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Chemistry

Extraction of Aluminium and Iron using Mineral acids from Talcher Thermal Power Station Fly Ash

KEY WORDS: Fly ash, Aluminium, Iron, Leaching, Mineral Acids

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ABSTRACT

Now a days the industries of developing nations are generating a huge amount of fly ash with more and more generation of power. Fly ash is a harmful pollutant and is linked to various environmental problems for which its proper disposal and utilization is highly needed. As fly ash is rich in various inorganic constituents it can be considered as a waste of recoverable resources for metal extraction. In the present study attempts have been made to study the leaching behaviour of TTPS fly ash using mineral acids such as HCl, HNO_3 , $\text{o-H}_3\text{PO}_4$ and H_2SO_4 . The study found HCl and HNO_3 suitable leachant for iron extraction compared to other acids. In a similar way $\text{o-H}_3\text{PO}_4$ and H_2SO_4 are found suitable leaching agent for Aluminium extraction. The extraction behavior is further supported by the characterization of post leached ash residues by XRD, Particle size and SEM.

Fly ash, the major industrial waste product generated from the thermal power plants has become environmental concern because of its exponential growth and improper disposal. It is one of the solid waste which reduces agricultural productivity by settling on the leaves by which photosynthesis reduces. The heavy metals present in fly ash may leach into ground water resulting contamination. Soil pollution arises due to the use of high ash content coal and non utilization of fly ash by industries. Soil pollution due to fly ash accumulation in the nearby power plant causes land degradation, particularly soil erosion and loss of soil fertility. Application of fly ash in the manufacture of bricks, cement, concrete, ceramic products, building materials, composites, construction fill, road base, mineral filler in asphaltic mix, waste land reclamation and in agriculture has been proved to be beneficial. As fly ash is rich in minerals, another alternative use can be metal extraction. It is obvious that no single application is likely to consume all the generated fly ash but mineral extraction could provide additional markets and increased utilization of fly ash.

The physical and chemical properties along with its quality of fly ash depend upon various parameters such as coal quality, coal pulverization, combustion process, furnace type, ash collection technique and many other operational parameters. The mineralogy and chemistry of the parent coal control the mineralogy and chemistry of fly ash produced. Fly ash is a complex heterogeneous material consisting of silt-size particles ranging in colour from grey to reddish brown. Both the crystalline and amorphous phases are seen in the morphological study. Generally, the fly ash matrix is composed of 15-35% crystalline and 65-85% amorphous glassy phase¹. According to Hulett et al.² fly ash consists of three major matrices glass, mullite quartz and magnetic spinel. The major oxides present in coal ash are SiO_2 , Al_2O_3 , Fe_2O_3 , TiO_2 , CaO , MgO , Na_2O and K_2O . The mineral matters of coal are composed of rock forming minerals such as quartz, calcite, dolomite, clay minerals and iron sulphides. Besides quartz and pyrite, a clay fraction consisting of kaolinite, illite and any expandable irregular mixed-layer mineral has also been reported³⁻⁵. A study on the composition of Indian fly ash agrees with the above as it shows the fly ash consisted of largely silica, alumina, iron oxides and carbon together with significant percentage of Ca, Mg, K, Ti and variable trace elements⁶. According to the report of Natusch et al.⁷ a number of potentially toxic elements are concentrated on the particle surface at varying depth. More refractory species like Al, Fe, Si and minor elements like Ca and Mg do not exhibit surface predominance rather they are strongly associated with the ash matrix. Most of the trace elements present in coal tend to be associated with the particle surface rather than core. The elemental concentration varies with particle size⁸.

