficient to leach out more than 82% poshourous from the ore matrix by using 1% concentrated H_2SO_4. In addition, **Figs. 7-8** representing the effect of leaching time on the percent removal of Ca and Fe respectively clearly demonstrate that leaching time should not be prolong than 30 min, otherwise sufficient amount of Ca and Fe will extract due to which selective dephosphorization will be affected.

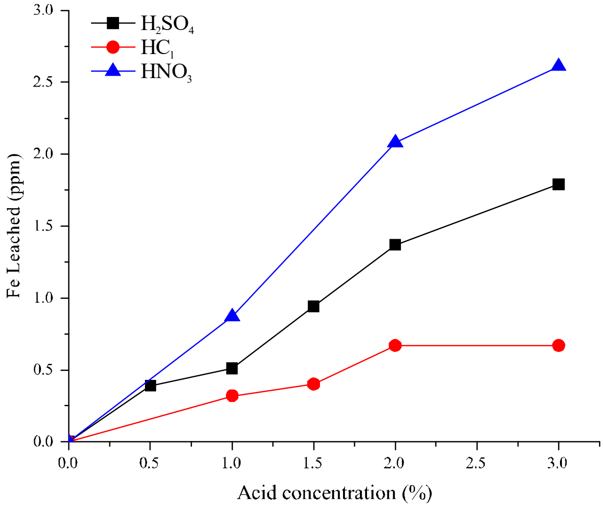

FIG. 4. EFFECT OF ACID TYPE AND CONCENTRATION ON LEACHING OF Fe PRESENT IN DILBAND IRON ORE

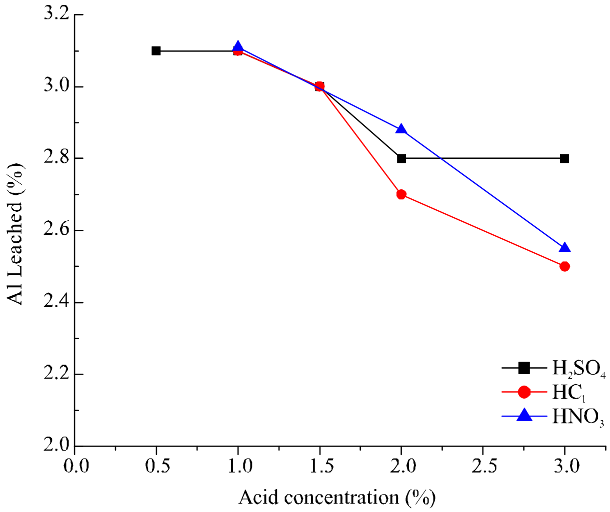

FIG. 5. EFFECT OF ACID TYPE AND CONCENTRATION ON LEACHING OF Al PRESENT IN DILBAND IRON ORE

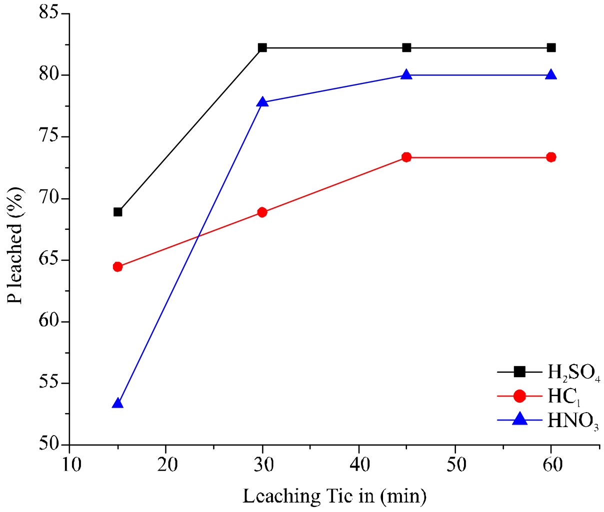

FIG. 6. EFFECT OF LEACHING TIME ON LEACHING OF P PRESENTIN DILBAND IRON ORE

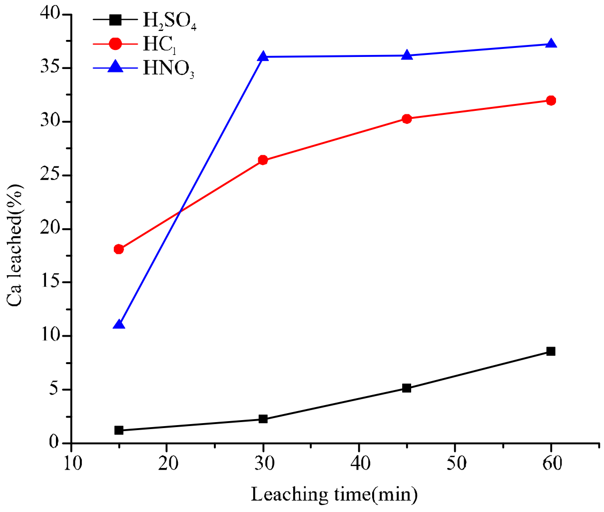

FIG. 7. EFFECT OF LEACHING TIME ON LEACHING OF Ca PRESENT IN DILBAND IRON ORE

3.4 Perforation Study

In order to witness the selective leaching of flourapatite mineral with H_2SO_4 as compared to HNO_3 and HCl acids perforation evidences were collected by illuminating the ore residue under SEM. **Fig. 9(a-c)** shows the SEM images of the ore residue collected after leaching experiments. By comparing