

Metal Leaching Potential in Coal Fly Ash

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Abstract Thermal power plants are the major source of electricity generation in India and most of them use pulverized coal as the fuel producing enormous quantities of coal fly ash every year. The method of disposal adopted is by wet sluicing in on-site fly ash ponds. This disposal in the form of dilute slurry has a high potential for leaching into the surrounding soil and groundwater. The coal fly ash contains trace metals like As, Cr, Zn, Cd, etc which are toxic in nature and thus, the wet disposal of coal fly ash has serious environmental concerns. This paper assesses the leaching potential of coal fly ash from six thermal power plants in Maharashtra, India. The maximum leachable quantities of some trace metals present in coal fly ash are computed by a Sequential Extraction Procedure (SEP) and results are compared with values obtained by Toxicity Characteristics Leaching Procedure (TCLP).

Keywords Coal Fly Ash, Leaching, Metals, SEP, TCLP

1. Introduction

India has 211 billion tonnes of coal reserves. Indian coal used in thermal power plants is of low grade quality and has an ash content of 40 to 50% [1]. The expected generation of fly ash in India is more than 175 million tonnes by the year 2012 [2]. The minerals in coal decompose during combustion and their alterations result in the formation of ash, which has silicates as its major component, while unburnt carbon/char constitutes a small fraction. The mineralogy of the coal governs the crystalline phases in fly ash, which include oxides of aluminium and iron, feldspars, quartz, mullite and gypsum. Fly ash also contains organic fractions like coke and char particles. Fly ash is enriched in most elements that are found in coal except for the most volatile elements, such as Hg [3]. The fly ash collected by electrostatic precipitators is carried from silos to storage ponds by flushing along with water through drains made especially for this purpose, the method referred to as 'hydraulic conveying'. This water is recycled for further conveying of fly ash from silos and this process results in increased concentration of metals in the ponds due to accumulation [4]. Fly ash and bottom ash and water are usually mixed in ash: water ratios varying from 1:4 to 1:20 [5]. The ash ponds contain fly ash and water mix in form of a dilute slurry and leaching of metals from this dilute slurry is a function of its pH, which itself is a dynamic parameter. pH can change due to physico-chemical reactions taking place in the pond itself or due to addition of

rain water which has an acidic pH of about 5.7 on interaction with atmospheric CO₂ [6]. Also, the leaching is affected by liquid to solid (L/S) ratio of the slurry. Maximum concentrations of many elements were found at lower L/S ratio and it decreased with increasing L/S ratio [7]. Composition of the leachate waters depends on many factors ranging from treatments given to coal (desulfurization, lime addition processes etc) to engineering process design (conditions of combustion systems and flue gas processes). It is also a function of the surface of fly ash [8]. The elements in the aluminosilicate glass phase of fly ash are released in lower concentrations initially and dominate in the leachate of the weathered fly ash. In the initial stages, the elements adsorbed on the surface of fly ash particles are prominent in the leachate [4]. The combustion of coal in thermal power plants may result in concentration of certain elements in fly ash, designated as enriched elements, such as Cd, Cr, Pb, and Zn. These elements have a greater tendency to leach out from the solid phase (fly ash). These toxic trace elements display subsequent enrichment in concentration from coal to bottom ash and to fly ash [9]. This enrichment in concentration can reach to about 100 times the metal concentration in the coal [10]. Initially when fly ash comes in contact with water, the alkaline elements present on its surface will tend to dissolve rapidly and move into solution. But, with subsequent increase in solution pH and element concentration, reprecipitation of elements may occur to form more stable secondary solids [11]. Under leaching conditions, the mobility of different elements from coal ash is critically dependent on the pH developed within the ash-water system and that the effect of the pH of the natural ash on the pH of the ash-water system decreases on increasing the dilution of the ash [12]. Oxyanions like B, As,

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and Se have high solubility and so these tend to leach more at both low and high pH values, while cations like Ca and Sr show decrease in solubility when pH increases[13].