As the fly ash is rich in mineral oxides, and most of them are in the glass matrix, it becomes essential to dissolve the glass matrix for the extraction of metal values. Thus, chemical process based on i) Sintering ii) Gas-solid reaction and iii) Leaching treatments alone or in combination are extensively studied. The leaching treatment is either acidic or basic, in which inorganic acids (mineral acids) like HCl, HNO_3 , $\text{o-H}_3\text{PO}_4$, H_2SO_4 are used and for basic/alkali leaching

solution of NaOH or alkaline salts such as Na_2CO_3 is used. A large number of processes for the recovery of alumina from clays and other alumina bearing minerals (other than bauxite) have been proposed by several workers⁹⁻¹¹. However, the recovery of Al, Fe, Ca, Mg, Si, Na, K and Ti from fly ash using classification, magnetic separation followed by leaching using HNO_3 , HCl and H_2SO_4 were studied¹²⁻¹⁴.

For the purpose of high value application some fly ashes of different thermal power plants of Odisha, India have been studied for the extraction of aluminium¹⁵⁻¹⁶. The present investigation is based on the leaching of fly ash by using different mineral acids such as HCl, HNO_3 , $\text{o-H}_3\text{PO}_4$ and H_2SO_4 . The effect of variation of acid concentration on the leaching behaviour of fly ash is investigated at a constant leaching time of 4h at a boiling temperature of the corresponding acids. A comparative study on the magnitude of aluminium and other metals extraction using these leachants has also been represented.

Materials and method

Sample collection and characterisation

For the above study, fly ash samples are collected from the electrostatic precipitator of Talcher Thermal Power Station (TTPS), Odisha. These samples are characterized physicochemically for their mineralogy, morphology, particle size and chemistry using XRD (Philips PW 1400), Scanning Electron Microscope (Jeol JSM), Malvern particle size analyser (UK, Model-3600), Atomic Absorption Spectrophotometer (Varian Spectra AA-20), Inductively Coupled Plasma Spectrophotometer (Perkin-Elmer, Plasma-400) and Flame photometer (Elico, Model CI-22D). AnalR grade reagents are used for the chemical analysis and commercial grade mineral acids are used for the leaching experiments.

Leaching using mineral acids (HCl, HNO_3 , $\text{o-H}_3\text{PO}_4$, H_2SO_4)

In each leaching experiment, 100g of fly ash were digested with different concentrations of mineral acids at its boiling point in a solid:liquid ratio 1:1. The mixture was taken in a one-liter flask, boiled at the boiling point on constant stirring. Boiling continued up to 4h with time-to-time addition of hot water to maintain the desired solid liquid ratio. Finally, the mixture was boiled to dryness. Then the mixture was further extracted with 500-ml hot distilled water followed by boiling and filtered through a G3 buchner funnel using suction pump. The residue was washed twice with hot water and washings were collected with leach liquor. The leach liquor was evaporated and made up to a known volume (1 liter) and analysed for various elements like Al, Fe, Ti, Ca and Mg. The residue was washed up to neutral and dried at 110 °C.

Mineralogy (XRD) -Phase Assemblies

The phase assemble study by X-ray diffractogram for fly ash was carried out on PHILIPS PW 1400, using $\text{CuK}\alpha$ radiation. Similar parameters like $\text{kV} = 40$, $\text{mA} = 20$, Chart speed = 1 cm/min. , Gonio meter speed = $2^\circ/\text{min.}$ are maintained during the analysis to get the uniform information about the samples. The JCPDS data files are used for matching of characteristic reflection peaks (d° values) to identify different minerals.

Morphology (SEM)

The morphology and crystal structure of fly ash was done by a Scanning Electron Microscope (JEOL JSM 840). Fly ash samples dried at 110°C were mounted on an aluminium stub with the help of adhesives to make the surface conducting and then sputtered in vacuum chamber at high voltage. These conducting samples were then observed under the microscope.

Particle Size

The particle size distribution pattern of fly ash samples were determined with the help of a MALVERN Particle size analyzer (UK, Model 3600) where the measurement is based on the He-Ne laser. Apart from the above it gives different mean sizes of the particle size distribution and also specific surface area of the particles in terms of m^2/cm^3 .