the deepness and wideness features of the perforations it can be comprehended that perforations caused by H_2SO_4 are more detailed than caused by HNO_3 and HCl. From this, it can be substantiated that H_2SO_4 was more effective and selective to react with phosphorous bearing mineral than other minerals present in the ore body.

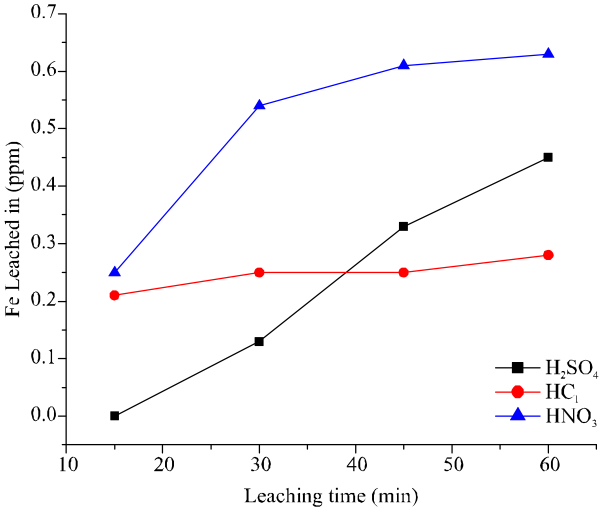

FIG. 8. EFFECT OF LEACHING TIME ON LEACHING OF FEPRESENT IN DILBAND IRON ORE

4. CONCLUSION

Present study concluded that percent removal of phosphorous from the ore body using H_2SO_4 is more selective and effective than HCl and HNO_3 acids. While evaluating the effective size it was noted that at 1% H_2SO_4 the 49.5, 55.6 and 82.2% dephosphorization was achieved in -600+212, -212+106, and -106 µm particle size fractions respectively, which meant that leaching rate of mineral is increasing with decrease in size. From this, it was conceived that leaching rate increase with increase in the liberation of flourapatite from host ore.

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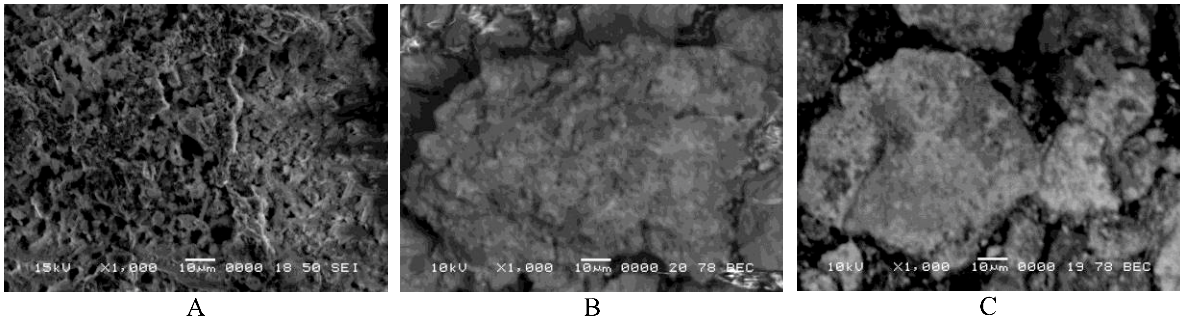

A

B

C

FIG. 9. SEM IMAGES INDICATING PERFORATION IN DILBAND IRON ORE SAMPLES

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