2. Materials and Methods

The fly ash samples were collected from the dust hoppers of electrostatic precipitators of the six thermal power plants in Maharashtra, India. The samples named as FA-1, FA-2, FA-3, FA-4, FA-5 and FA-6 were characterized on the basis of particles size, surface area, mineralogy by X-Ray Diffraction (XRD), metal oxides content by X-Ray Fluorescence (XRF), leachable metal concentrations by Sequential Extraction Procedure (SEP) and Toxicity Characteristic Leaching Procedure (TCLP) using Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES). All the samples were sieved finer than 250 μm and oven dried at 105°C for 24 hours before analysis. All glassware (Borosil make) were regularly washed using chromic acid, neutralized with dilute alkali, washed using tap water and ultra pure water and oven-dried after each experiment. Stock solutions of all reagents of 1000 mg/L were prepared using Merck Chemicals, India and multi element standards of 1000 ppm solution were purchased from the VGH laboratory, USA and diluted as per experimental requirements. Metal free nitric acid, hydrochloric acid and acetic acid and metal free ultra pure water generated using Ultraclear water kit TWF EDI UV TM (Siemens, Singapore) were used for all experiments.

2.1. Particle Size Distribution

The particle size analyses of coal fly ashes were carried out with the help of Beckman Coulter Laser Diffraction Particle Analyser (LS 13320, Japan). A well-mixed slurry using 1 g of fly ash sample and 20 mL of ultrapure water was kept in the Beckman Sonication Control Unit, where the aqueous liquid module was capable of suspending samples in the range of 0.04 to 2000 μm . Samples were diluted to omit interference by re-scattering.

2.2. Specific Surface Area

The specific surface area of the fly ash samples was determined with the help of BET surface area analyzer (Smart Instruments, India) based on single point. First step in this experiment was to regenerate the sample (1 g of sample) to remove the moisture or adsorbed gases. Regeneration was done at 110° C for 2 hr for the sample. In second step, the regenerated sample was placed in sampler, which was connected to the instrument for the experiment. Initially the sample was dipped in liquid nitrogen. This led to adsorption of N₂ on the sample from the flow of gas mixture of He and N₂. After this adsorption was over, the sample was dipped in water which started the desorption process. The adsorbed N₂ got released in the gas flow. The quantity of gas released was measured with the help of thermal conductivity detector (TCD) and was then integrated with the help of electronic circuit in terms of number or counts (NCs). The instrument is

then calibrated by injecting known quantity of N₂. All these measurements are used to calculate the surface area of the sample. The software package provided with instrument is user friendly and guides the user about every procedure. It does all the calculations internally and displayed the final result on the computer screen.

2.3. Mineralogical Composition

Mineralogical investigations of coal fly ashes were carried out by powder X Ray Diffractions (XRD) (Rigaku Geigerflex, Japan), coupled with PW 1729 X-ray generator using CuK α radiation. All samples were run at a tube voltage 20 kV and 30 mA. The goniometer and chart speed were maintained at 0.05 mm/2 θ and 10 mm/2 θ , respectively. All samples were dried at 110°C before analysis.

2.4. Elemental Composition

The elemental composition of the coal fly ash samples was obtained with the help of Philips X-Ray Fluorescence Spectrometer (2404, Netherlands), which determines all major oxides present in the sample. 4 g of oven-dried, finely ground coal fly ash sample and 1 g of micro crystalline methyl cellulose were mixed uniformly with isopropyl alcohol and kept for slow drying under a 200 W infrared lamp. This dried sample was made as a pellet by filling in an aluminium dish and was compressed under a load of 1-2 ton for 1 minute with the help of a hydraulic jack. The compressed pallet was run in the XRF setup for computing the composition as oxides of elements as percentage by weight of the ash sample.

2.5. Sequential Extraction Procedure

The fly ash samples were subjected to a leaching procedure using leaching solutions sequentially in order of increasing aggressive nature and the concentrations of different metals leached during each step were calculated using ICP-AES[14]. 0.5 g of dried fly ash samples were mixed with 50 mL of the corresponding leaching solution in a centrifuge tube and set in an end-to-end agitator (Trishul Equipment, India) specifically fabricated for SEP at 30 rpm for specified time period. Thereafter the samples were centrifuged at 10000 rpm for 10 minutes. The supernatant was decanted, filtered with a 0.2 μm PTFE filter and then acidified to 2% (by mass) using concentrated nitric acid and preserved for analysis by ICP-AES. The solids remaining after the centrifugation for one step were then contacted with the next leaching solution in the sequence. The concentrations of metals of interest in all leaching solutions for all fly ash samples were obtained from ICP-AES.