Atomic Absorption Spectrometry (AAS)

The quantitative analysis of Ca, Mg, Fe, Mn and Ni in fly ash and leached liquor are determined by Atomic Absorption Spectrometer (VARIAN-SPECTRA AA-20 plus), where suitable hollow cathode lamps are used for the absorption of characteristic lines by the analyte, and the concentration vs absorbance plot obeying the Beers-Lambert's law counts the concentrations of the corresponding elements¹⁷.

Inductively Coupled Plasma Spectrometry (ICP-OES)

The elements namely Al, Ti, Si and B present in fly ash and leached liquor were done by ICP (PERKIN ELMER, PLASMA-400) Spectrophotometer. A high temperature (10,000K) Argon Plasma source is used for the excitation of the element and the concentrations are measured as a function of the emission intensity of the emission spectra generated by the particular element¹⁸.

Flame Photometry

The concentration of Na and K was determined by Flame Photometer (ELICO - MODEL CL 22D) using particular filter for the wavelength of particular element. Calibration curves were made with suitable standard solutions made from AR KCl and AR NaCl. Then the concentration of the test sample was calculated by using the methods as outlined by Vogel¹⁹.

Results and Discussion

The chemical characterization of TTPS fly ash sample shows the presence of major metallic constituent like Si, Al, Fe, Ti, Ca, Mg, Na and K. These metals are present in fly ash as metal oxide and their concentration are given as SiO₂: 59.490, Al₂O₃: 29.091, Fe₂O₃: 3.901, TiO₂: 1.902, K₂O: 0.722, CaO: 0.993, MgO: 0.332, Na₂O: 0.129 and P₂O₅: 0.619. Results from XRD shows the presence of both crystalline and amorphous phases in fly ash. The crystalline phases identified are quartz, mullite, haematite, magnetite, sillimanite and spinel. Similarly, the amorphous phase consists of spherical micron sized particles composed of mullite enclosed in a glassy matrix. The external glass is enriched in Ca, Mg, Fe and Al. The interior glass matrix is composed primarily of Si and a major portion of the total Na and K. Thus, leaching is primarily controlled by dissolution of the external glass matrix after rapid dissolution of surface salts in amorphous phase along with the dissolution of crystalline phases.

Leaching with HCl

In order to study the effect of HCl on the extraction of aluminium from fly ash different concentration of HCl are chosen. The metal present in flyash leached out and their concentrations in the leached liquor are given in Table 1. It is observed from the data that leaching of iron is more than the other elements present in fly ash. The percentage extraction of aluminium increases slightly in the concentration range of 2N to 11.3N from 2.3 to 4.03% where as the iron extraction is 17.4 to 34.82%. This lower in concentration of aluminium may be due probably to the formation of relatively insoluble aluminium, silicon compounds. The iron in the ash is predominantly in the form of Fe₂O₃ and Fe₃O₄ produced by the oxidation of pyrite during the combustion process, whereas aluminium is present in the structure of crystalline phases and so is dissolved more slowly, in case of HCl as leaching agent. The

dissolution of aluminium and iron in fly ash results AlCl₃ and FeCl₃ as suggested by Livingston et al²⁰. Although the concentration of the other metal values in leached liquor shows increasing trend but their concentrations are not appreciable. Hence, iron is the most preferred metal ion for a leachant like HCl²⁰.

Table 1 : Metal extraction from fly ash with varied HCl concentration

(Solid : Liquid =1:1, metal concentration in gms / lit)

HCl Concentration	Al	Fe	Ti	Mg	Ca	Na	K	% Extraction of Al	% Extraction of Fe
2N	0.355	0.475	0.014	0.047	0.024	0.012	0.020	2.30	17.40
4N	0.555	0.581	0.015	0.068	0.024	0.013	0.029	3.60	21.29
6N	0.595	0.884	0.015	0.068	0.024	0.015	0.030	3.86	32.39
8N	0.593	0.906	0.016	0.070	0.028	0.017	0.030	3.85	33.20
10N	0.608	0.925	0.017	0.071	0.030	0.017	0.031	3.95	33.88
11.3N	0.620	0.950	0.020	0.072	0.031	0.024	0.034	4.03	34.82

