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# Leaching Behavior of Selected Trace and Toxic Metals in Coal Fly Ash Samples Collected from Two Thermal Power Plants, India

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**Abstract** Studies on leaching behavior of metals associated with coal fly ash (FA) are of great concern because of possible contamination of the aquatic environment. In the present study, leaching behavior of metals (As, Se, Cr, Pb, V, Zn, etc.) in two different FA samples (FA1 and FA2) was investigated at various pH (2–12), temperatures of leachate solution and using TCLP. At pH 2, the highest leaching was observed for Fe (21.6 and 32.8 µg/g), whereas at pH 12, Arsenic was found to have the highest leaching (1.5 and 2.4 µg/g) in FA1 and FA2. Leachate solution temperature showed a positive effect on the metal's leachability. In TCLP, most of the metal's leachability was observed to be higher than that of batch leaching tests. The present study suggests that, leaching of As and Se from FA samples can moderately affect ground/surface water quality at the study locations.

**Keywords** Fly ash · Leaching · TCLP · Trace and toxic metals

With growing energy demand, many industrialized and developing countries will continue to rely on coal for power generation in the decades to come. In India, coal is one of the major fossil fuels and about 70 % of electricity is produced from coal-based power plants (Singh et al. 2012). In the case of coal based power plants, coal fly ash (FA) is a byproduct that was produced approximately 170 million tonnes per annum in 2012 and is expected to increase in the range of 300–400 million tonnes per annum by 2017. Over the years, generated FA was used in landfillings (Hongling et al. 2009), but the physical and mechanical properties of FA makes it an attractive material for many different applications including the usage in building material, asphalt, concrete pavements etc. However, only a small fraction of FA is currently used in cement and concrete industry and the remaining is disposed in landfills or in ash ponds. Current disposal practices (land filling, surface impoundment, placement in mines and quarries) can potentially affect surface/ground water and air quality through fugitive dust or runoff (Hongling et al. 2007).

During coal combustion, FA gets concentrated with various trace and toxic metals such as Arsenic (As), Selenium (Se), Chromium (Cr), Lead (Pb) etc. The accumulation of metals concentration in FA majorly depends on the type of coal and combustion processes used in the power plants. Toxic elements associated with FA can leach out to water bodies leading to disruption of aquatic life as well as contamination of underground water resources (Hongling et al. 2012).

It is important to understand the pH-dependent leaching behaviour of trace and toxic metals associated with FA. These studies will give an idea about the extent of leaching of metals at a given pH, which will help in assessing the impact of leaching on ground water quality as well as pollution control strategies. The pH-dependent leachability information is also needed for geochemical modelling to identify leaching controlling mechanisms (Van der Sloot et al. 2007), which is recommended for future studies. Further, studying the effect of leachate temperature on leachability of metals from FA is also crucial, as the atmospheric temperature has a wide variation depending upon the season. In recent years, studies on the leaching behavior of elements associated with FA have received

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more attention, particularly in India. Contamination of surface and ground water with heavy metals have been reported in many studies (Kapoor and Christian 2016; Tiwari et al. 2015; Singh et al. 2014) carried out at locations near the FA disposal facilities. Therefore, it is important to study further, the leaching behavior and the extent of leaching of metals in FA under varying conditions to estimate the probable surface/ground water contamination.

In view of this, a study was carried out with an aim to investigate the leaching behavior of various metals associated with FA samples collected from two different thermal power plants in India. People residing near the study sites mostly depend on the ground water for drinking purposes; hence, the present work was also aimed to evaluate the possible ground/surface water contamination due to leaching of metals from FA. The initial concentration of elements in FA, pH/temperature of leachate, leaching time, solid–liquid ratio and various other parameters can affect the leachability of elements into aqueous environment. Hence, short-term (i.e., 24 h) leaching tests were carried out for obtaining critical information in considerably shorter interaction periods, compared with those carried out for weeks (Singh et al. 2014). These tests are the simplest means to compare the leaching behaviour of various metals in FA at different leachate conditions (viz. pH, temperature, etc.) as mentioned above.