The leaching solutions used and the corresponding time for agitation are compiled in Table 1.

2.6. Toxicity Characteristic Leaching Procedure

The USEPA SW 864 method 1311[15] was used for testing the leachability of the fly ash samples. Initial tests for determination of the extraction fluid to be used for analysis

were done by obtaining the pH of the fly ash – ultrapure water mix in the ratio of 1:20. The corresponding extraction fluid used for the analysis was acetic acid having pH of 2.88. For analysis, 0.5 g of fly ash sample was mixed with 20 mL of extraction fluid in a 50 mL centrifuge tube and put in an end-to-end rotary agitator for 18 hours at 30 rpm and then suspension was centrifuged at 10000 rpm for 10 minutes. The supernatant was filtered through a 0.2 μm PTFE filter and then acidified to 2% (by mass) using concentrated nitric acid and preserved for analysis by ICP-AES. The solids remaining from the centrifugation were discarded.

Table 1. Steps for SEP.

Steps	Phase for Leaching	Leaching Solution	Duration for Agitation
1	Water Soluble (WS)	Ultrapure Water	4 hr agitation at ambient temperature
2	Ion Exchangeable (IE)	1.0 M Ammonium Nitrate	4 hr agitation at ambient temperature
3	Acid Soluble (AS)	0.11 M Acetic Acid	24 hr agitation at ambient temperature
4	Reducible (RD)	0.158 M Dithionite, 0.3 M Citrate and 1.0 M Bicarbonate	24 hr agitation at ambient temperature
5	Residual Solids(RS)	12.5 mL concentrated Nitric Acid and Hydrochloric Acid(4:1 v/v)	20 minutes in Microwave Acid Digester at 210°C

2.7. Metal Analysis

Concentrations of trace metals in samples from SEP, and TCLP were obtained using Jobin Yvon Horib ICP-AES (ULTIMA 2000, France). Prior to analysis the samples were diluted with 2% nitric acid solution. Samples from the SEP were diluted 1:2 for the water soluble step, 1:10 for the acid-soluble step, and 1:20 for the other three steps. TCLP samples used a 1:10 dilution factor. Calibration standards were analyzed in the same matrices as the samples and the extractions for SET and TCLP were carried out in duplicate. Table 2 shows the operating conditions of ICP-AES.

Table 2. ICP-AES Instrumental and Operational Conditions.

Parameters	Value/Type
RF generator power	1.0 kW
RF frequency	40 MHz
Plasma	12.0 L/minutes
Auxillary	0.75 L/minutes
Nebulizer type	V-type
Nebulizer make	Glass concentric
Nebulizer pressure	2.90 bars
Nebulizer flow rate	0.85 L/min
Spectrometer	Monochromator
Analyzer	PMT
Integration time	3 Seconds
Replicates	2

3. Results and Discussions

The results obtained by the experiments mentioned in previous section are discussed below.

3.1. Particle Size Distribution

The results of the Particle Size Distribution Curves for the samples of fly ashes are tabulated in Table 2.

It can be seen that the mean diameters vary from 4.2 μm to 207 μm . The finer particles formed during combustion tend to coagulate and form larger particles, whereas larger particles too can destabilize during combustion to form smaller particles. Thus, the mean particle size of fly ash particles may be dependent on the combustion process of the thermal power plants.

3.2. Specific Surface Area

The specific surface area of the fly ash samples is shown in Table 3. The surface area varies between 0.89 and 1.2 m^2/g . The specific surface area is more if finer particles are in abundance in the sample. The surface area increases as percentage of finer particles increases in the sample. But as particle size increases, the shape ceases to be spherical and these irregular shapes tend to have larger specific surface area[9].

Table 3. Mean Particle Diameter and Specific Surface Area.

Sample	Mean Diameter (μm)	Specific Surface Area(m^2/g)
FA-1	5.2	1.00
FA-2	4.2	1.06
FA-3	75.3	0.99
FA-4	29.87	1.20
FA-5	96.17	0.89
FA-6	207	1.10

3.3. Mineralogical Composition

The results of XRD (Figure 1) for the fly ash samples indicated Quartz to be the most predominant element in all the samples and Mullite too was present in all the samples except FA-1 and FA-2.

3.4. Elemental Composition

The composition of fly ash samples by XRF(Table 4) indicated that all the samples were class F ashes as the SiO_2 content was very high and the CaO content was low.