Leaching with HNO₃

Table 2 represents the concentration of metals leached out from fly ash samples using HNO₃. It is observed that extraction of aluminium shows a slight increasing trend from 2.17 % to 3.80 % in the concentration range of 2N to 16N HNO₃. The dissolution of iron also shows an increasing trend. At an initial concentration of 2N HNO₃ extraction of iron is 2.62 % whereas at 16N extraction value is 19.4%. The aluminium is taken into aqueous solution, as Al³⁺ and lies as Al(NO₃)₃ in the solution²¹.

On comparison between HCl and HNO₃ it is found that the Al and Fe dissolution using HNO₃ are significantly lower than those obtained using HCl²⁰. As the extraction of iron is more than extraction of aluminium in both the acids, HCl and HNO₃ as leachant are effective in recovering iron from fly ash than aluminium. Iron occurs as iron oxides, i.e. Fe₂O₃, Fe₃O₄ and does not present in matrix, therefore are easily dissolved out. The dissolution of aluminium is less due to its presence as aluminium, silicon crystalline phase. Other elements like Ti, Ca, Mg, K, Na and their salt, which generally lies on the surface of the glassy phase, are also dissolved to some extent.

Table 2. Metal extraction from fly ash with varied HNO₃ concentration

(Solid: Liquid =1: 1, metal concentration in gms/ lit)

HNO ₃ Concentration	Al	Fe	Ti	Mg	Ca	Na	K	% Extraction of Al	% Extraction of Fe
2N	0.335	0.072	0.012	0.034	0.013	0.004	0.019	2.17	2.62
4N	0.410	0.204	0.015	0.043	0.016	0.005	0.026	2.66	7.45
6N	0.460	0.293	0.019	0.045	0.017	0.005	0.027	2.99	10.71
8N	0.475	0.335	0.020	0.049	0.020	0.005	0.027	3.08	12.27
10N	0.460	0.378	0.022	0.068	0.027	0.007	0.029	2.99	13.87
12N	0.475	0.454	0.026	0.077	0.028	0.016	0.030	3.08	16.63
14N	0.500	0.508	0.029	0.086	0.032	0.016	0.030	3.25	18.60
16N	0.585	0.530	0.030	0.105	0.029	0.018	0.031	3.80	19.40

Leaching with ortho-H₃PO₄

In order to study the effect of H₃PO₄ on the extraction of metals from fly ash varying concentration of H₃PO₄ are chosen. Aluminium along with other elements like Fe, Ca, Mg, Na and K are leached out and their concentrations are given in the Table 3. The extraction of aluminium at an initial concentration (4N) of ortho-phosphoric acid is 1.68% and the percentage of extraction increases gradually with the increase in concentration of acid.

Using 41.1N ortho-phosphoric acid, the extraction value of aluminium reaches 32.98%. In all the cases, considerable amount of iron is extracted and the extraction value varies from 4.51 % to 38.59 %. On comparison with HCl and HNO₃, ortho-phosphoric acid is found to be strong and effective leachant, which enters into the matrix of the aluminium silicate^{22,23}. Results on the extraction of various metal values show a gradual increasing tendency with the increase of acid concentration except calcium and magnesium. The metal in fly ash dissolve forming mono metal phosphate²² whereas the increasing concentration of Ca and Mg may be attributed due probably to the formation of di- and tri calcium and magnesium phosphates.