## Materials and Methods

FA samples used for the leaching experiments were collected from two different thermal power plants in India; one from Andhra Pradesh (FA1) and the other from Maharashtra (FA2). The coal used in both the power plants is of low grade (fixed carbon: 34.1 %–47.7 %) with 25 %–32 % ash content. Thermal power plant in Andhra Pradesh and Maharashtra nearly generates 1.24 mtpa and 0.38 mtpa of FA respectively. Approximately 0.41 mtpa of FA1 and 0.16 mtpa of FA2 was being utilized in roads, embankments, ash dykes and the remaining FA was disposed in landfills. In the present study, multiple number of samples ($n = 15$ at each location) were collected from the dust hoppers of electrostatic precipitator. Sampling was carried out on 5, 7, 11, 13 and 15th of May 2015, covering different time periods (morning, afternoon and evening) of the sampling day. Collected samples at an individual site were mixed to get one representative sample for each location. Samples were stored in a clean polyethylene bags until chemical analyses and leaching studies were carried out.

Estimation of total metal concentration in FA is a crucial step prior to leaching experiments. To achieve this purpose, microwave assisted acid digestion method was used. About 1 g of FA1 and FA2 was weighed in replicate into Teflon beakers containing 10 ml $HNO_3$ and 2 ml HF. After microwave digestion, the resulting solutions were transferred into Teflon beakers and heated on a hot plate to remove excess acid. The remaining solutions were filtered into the 25 ml volumetric flask by giving multiple washing to the sample beakers using 0.25 % $HNO_3$. For As and Se analysis, FA samples were acid digested at a relatively lower temperature to avoid volatilization of these metals. Resulting sample solutions were analyzed for various metals of interest using (ICP-AES, Jobin–Yvon Horib, and model ULTIMA 2), Hydride Generation Atomic Absorption Spectroscopy (HG-AAS) and Differential Pulse Anodic Stripping Voltametric technique (DPASV).

In the present work, all the leaching studies were carried out using Grant shaking water bath (model OLS200), which has both the orbital and linear motion facility. Operating temperature, orbital and linear shaking speed of shaking water bath are in the range of 25–99°C, 20–200 rpm and 40–360 rpm respectively. In the current study, batch leaching experiments were carried out for 24 h at 150 rpm (Deepak et al. 2012; Singh et al. 2012; Jankowski et al. 2006) shaking speed and at different leachate solution temperatures like 30, 40, 50°C. Concentrations of trace metals in FA leachate solutions were determined by various analytical techniques as mentioned above. During metal analysis, instruments were calibrated for each metal using a set of three to five high-quality standards supplied by VHG Labs manufactures, USA. The calibration curve was checked frequently by injecting a known standard and the data was subsequently corrected for any observed minute deviation.

To verify the leachability of metals as a function of L/S (Liquid to Solid i.e., Water to Fly ash) ratio, four parallel batch leaching tests with different L/S ratios (20, 30, 50 and 100) were carried out at room temperature using deionized water as an extractant. In the present study, the minimum L/S ratio of 20 was chosen, as at L/S ratios lower than 20, the mixing of FA and water was observed to be poor. After leaching for 24 h, the leachate solutions were centrifuged at 6000 rpm for 15 min and separated from the solid residue by filtering through a 0.45 µm filter paper. The leachate solutions were poured into acid cleaned beaker and digested for 8 h on a hot plate by adding 1–2 ml of concentrated $HNO_3$. Excess solution was reduced to near dryness and left out solution was filtered into the 25 ml volumetric flask by giving multiple washing to the sample beaker to assure complete transfer of leached out elements (Bably and Kajal 2008; Singh et al. 2012). Processed leachate solutions were analyzed for various trace and toxic metals of interest.