The SiO_2 content was between 60 - 65% in all samples, being slightly less for FA-1, while the Al_2O_3 content varied between 20 - 30% for all fly ash samples. Fe_2O_3 content for the fly ash samples was between 4 - 6%. CaO content was between 1 - 2%, K_2O between 0.6 - 1.3% and TiO_2 between 1.2 - 1.7%. MgO , Na_2O , MnO and SrO were less than 1% with Na_2O being below detectable limits(less than 0.001%) in samples FA-5 and FA-6.

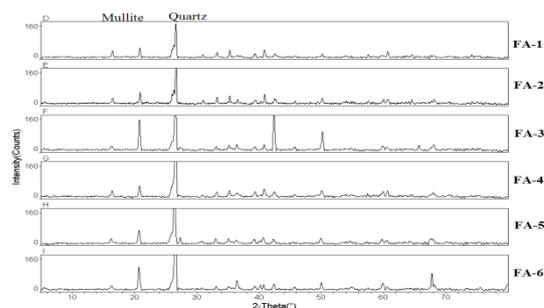


Figure 1. Mineralogical Composition of Fly Ash Samples using XRD.

The samples FA-3 and FA-6 having highest percentages of SiO₂ also showed the highest percentages for CaO and lower percentages for Al₂O₃. These two samples also have the highest peaks for Mullite and Quartz(Figure 1). Thus, the relative abundance of SiO₂ and CaO in coal fly ash may be a function of the Quartz and Mullite content, possibly exhibiting a direct dependence.

Table 4. Elemental Composition of Fly Ash by XRF.

Element Oxide	FA-1 (%)	FA-2 (%)	FA-3 (%)	FA-4 (%)	FA-5 (%)	FA-6 (%)
Al ₂ O ₃	30.098	27.074	23.711	26.672	25.824	23.926
SiO ₂	59.027	61.134	64.498	63.863	64.270	64.500
Fe ₂ O ₃	5.137	4.692	5.589	4.123	6.024	5.270
CaO	1.062	1.007	1.810	1.512	1.299	2.075
K ₂ O	0.989	1.061	1.159	0.678	0.843	1.227
MgO	0.677	0.428	1.204	0.593	0.432	0.642
MnO	0.043	0.046	0.057	0.047	0.057	0.055
Na ₂ O	0.272	0.229	0.234	0.286	0.000	0.000
SrO	0.035	0.036	0.036	0.031	0.026	0.031
TiO ₂	1.624	1.718	1.205	1.315	1.351	1.401
SO ₃	0.610	0.095	0.000	0.012	0.049	0.001

3.5. Metals Leaching in SEP

The samples showed different leaching trends for the different leaching solutions used for SEP. Some elements showed leaching for specific chemical phases. As, Cr, Mn, Zn, Cd, Ba, Pb and Co showed maximum leaching percentage in acid soluble (AS) phase. Se, Cd, Ni and V showed maximum leaching percentage in ion exchangeable (IE) phase, while Fe leached maximum in Residual Solids (RS) phase. Also, sample FA-1 exhibited maximum leaching in IE phase for As, Se, Cd, Ni, Pb, Co and V. The leaching trends exhibited by different elements for different leaching solutions are presented in Figure 2(a) to (m) and are summarised in Table 5.

Table 5. Leaching Trends for Elements during SEP.

Element	Probable Leaching Trends	Remarks/Deviations from Probable Trends
As	AS > IE > RS; WS and RD = 0%	FA-1: IE > RS; WS = 0%
Ba	AS > IE ~ RS (except FA-1); WS and RD < 5%	FA-1: RS > AS > IE > RD
Ca	Leaches in all phases Less in WS and RD	No definite trend for any sample
Cd	IE ~ AS > RD ~ RS; WS = 0	FA-1: IE > RD > RS FA-2: AS > IE > RD > RS
Co	AS > IE > RD ~ RS (except FA-1); WS = 0%;(RD ~ RS ~ 6%)	FA-1: IE >> RD ~ RS > AS
Cr	Maximum leaching in AS; AS > IE > RS	100% Leaching for FA-1 in WS; FA-3 and FA-4 in AS
Fe	RS > RD > AS; WS and IE = 0%	FA-2: RD > RS > AS FA-6: RS > AS > RD
Mn	AS > RS > IE ~ RD; WS ~ 0%;(IE ~ RD < 10%)	Similar trends for all samples
Ni	IE > RS; RD = 0%; WS and RS < 3%	Leaching for all Samples IE ~ 60% and RS ~ 40%
Pb	AS > IE > RD(except FA-1); WS and RS = 0%	FA-1: IE > RD(AS = 0%)
Se	IE > AS > RS; WS and RD = 0%	FA-1: IE > RS; AS = 0%
V	IE > AS > RD; WS=0%(except FA-3 and FA-4) RS=0%(except FA-1)	FA-1: IE > RD > RS FA-2: AS ~ IE > RD
Zn	Maximum leaching in AS; RD = 0%; IE = 0% except FA-2;	FA-1: RS > AS > WS FA-2: AS > IE > WS > RS