Table 3 : Metal extraction from fly ash with varied H₃PO₄ concentration

(Solid : Liquid =1:1, metal concentration in gms/lit)

H ₃ PO ₄ Concentration	Al	Fe	Ti	Mg	Ca	Na	K	% Extraction of Al	% Extraction of Fe
4N	0.259	0.123	0.001	0.064	0.046	0.006	0.006	1.68	4.51
8N	0.370	0.375	0.004	0.064	0.046	0.006	0.007	2.40	13.74
12N	0.529	0.791	0.012	0.048	0.034	0.007	0.008	3.43	28.99
16N	0.786	0.882	0.019	0.035	0.025	0.009	0.011	5.10	32.29
20N	0.830	0.905	0.034	0.034	0.025	0.010	0.011	5.38	33.16
24N	1.220	0.923	0.049	0.032	0.023	0.013	0.011	7.91	33.83
28N	1.251	0.979	0.051	0.030	0.021	0.015	0.021	8.13	35.88
32N	1.650	0.990	0.051	0.028	0.020	0.016	0.022	10.70	36.25
36N	2.451	0.997	0.084	0.027	0.019	0.016	0.018	15.91	36.50
38N	2.881	1.008	0.084	0.021	0.019	0.018	0.032	18.69	36.91
40N	3.651	1.051	0.097	0.025	0.018	0.021	0.025	23.69	38.48
41.1N	5.082	1.053	0.113	0.024	0.017	0.024	0.035	32.98	38.59

Leaching with H₂SO₄

The dissolution of metals with different concentration of sulphuric acid are summarized in Table 4. The concentration of different metals have an increasing tendency with the increasing concentration of the acid used²⁴. It is observed that there is a gradual increase in aluminium extraction with the increase in acid concentration. The extraction of aluminium with 3N H₂SO₄ is found to be 2.66% whereas at 36N H₂SO₄ it is 41.3%. Similarly, iron extraction varies from 13.33% to 53% with 3N to 36N H₂SO₄, respectively. In all the cases, along with aluminium and iron, the other metals Ca, Mg, Na, K and Ti are also leached out to some extent. Aluminium and iron dissolves as Al³⁺ and Fe³⁺ in sulphuric acid whereas calcium precipitates as CaSO₄²⁴ causing particles inhibition effect. It is assumed that initial dissolution of aluminium is from glassy phase only and more stable quartz-mullite phase dissolves in the latter stage²⁵.

Table 4: Metal extraction from fly ash with varied H₂SO₄ concentration

(Solid : Liquid =1:1, metal concentration in gms/lit)

H ₂ SO ₄ Concentration	Al	Fe	Ti	Ca	Mg	Na	K	% Extraction of Al	% Extraction of Fe
3N	0.410	0.364	0.014	0.040	0.010	0.002	0.024	2.66	13.33
6N	0.588	0.491	0.044	0.043	0.012	0.002	0.022	3.82	17.99
9N	0.694	1.032	0.101	0.078	0.012	0.003	0.033	4.51	37.80

12N	1.163	1.077	0.111	0.089	0.012	0.002	0.039	7.55	39.45
15N	1.574	1.133	0.109	0.104	0.013	0.004	0.042	10.22	41.50
18N	2.358	1.138	0.117	0.132	0.013	0.005	0.053	15.31	41.69
21N	2.822	1.179	0.123	0.233	0.013	0.006	0.049	18.32	43.19
24N	3.174	1.199	0.129	0.350	0.013	0.006	0.063	20.61	43.92
27N	4.040	1.247	0.138	0.497	0.013	0.008	0.075	26.23	45.68
30N	4.964	1.301	0.177	0.520	0.014	0.009	0.077	32.23	47.66
33N	5.406	1.403	0.228	0.532	0.015	0.010	0.082	35.10	51.39
36N	6.362	1.447	0.258	0.530	0.017	0.012	0.100	41.30	53.00

Post leached Ash studies

The post-leached samples have been characterized for particle size distribution, XRD and SEM²⁶⁻³⁰ in order to compare and detect the changes in the content of many strategic metals already leached out. Leached residues are washed thoroughly with distilled water, dried at 110°C, and then subjected to physical and mineralogical characterization.