The pH dependent leaching tests were performed on both the FA samples at different leachate solution pH values ranging from 2 to 12. Deionized water was added to the flask containing FA in combination with $HNO_3$ or NaOH to obtain the desired endpoint pH of the FA-water system. All the FA samples were leached at optimized condition (L/S = 20 and 24 h leaching period) and resulting leachate solutions were analyzed for metals of interest.

In the present work, the leaching study was carried out at different leachate temperatures viz. 30, 40 and 50°C to find the influence of temperature on metals leachability. A 12.5 g of each FA was weighed into a volumetric flask and a 250 ml leachate (double distilled water) was added to it. The flasks were then kept in a shaking water bath, which was maintained at desired temperatures. The resulting leachate solutions were chemically processed and analyzed for metals of interest.

Toxicity characteristic leaching procedure (TCLP) was applied to evaluate the potential leachability of toxic metals in FA samples. TCLP requires the use of an acetic acid buffer having pH 4.99 as per United States Environmental Protection Agency (USEPA) procedure (USEPA 1990c). Both the FA samples were leached using an acetic acid buffer (pH 4.99) for 18 h at 30 ± 2 rpm shaking speed maintaining buffer to FA ratio of 20. Duplicate extractions were performed under the same experimental conditions and resulting leachate solutions were analyzed for metals of interest.

## Results and Discussion

Natural pH of FA is an important property which can affect the metals leachability in aqueous environment. The pH of FA1 and FA2 was found to be 8 and 9.5 respectively in a slurry solution prepared by mixing deionized water and FA samples. The average total concentrations of various metals associated with FA samples are presented in Table 1. A comparable elemental concentrations of FAs were reported in the literature (Bably and Kajal 2008; Sarode et al. 2010; Tiwari et al. 2014; Belviso et al. 2015). Among different elements analyzed, Fe was observed to have the highest concentration in both the FA samples followed by Mn and Cr in FA1 and FA2 respectively. Of the two samples, FA from Maharashtra has marginally higher concentrations of most of the elements, except Fe and Mn. Observed difference could be due to differences in type of coal burned, coal pre-processing methods and the pollution control devices employed at the respective facilities.

Chemical analyses of leachate solutions from L/S ratio test indicated that, at L/S ratio of 20, the highest leaching of all the metals was observed. Leaching of metals could be slightly higher at L/S ratio less than 20, but we have started with L/S ratio of 20 for the reasons mentioned in Materials and methods section. On further increase in L/S ratio, dilution of leachable metal's concentration was observed. Based on experimental results (Table 2) and ease of handling, L/S ratio of 20 was fixed for further leaching tests.

In the present study, pH of leachate solution was observed to have great influence on the leaching behavior of metals associated with FA. Figure 1 demonstrates the leachability of metals (Y-axes with different scales) into the leachate solution at different leachate pH values. Throughout the results discussion, only, the leachability of metals (present in FA) into the leachate solution was discussed in terms of µg/g.

In the present study, most metals (except As and Se) were observed to have their highest leachability at pH 2 as compared to their leachability at other leachate pH values. Similar leaching pattern has been reported by many authors (Izquierdo and Querol 2012; Kim et al. 2003). As shown in Fig. 1, Fe was observed to have the highest relative leaching (21.6 and 32.8 µg/g) at pH 2 followed by Mn (3.2 and 4.9 µg/g) and V (2.4 and 3.9 µg/g), whereas, the least leaching was observed for Pb in both the FA samples. Pb was found to be highly insoluble and virtually immobile irrespective of the pH due to its dominant silicate association (Kim and Kazonich 2004). High leachability of most of the metals at pH 2 could be due to the highly acidic nature of leachate solution which can easily dissolve metal oxides and metals bound to organic compounds. On the contrary, As and Se were observed to have the lowest solubility at pH 2 (shown in Fig. 1), which could be due to the strong affinity of Al and Fe-oxyhydroxides to sorb these metals at low pH region.