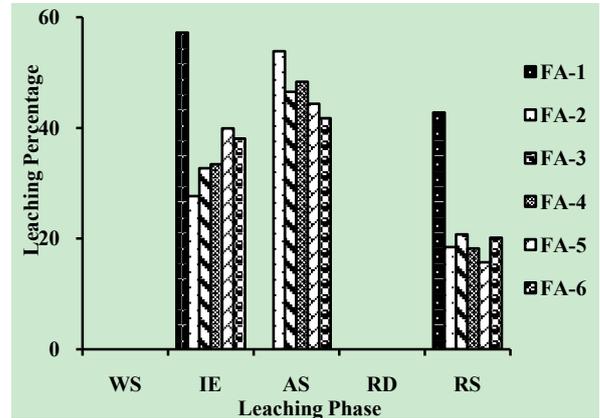


Figure 2(a). Leaching Trend for As in SEP.

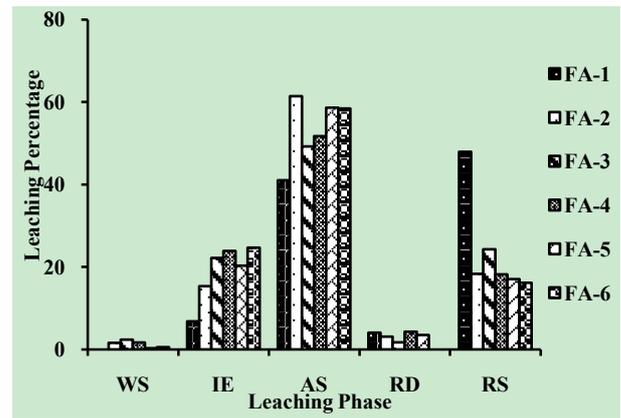


Figure 2(b). Leaching Trend for Ba in SEP.

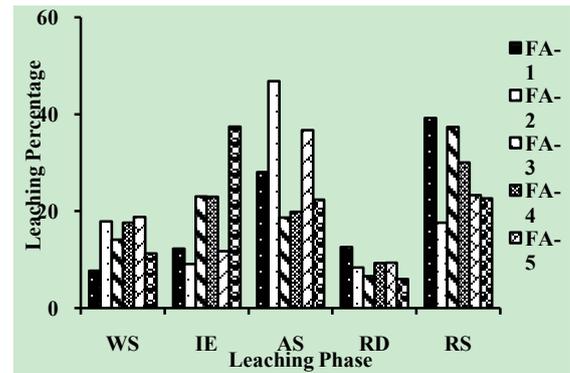


Figure 2(c). Leaching Trend for Ca in SEP.

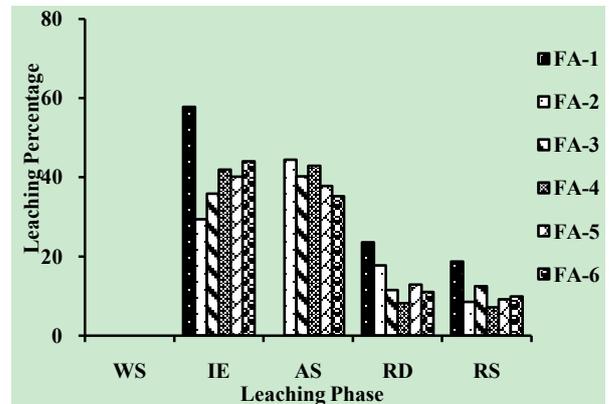


Figure 2(d). Leaching Trend for Cd in SEP.