SEM Studies

The SEM photomicrographs of the pre and post leached TTPS fly ash are given by Fig. 1 a&b. These photographs depicts the leaching behaviour of fly ash with of H₂SO₄. SEM photomicrographs of pre leached fly ash show a smooth surface with an abundance of extremely small (submicron) dust like flecks located on or slightly embedded in the surfaces. Ash particle contains an exterior glass hull with some salt grain deposits, then a near surface layer of crystalline phases i.e. mullite and an interior glass matrix³¹. In the post leached ash (Fig. 1 b) the photograph shows corroded surface, predicting the acid attack on the smooth surface during leaching. The attack of H₂SO₄ on the aluminosilicate matrix is prominent. The presence of some unattacked particle suggests that the total recovery of metal values can be attributed to the crystalline as well as amorphous phase.

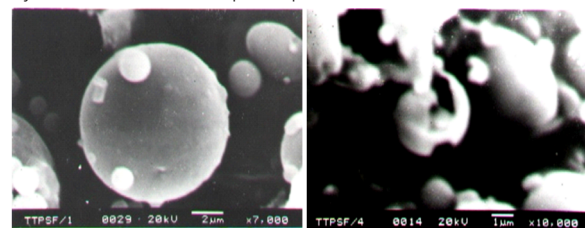


Fig 1 a&b. Pre leached fly ash and post leached fly ash with sulphuric acid Particle size

The particle size and specific surface area of the pre and post leached ash using HCl, HNO₃, o-H₃PO₄ and H₂SO₄ at a ash : acid ratio (1:1) are described in Table 5. The particle size of the leached ash using sulphuric acid are less than the parent fly ash i.e. 19.84 µm, whereas the particle size of post leached fly ash using nitric, hydrochloric and phosphoric acid are more, mostly in D₅₀ micron size than the parent one. In case of nitric and hydrochloric acid, the reason for higher particle size and lower specific surface area may be due to formation of some insoluble compounds. On the other hand, orthophosphoric acid forms monometal phosphate (with an H₂PO₄²⁻ anion), dimetal phosphate (with an HPO₄²⁻ anion) and trimetal phosphate (with an PO₄³⁻ anion)²¹. Of which di- and tri substituted phosphates of alkaline earth metals have negligible solubility compared to other metal salts of mono-, di- and trisubstituted phosphates. This gives rise to a larger particle size and thus smaller is the specific surface area.

Table 5 :Particle size distribution of Pre and Post leached fly ash

Particle Size Characteristics	TTPS (Pre leached)	TTPS (Post leached)			
		HCl (1:1)	HNO ₃ (1:1)	H ₃ PO ₄ (1:1)	H ₂ SO ₄ (1:1)
D50, Micron	19.84	17.51	23.48	18.67	11.32
D90, Micron	58.68	76.26	65.85	66.74	43.84
D10, Micron	5.89	3.43	3.81	5.13	3.21
Sp.Surface Area, m ² /cc	0.5509	0.6308	0.5017	0.4746	0.8297

But with sulphuric acid the aluminium (alumino silicate) along with

other metals present in fly ash are dissolved. Thus, the particle size decreases with increasing specific surface area. It is also suggested that acid leaching removes most of the amorphous phase that surrounds crystals of the insoluble phases such as mullite and quartz. It is seen that the leached ash has a relatively high specific surface area. By dissolution of amorphous phase, high surface area crystalline phases are exposed³². The dissolution of the crystalline phases is further supported by XRD.