Although Fe found to have relatively higher leaching, the percentage leachability (with respect to its total metal concentration in FA) was found to be the least as shown in Table 3. This could be due to the occurrence of Fe in FA1 and FA2 mainly in hematite form, although a minor proportion can be assimilated in the glassy matrix. Fe in this form is highly stable, therefore resistant to weathering and is not easily released (Kukier et al. 2003; Izquierdo and Querol 2012). At pH 2, Se was observed to have the highest percentage leachability followed by V in both the FA samples. Surface association of V in FA (Finkelman

**Table 1** Total concentrations (µg/g) of various metals present in collected FA samples

| S. No | V | Cr | Mn | Fe | Ni | Cu | Zn | As | Se | Pb | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 112.8 | 99.2 | 170.0 | 17,922.5 | 42.6 | 42.9 | 58.7 | 9.0 | 2.9 | 25.2 | (FA1) |
| 2 | 115.7 | 127.9 | 124.3 | 13,872.7 | 54.8 | 45.0 | 100.7 | 11.4 | 3.8 | 31.6 | (FA2) |

**Table 2** Variation of the leachability of different metals (µg/ml) at different L/S ratios

| | V | Cr | Mn | Fe | Ni | Cu | Zn | Pb | Se |
|---|---|---|---|---|---|---|---|---|---|
| L/S = 20 | 0.319 | 0.028 | 0.066 | 0.286 | 0.014 | 0.026 | 0.062 | 0.017 | 0.119 |
| L/S = 30 | 0.222 | 0.023 | 0.056 | 0.270 | 0.011 | 0.021 | 0.056 | 0.015 | 0.071 |
| L/S = 50 | 0.190 | 0.018 | 0.043 | 0.262 | 0.009 | 0.017 | 0.050 | 0.012 | 0.043 |
| L/S = 100 | 0.123 | 0.015 | 0.029 | 0.240 | 0.005 | 0.012 | 0.042 | 0.011 | 0.025 |

