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ORIGINAL ARTICLE

Leachability of trace elements from two stabilized low lime Indian fly ashes

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Abstract Fly ash is a waste by-product obtained from the burning of coal by thermal power plants for generating electricity. When bulk quantities are involved, in order to arrest the fugitive dust, it is stored wet rather than dry. Fly ash contains trace concentrations of heavy metals and other substances in sufficient quantities to be able to leach out over a period of time. In this study an attempt was made to study the leachabilities of a few selected trace metals: Cd, Cu, Cr, Mn, Pb and Zn from two different types of class F fly ashes. Emphasis is also laid on developing an alternative in order to arrest the relative leachabilities of heavy metals after amending them with suitable additives. A standard laboratory leaching test for combustion residues has been employed to study the leachabilities of these trace elements as a function of liquid to solid ratio and pH. The leachability tests were conducted on powdered fly ash samples before and after amending them suitably with the matrices lime and gypsum; they were compacted to their respective proctor densities and cured for periods of 28 and 180 days. A marked reduction in the relative leachabilities of the trace elements was observed to be present at the end of 28 days. These relative leachability values further reduced marginally when tests were performed at the end of 180 days.

Keywords Leachability · Trace elements · *L/S* ratio · pH · Curing period · Pozzolanic reactivity

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Introduction

Due to India's economic rise, the demand for energy has grown at an average of 3.6% per annum over the past 30 years. More than 50% of India's commercial energy demand is met through the country's vast coal reserves. As about 76% of the electricity consumed in India is generated by thermal power plants, huge quantities of ash get generated from the combustion of coal. This is about 40–50% of the coal burned, whereas in the USA, UK and Canada, it is hardly 10%. In India the annual production of fly ash is nearly 45 million tons and is likely to increase to 70 million tons by 2010. Many attempts have been made to utilize this fly ash in several civil engineering applications, such as use as a fill material (Digioa and Nuzzo 1972; Faber and Digioa 1974; Horiuchi et al. 2000), as a lightweight aggregate (Kishan 1988; Bin-Shafique et al. 2004), for manufacture of clay flooring and terracing tiles (Bhatnagar et al. 1988; Bou et al. 2009), as an absorbent material (Ayala et al. 1998; Héquet et al. 2001; Alinnor 2007), in concrete technology as a binding material (McCarthy and Dhir 1999), and as a volume replacement and a self-compacting material for increasing the workability of concrete (Ganesh and Siva 1993; Siddique 2003; Jaturapitakkul et al. 2004; Tsimas and Tsima 2005; Yen et al. 2007; Sahmaran et al. 2009). With only a very small utilization of about 12% of fly ash produced in India compared to global utilization of 25%, the ash deposits at the thermal power plants are increasing rapidly, and the problem of disposal is expected to become alarming because of the limited space available for its disposal near most of the thermal power stations. To reduce the problem of disposal of huge quantities of fly ash, efforts are being made to find applications where it can be utilized in bulk quantities. One major drawback limiting its wide use is the problem of leaching of the trace elements present in the ash into the environment.

Many researchers have conducted extensive studies on the leachabilities of heavy metals from different fly ashes (Eisenberg et al. 1986; Garcez et al. 1987; Warren and Dudas 1989; Fytianos et al. 1998; Ugurlu 2004; Baba and Kaya 2004; Jegadeesan et al. 2008; Dutta et al. 2009). For sub-bituminous coal ashes from India, it was observed that the concentration of trace elements leached was more at lower *L*/*S* ratios than at higher ones (Praharaj et al. 2002). The leachability of trace elements present in the ash increases with decreasing pH. Based on the release of 19 cations from 32 fly ash samples in the pH range of 1.2–12, Kim et al. (2003) have, suggested that the principal mechanisms of metal leachability are the dissolution of primary solids under aggressive environments and/or precipitation sorption reactions, which are primarily dependent on pH. On the other hand, the relatively low leachability of cationic metals from fly ashes is due to their alkaline nature (Kim 2006). Kim and Hesbach (2009) adopted five leaching methods: the serial batch leaching procedure (SBLP), synthetic groundwater leaching procedure (SGLP), mine water leaching procedure (MWLP), 3TIER integrated framework leaching protocol (3TIER) and the toxicity characteristic leaching procedure (TCLP) to study the maximum availability and long-term release of 23 cations from Class C and Class F fly ashes. The release of cations was found to depend very much on the type of extracting fluid used and the time required, which varied from 1 TCLP to 60 SGLP days. Praharaj et al. (2002) conducted serial batch leaching tests simulating the rain water condition to understand the behavior of elements during the process of leaching. Table 1 summarizes leachabilities of various elements from different coal ashes observed by earlier investigators.

Table 1 Summary of leachabilities of various elements from different coal ashes observed by earlier investigators