X-Ray Diffraction Studies

Comparing the leaching data of HCl, HNO₃, o-H₃PO₄ & H₂SO₄ given in Table 1, 2, 3 and 4, sulphuric acid is found to be the best leachant with respect to aluminium extraction. This can be further confirmed by the X-ray diffractogram comparison of the post-leaching fly ash residues. The mineralogy of pre leached and post leached residues of HCl, HNO₃, o-H₃PO₄ and H₂SO₄ of TTPS fly ashes are studied by X-ray diffractogram and are shown in Fig. 1. The mullite, magnetite and hematite peaks at (2θ - 26.3, 35.1 and 33.1) differ from each other. In HCl & HNO₃, the mullite peak (2θ - 26.3) are not deformed and are same as the parent fly ash whereas in ortho phosphoric and sulphuric acid the mullite peak height (intensity) decreases. Some other mullite peaks (2θ - 54.5) shows similar trend, which differs from the singlet peak obtained for the parent one. At 2θ - 45.85 the quartz peaks of post leached residues of o-H₃PO₄ and H₂SO₄ are enriched due to increase in concentration through loss of other elements in the leached sample. Similarly, at 2θ - 26.75 the intensity of the quartz peaks are found to be more distinct than the parent one. Considering the leachants HCl and HNO₃, it is found that the only hematite and magnetite peaks are affected keeping the mullite peak intact, which confirms that extraction of iron is more than the aluminium. Thus, observing the change in peak intensity in the diffractogram it can be proved that H₂SO₄ is the best leachant when aluminium extraction is considered as evident by the published work¹⁶.

However, the slow dissolution of aluminium from the more stable quartz-mullite phase and rapid dissolution of glassy phase were strongly supported by SEM and XRD.

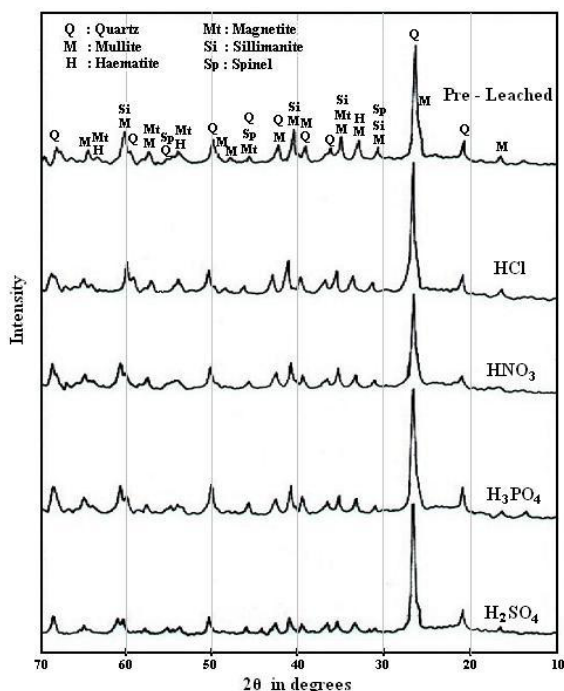


Fig. 2 : X-Ray Dffractogram of TTPS Fly ash leached with different acids

Conclusion

Leaching behaviour of fly ash with different mineral acids has been studied with respect to percentage of extraction of aluminium and iron. The direct acid leaching at low concentration and ambient temperatures is not satisfactory for high recoveries of these metals

from the fly ash. The rate of extraction of Al and Fe increased sharply with increasing acid concentration. The rate of extraction of Aluminium is slightly higher using HCl as the leaching agent than with HNO₃ at the same concentration but both the acids gives satisfactory result with respect to extraction of iron. However, o-phosphoric acid and sulphuric acid are proved to be suitable leaching agents for metal extraction. As the concentrations of the acids were varied from low to concentrated reagent, low to moderate recoveries of metal values from fly ash by direct acid leaching is not surprising since fly ash consists primarily of iron and aluminium silicates together with silica fused into a refractory glassy mullite material. However sulphuric acid is found to be effective leachant with respect to aluminium extraction due to dissolution of the stable glassy mullite phase. The leachability of metals from fly ash depends on the nature of leaching medium, solid: liquid ratio, temperature and leaching time. By acid routes silica remains substantially insoluble where as the aluminium is taken into aqueous solution as Al³⁺. The acid leaching is not selective for aluminium and iron as Ti, K, Na, Mg and Ca generally dissolve simultaneously. For any kind of specific use the impurities other than the desired metal need to be removed through a suitable process.

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