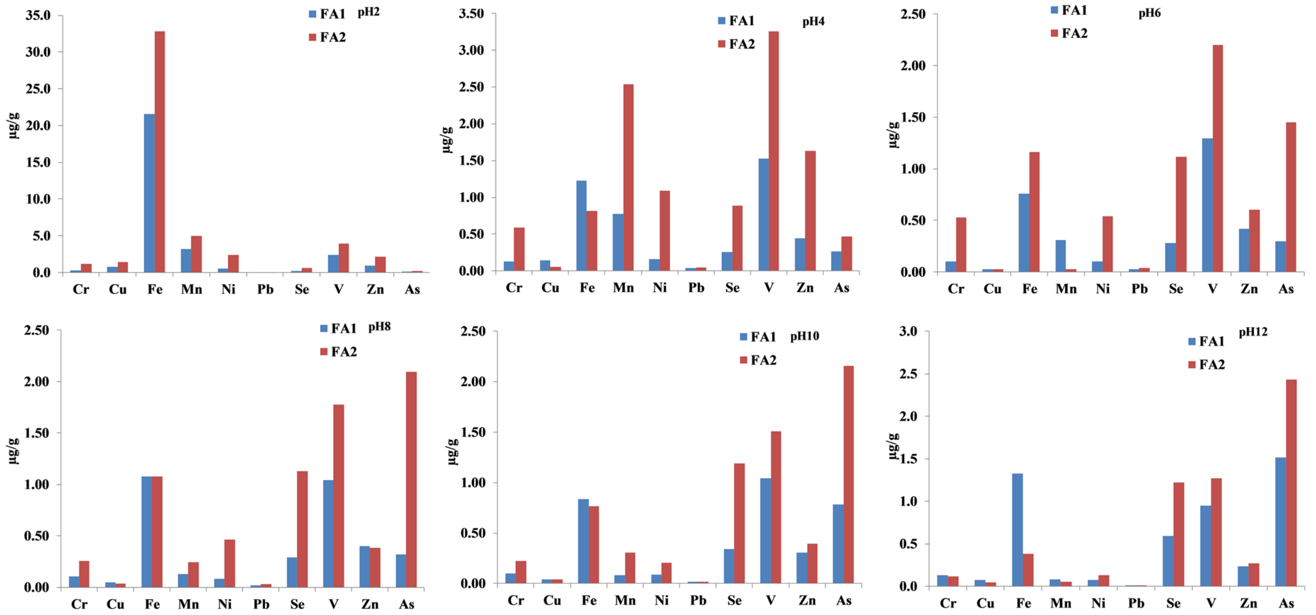

**Fig. 1** Influence of leachate pH on leaching behavior of metals from FA1 and FA2 into the leachate solution (with different y-axes scales)

**Table 3** Percentage leachability of metals at different leachate pH

| | pH 2 | | pH 4 | | pH 6 | | pH 8 | | pH 10 | | pH 12 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | FA1 | FA2 | FA1 | FA2 | FA1 | FA2 | FA1 | FA2 | FA1 | FA2 | FA1 | FA2 |
| Cr | 0.33 | 0.90 | 0.13 | 0.46 | 0.10 | 0.41 | 0.11 | 0.20 | 0.10 | 0.18 | 0.13 | 0.09 |
| Cu | 1.80 | 3.15 | 0.34 | 0.13 | 0.07 | 0.06 | 0.11 | 0.09 | 0.09 | 0.10 | 0.18 | 0.10 |
| Fe | 0.12 | 0.24 | 0.01 | 0.01 | <0.01 | 0.01 | 0.01 | 0.01 | <0.01 | 0.01 | 0.01 | <0.01 |
| Mn | 1.87 | 4.01 | 0.46 | 2.04 | 0.18 | 0.02 | 0.08 | 0.20 | 0.05 | 0.25 | 0.05 | 0.04 |
| Ni | 1.18 | 4.39 | 0.37 | 1.99 | 0.24 | 0.99 | 0.20 | 0.85 | 0.20 | 0.38 | 0.17 | 0.24 |
| Pb | 0.21 | 0.21 | 0.15 | 0.16 | 0.11 | 0.13 | 0.08 | 0.10 | 0.06 | 0.05 | 0.04 | 0.03 |
| Se | 7.57 | 15.87 | 8.95 | 23.39 | 9.70 | 29.38 | 10.13 | 29.76 | 11.82 | 31.36 | 20.34 | 32.10 |
| V | 2.13 | 3.38 | 1.35 | 2.81 | 1.15 | 1.90 | 0.93 | 1.54 | 0.92 | 1.30 | 0.84 | 1.10 |
| Zn | 1.60 | 2.11 | 0.76 | 1.62 | 0.71 | 0.60 | 0.68 | 0.38 | 0.52 | 0.39 | 0.40 | 0.27 |
| As | 1.30 | 1.86 | 2.94 | 4.11 | 3.32 | 12.74 | 3.58 | 18.38 | 8.69 | 18.93 | 16.85 | 21.34 |

1995) could be the reason for observed higher percentage leachability as compared to other metals (viz. Cr, Cu, Ni and Zn). These metals are believed to be mostly associated with glassy matrix/alumina-silicate phase of FA during combustion (Spears 2004; Goodarzi et al. 2008), leading to their lower leachability even in the highly acidic medium. The Percentage leachability observed for Cu and Zn in the present study was comparable with that reported in the literature (2 %–9 % at pH 1–3; Kukier et al. 2003; Kim et al. 2003).

At pH 4, leachability of most of the metals was decreased as compared to that observed at pH 2. Among different metals, V was observed to have the highest relative leachability followed by Fe and Mn in both the samples. A drastic decrease in the leachability of Fe and Cu could be due to the decrease in acidic strength of leachate solution. In contrary, higher leaching of As and Se were observed at pH 4 than that observed at pH 2.