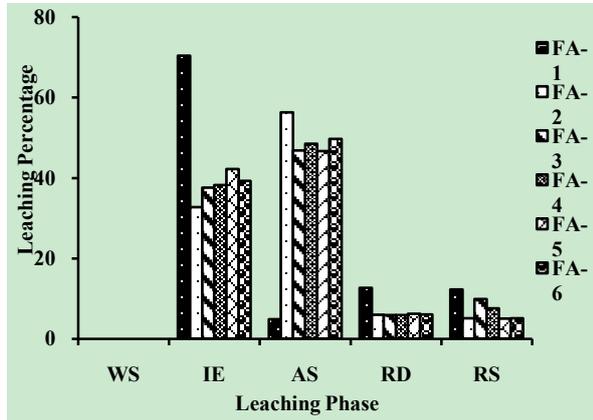


Figure 2(e). Leaching Trend for Co in SEP.

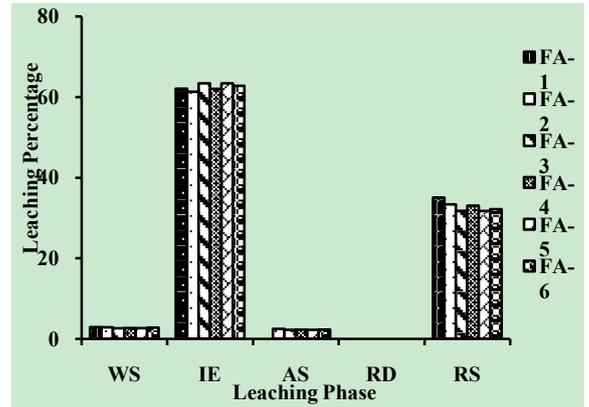


Figure 2(i). Leaching Trend for Ni in SEP.

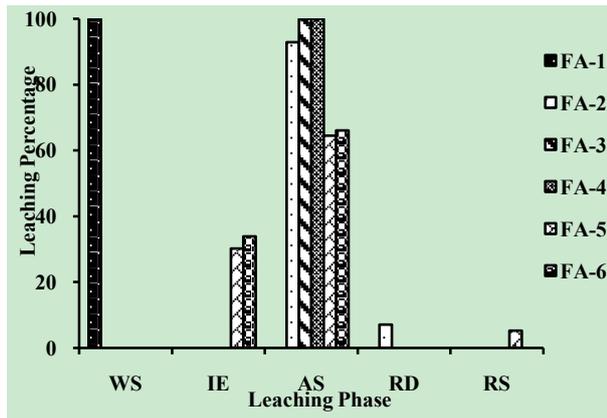


Figure 2(f). Leaching Trend for Cr in SEP.

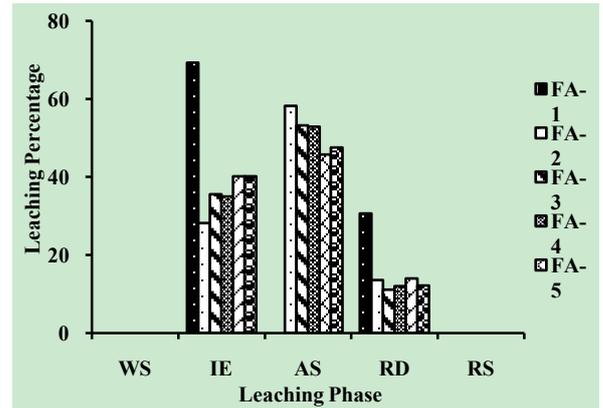


Figure 2(j). Leaching Trend for Pb in SEP.

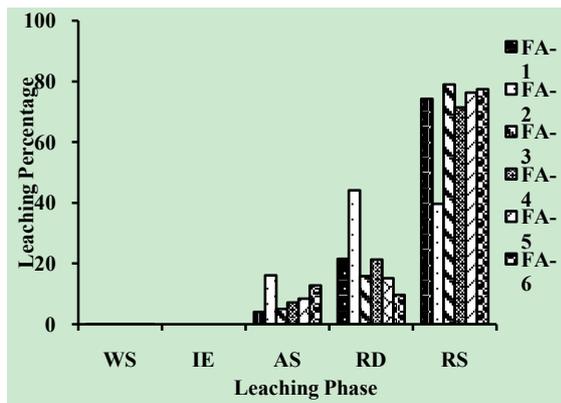


Figure 2(g). Leaching Trend for Fe in SEP.