Leachability of most of the metals (except As and Se) was decreased furthermore at pH 6, 8 and approached the least values in the pH range of 10–12. However, As and Se were observed to have their highest leachability in the pH range of 10–12. This could be due to the existence of these metals predominantly as $CaAsO_4^-$, $AsO_4^{3-}$, $SeO_3^{2-}$ at pH >7.5 (Cornelis et al. 2008). At this pH and higher, FA particles are negatively charged; therefore, sorption of negatively charged As and Se species onto negatively charged FA particle surfaces are unfavourable, leading to higher leachability. The increased leaching behaviour of As and Se at alkaline pH was also observed in other studies (Komonweeraket et al. 2015; Van der Sloot et al. 2007). The observed results in the present study are comparable with those reported for water extractable As (0.3 %–15 %) and Se (10 %–50 %) from FA samples worldwide (Iwashita et al. 2005; Ward et al. 2003).

Table 4 demonstrates the results of leachability of various metals of interest with respect to temperature. Increase in leachate temperature was observed to have a positive effect on the leaching of most of the metals, except Fe, as shown in Table 4. Similar studies were conducted by Otero-Rey et al. (2005) and Baba et al. (2008); their study results indicated that, with the increase in temperature, the leachability of metals also increases. The increase in temperature increases the agitation in the FA-leachate system, leading to increased release of surface adsorbed metals and partial releases from internal glassy matrix of FA.

In this study, V was observed to have the highest leachability, whereas, the least solubility was observed for Pb in both the samples. The non increasing leachability of Fe can be attributed to the reasons mentioned by Kukier et al. (2003).

In terms of percentage leachability, Se has the highest leaching (18.5 % from FA1 and 19.2 % from FA2 at 50°C) followed by As (12.7 % from FA1 and 16.6 % from FA2 at 50°C). These values were found to be lesser than that observed at pH 12 for Se and As. Here, it is a comparison between the percentage leaching of metals at the natural pH of FA and at pH 12 of leachate.

Leachable trace and toxic metal concentrations of both the FA samples as a result of TCLP are presented in Fig. 2a. Many authors (Yilmaz 2015; Sarode et al. 2010) have used TCLP procedure to estimate the highest leaching concentration of metals from FA. Their study indicates that, the metals leachability is higher in TCLP as compared to batch leach tests. In the present study, the metals concentration in TCLP was also found to be higher as compared to the leaching tests conducted for L/S ratio (Table 1). This could be due to the precipitation of metal ions as insoluble hydroxides, resulting in low metal's leaching in batch leaching tests.

In TCLP also, Fe was observed to have the highest leaching concentration (8.8 and 7.6 µg/g) followed by Mn (2.9 and 4.4 µg/g) in FA1 and FA2 respectively. Among different metals, the least leaching was observed for Pb followed by Cr in both the FA samples. Higher leachability of metals observed in TCLP could be due to the formation of soluble metal complexes by acetate buffer solution.

Figure 2b indicates that, Fe has the lowest (0.05 % from both the samples) percentage leaching followed by Pb (<0.2 %). Whereas, the highest percentage leaching was observed for Se (17.1 % and 21.5 %) followed by As (11.7 % and 19.9 %) from FA1 and FA2 respectively. These values were observed to be in the comparable range reported by Ward et al. (2003). TCLP study gives an idea about the worst possible leachability of metals into leachate solution. Present TCLP study results can be useful in a situation where the FA is used to neutralize acid mine drainage.

This study gives an idea about the leaching behavior of metals in two different FA samples using batch leaching tests (at various pH, temperature of leachate) and TCLP. Results indicated that, the release of metals from FA samples is strongly dependent on the pH of leachate solution. Leaching pattern of metals such as V, Cr, Cu, Fe, Mn, Ni, Pb and Zn was observed to follow a cationic leaching behaviour, where the leachable concentration increases monotonically as pH decreases. In contrary, the oxyanionic leaching pattern was observed for As and Se; among all the metals, the highest relative leaching was observed for Fe and the least leaching was observed for Pb.