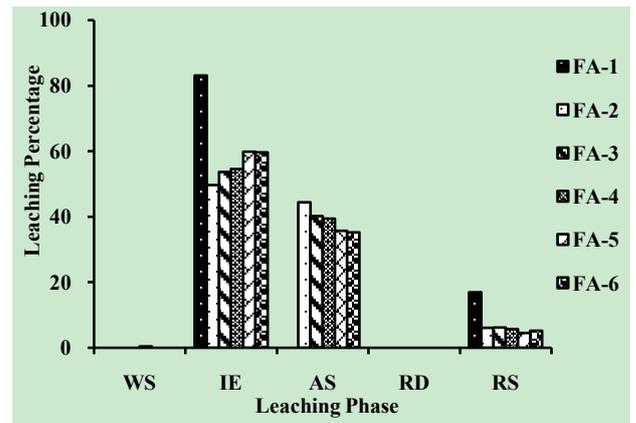


Figure 2(k). Leaching Trend for Se in SEP.

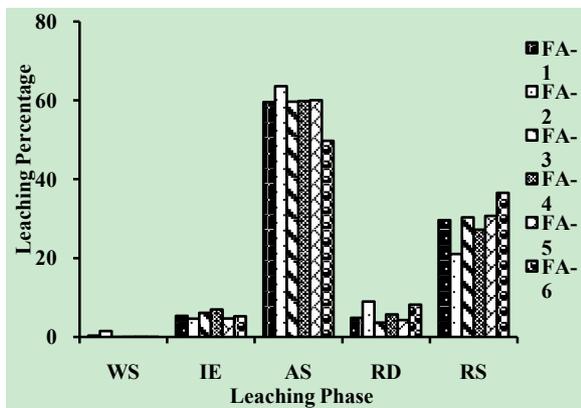


Figure 2(h). Leaching Trend for Mn in SEP.

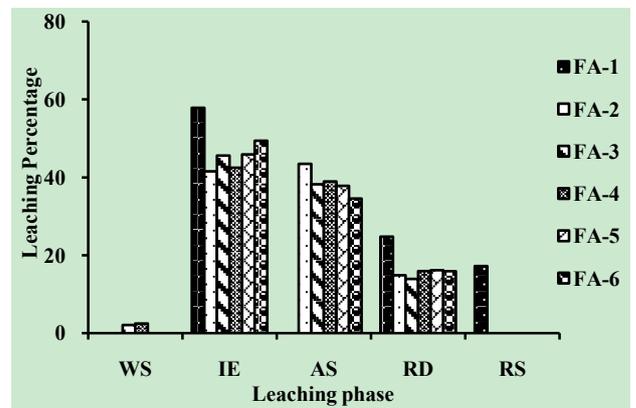


Figure 2(l). Leaching Trend for V in SEP.

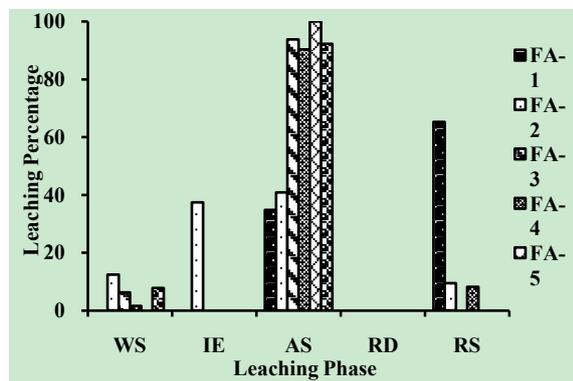


Figure 2(m). Leaching Trend for Zn in SEP.

The maximum leachable concentrations of the above mentioned elements computed in SEP are shown in Table 6.

Table 6. Total Leachable Concentration of Elements in SEP ($\mu\text{g/g}$).

	FA-1	FA-2	FA-3	FA-4	FA-5	FA-6
As	188.59	292.96	333.59	322.78	336.56	346.10
Ba	206.36	273.07	287.69	233.99	265.95	255.06
Ca	3929.45	5102.08	8362.60	5824.85	5312.36	8530.24
Cd	44.10	71.97	81.57	76.25	77.49	86.71
Co	63.26	130.83	136.60	134.33	140.77	134.52
Cr	53.95	60.20	56.66	50.46	88.61	79.46
Fe	10475.7	15702.3	15484.9	9711.04	16992.7	14081.6
Mn	328.55	391.90	540.28	383.93	535.42	449.13
Ni	11733.5	11878.4	12825.8	12616.1	12848.1	12405.9
Pb	253.52	518.84	570.46	571.74	658.18	636.50
Se	410.71	519.89	569.74	582.35	633.95	606.66
V	120.50	171.57	191.46	192.77	182.93	181.93
Zn	55.95	176.25	48.56	164.82	63.16	55.57

3.6. Metals Leaching in TCLP

The leaching trends observed for the six fly ash samples by using acetic acid of 2.88 pH, as leaching solution, are shown in Figure 3(a) and(b). Ca showed very high concentration in the range of 900 to 4000 $\mu\text{g/g}$. FA-3 has high leaching for Fe and Ba, showing high leaching concentrations of more than 100 $\mu\text{g/g}$, while FA-2 exhibits moderate leaching of Fe, Zn and Mn; FA-6 of Ba, Fe and Mn; and FA-1 of Ba.

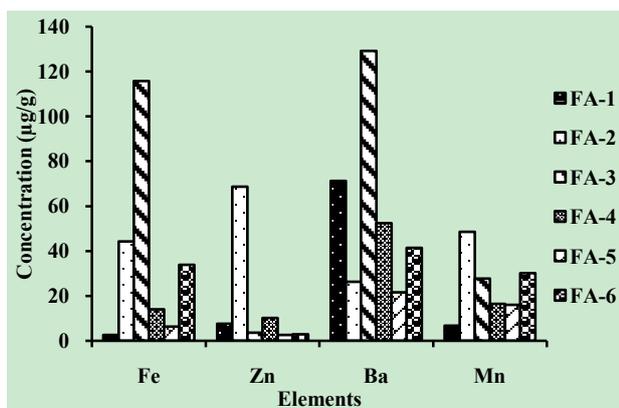


Figure 3(a). Leaching Trend in TCLP for Elements exhibiting Higher Concentrations in Extractant.

All fly ash samples show leaching of as in low concentrations but it may still have the potential to harm human health. V is leached from three fly ash samples, while Co and Cr are leached only by FA-3 and FA-6 respectively.

Ni, Se and Pb cease to show any leaching with acetic acid, which is not similar to SEP acid soluble results.

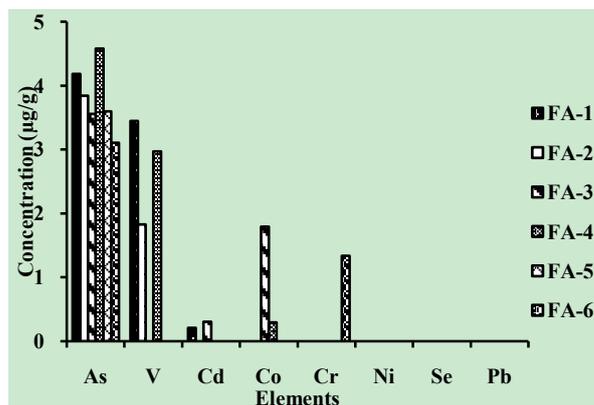


Figure 3(b). Leaching Trend in TCLP for Elements exhibiting Lower Concentrations in Extractant.

5. Conclusions

The elements in coal fly ash exhibit varying behaviour for different leaching conditions like leaching medium and pH. The fly ash may be enriched in Ca, Ni and Fe and show greater leachability in acidic or ion-exchangeable conditions. Fe is tightly bound to the ash and does not leach easily while Ca is highly soluble and leaches out in almost all mediums. Se, Cd and Ni leach out at less aggressive conditions in ion exchangeable conditions, while As, Cr, Cd, Pb and Zn leach under more aggressive conditions. Also, the SO_3 content of coal fly ash may influence the leaching behaviour of fly ash as exhibited by FA-1, which possesses relatively higher percentage of SO_3 and showed different leaching trends during SEP. Thus, toxic metal mobility is also influenced by the mode of occurrence of metals within the ash, especially for the metals which condense on the surface of the particles in the furnace[16].

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