**Table 4** Influence of leachate solution temperature on leachability of metals (µg/g) in FA samples

| | Temp. (°C) | Cr | Cu | Pb | Se | V | Zn | As | Mn | Ni | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|
| FA1 | 30 | 0.126 | 0.037 | 0.024 | 0.472 | 1.132 | 0.244 | 0.861 | 0.122 | 0.080 | 1.033 |
| | 40 | 0.142 | 0.061 | 0.032 | 0.500 | 1.307 | 0.293 | 1.020 | 0.160 | 0.100 | 1.144 |
| | 50 | 0.170 | 0.060 | 0.034 | 0.536 | 1.691 | 0.320 | 1.140 | 0.230 | 0.110 | 0.962 |
| FA2 | 30 | 0.212 | 0.039 | 0.031 | 0.663 | 1.376 | 0.422 | 1.297 | 0.084 | 0.130 | 0.784 |
| | 40 | 0.250 | 0.043 | 0.041 | 0.740 | 1.633 | 0.485 | 1.694 | 0.114 | 0.150 | 0.846 |
| | 50 | 0.351 | 0.063 | 0.044 | 0.866 | 1.960 | 0.550 | 1.894 | 0.150 | 0.170 | 0.944 |

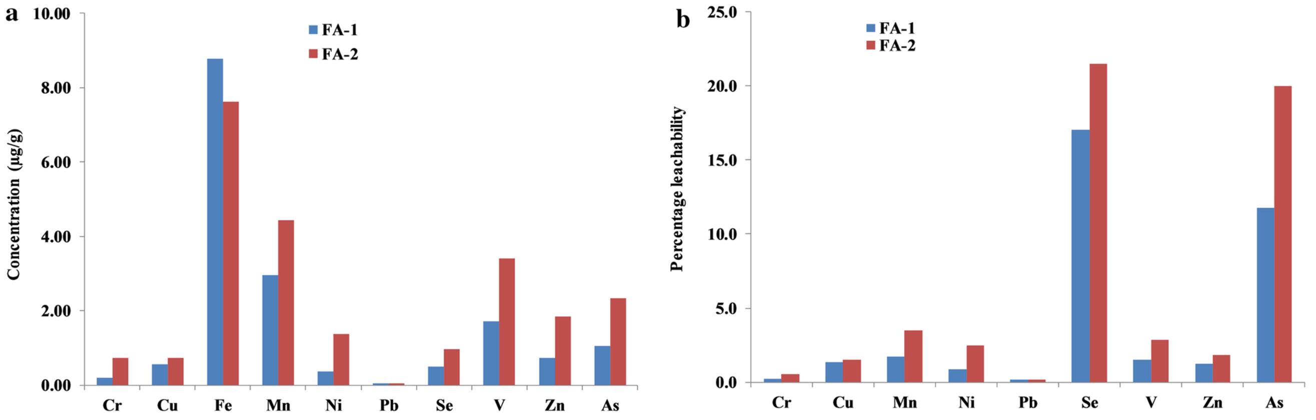

**Fig. 2** Leachability of metals into leachate solution in terms of (**a**) concentration (µg/g) (**b**) percentage in TCLP

In terms of percentage leaching, Se was observed to have the highest leaching followed by As and the least percentage leaching was observed for Fe. Higher leachability of the metals was observed in TCLP as compared to batch leaching tests. Leachate temperature was also observed to affect the leaching behaviour of most of the metals. From the study results, it can be concluded that, leaching of As and Se may moderately affect ground water quality, whereas other metals (Cr, Pb, Cu, Zn, etc.) may not cause any ground water contamination due to their very low leachability.

Most of the fly ashes (FAs) produced in India are alkaline in nature; the current leaching study was carried out using two such alkaline FA samples. Some power plants also generate acidic FAs, for which, leaching patterns of metals may entirely be differ from alkaline FAs. Hence, current study presents limited information about the overall leaching patterns of metals in FAs generated in India.

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