Fly ash	Trace elements studied	Method/conditions	Remarks	References
Alberta Power's Battle river power plant ash	Al, As, B, Ca, Fe, K, Mg, Mn, Na, Se, Si	Sequential extract of ferromagnetic fraction using 0.5 mmol/l HNO_3	Release of metals is governed by presence of elements in intersticial alumino silicate phase over minor dissolution of crystalline magnetite	Warren and Dudas (1989)
4 samples of Greek fly ash (Kardia, Agios Dimitrios, Ptolemais, Amideo)	Pb, Cd, Cr, Cu, Zn, Ca, Mn	Vander Sloot Method; *L*/*S* 5 to 100; pH 4, 6 and 8 adjusted using HCl and NaOH	Ca showed highest leachability and Mn least. Maximum leachability was obtained at lower pH values (pH 4)	Fytianos et al. (1998)
FA 1, 2, 3 and 4; Orissa, India	Al, Ca, K, Mg, Na, P, S, Si, As, Ba, Fe, Mn, Mo, Ti, V, Pb, Zn, Co, Cr, Cu, Ni, Cd	Serial batch leaching test; 125 g fly ash; 500 ml water; *L*/*S* ratio 4, 8, 12 and 16	Maximum extractability observed at *L*/*S* ratio of 4 and 8. Cd, Cr, Co and Ni did not leach	Praharaj et al. (2002)
Kemerkoy Power Plant fly ash and pond ash, Turkey	Ca, Mg, Na, K, SO_4, Pb, Mn, Co, Cu and Fe	Batch leaching test; 5 g fly ash at *L*/*S* ratio of 5	EC values decreased after each extraction, Ca, Na, K, Mn, Fe, S and Pb showed maximum leachability, whereas, Cd, Mg, Cu, Cr, Zn and Co showed minimum leachability	Ugurlu (2004)
Soma and Tunçbilek fly ash, Turkey	Ca, Cd, Cr, Co, Cu, Fe, K, Mn, Mg, Na, Ni, Sr, Pb and Zn	CEN and TCLP procedure was followed to evaluate leachability	Concentrations obtained by TCLP were high compared to CEN, since the extraction is pH dependent. Cd and Pb concentrations at Soma power plant were high	Baba and Kaya (2004)
CFFA	As, Cr, Pb, Fe, Cu, Zn	SCE, EDTA, TCLP, SPLP extraction procedure. Constant pH extraction tests	Release of Cu, Cr, Pb, Zn and Fe is less under most pH conditions	Jegadeesan et al. (2008)
CFA-1,2,3 and 4; West Bengal, India	Fe, Mn, Ca, Na, K, Cu, Cr, Zn, As, Pb	40 g of fly ash; 200 ml acidified water (using dilute H_2SO_4); *L*/*S* ratio 1:5; 7 days	Release of toxic heavy metals would occur particularly under acidic leaching conditions	Dutta et al. (2009)

In this work the laboratory leaching test for combustion residues developed by Van der Sloot et al. (1984) was adopted to study the leaching phenomenon from two low lime fly ashes. Based on the critical review of the literature the trace elements selected are Cd, Cr, Cu, Mn, Pb and Zn. It is common to incorporate lime and gypsum to enhance the strength and durability of fly ash for many bulk applications. Lime was added to increase the pozzolanic activity of low lime fly ash (Sivapullaiah et al. 1995), and gypsum was added to arrest the leaching of lime from it (Ghosh and Subbarao 2006). Thus, the leachability of the fly ashes without any additive and after amending the same with lime and gypsum was studied. The improvement in strength with the curing period because of the pozzolanic reaction was found to be the main reason for many applications of fly ash. In the present case in order to study the effect of pozzolanic reactivity on the leachability characteristics, studies have been carried out on fly ash samples mixed with these additives.

Materials and methods

Two low lime fly ashes collected directly from the electrostatic precipitators of thermal power plants located at Neyvelli town (11°36′28.39″N and 79°29′28.81″E) of Tamil Nadu and Muddanur town (14°41′01.43″N and 78°23′32.71″E) of Andhra Pradesh, named NFA and MFA, respectively, were used in this study. The physical properties and the chemical composition of these fly ashes along with compaction characteristics are presented in Tables 2 and 3. These fly ashes have been classified as F type as per the ASTM classification [ASTM C618-03 (2003)].

Table 4 shows the total concentrations of selected trace elements present in both the fly ash samples. Acid digestion technique using US EPA Method 3050B (USEPA 1996) was employed below the boiling point to determine the heavy metal concentrations available for leaching. For the leaching tests the Van der Sloot et al. (1984) method was used with different liquid to solid weight ratios varying from 5 to 100 on fly ash of 5 g. It was generally found that the period required to complete leachability was around 18 h. With a buffer period of another 8 h, the extraction time was taken as 24 h. The extract was filtered through Whatman no. 42 filter paper to separate fly ash from the fluid. To save the trouble of rinsing, the ash was transferred along with the filter paper to the same bottle. For the next extraction, another aliquot of the same volume of fluid was added, which gives a cumulative liquid solid ratio twice that of the initial ratio. This process was repeated till the liquid solid ratio of 100 was achieved. Another series of leaching tests was conducted where the pH of the fluid was varied from 2 to 10 at a fixed *L/S* ratio of 20. The pH of the solution was kept constant by suitably adding HCl and NaOH solutions, and was monitored constantly by using Jenway pH electrode. The fly ash was oven dried at 105°C for 2 h prior to the conduction of the test. The 500-ml capacity polyethylene bottles used to carry out leachability tests were kept soaked in 0.1 N HCl for a week and were washed thoroughly with distilled water before the test each time in order to avoid any interference effect. The elemental analysis was carried out using inductively coupled plasma atomic emission spectrometry (ICP-AES).

To study the effect of pozzolanic reactivity on the leaching behavior of the fly ashes, the samples were dry

Table 2 Physical properties of fly ashes

Property	NFA	MFA
Specific gravity	2.83	2.17
Grain size distribution		
Silt fraction (%)	87	94.5
Clay fraction (%)	13	5.5
Atterberg limits		
Liquid limit (%)	39	29
Plastic limit (%)	NP	NP
Plasticity index (%)	NP	NP
Compaction characteristics		
Optimum moisture content (%)	24	23.8
Maximum dry density (kN/m^3)	14.2	13.3
Surface area (m^2/g)	9.6	8.2

NP non plastic

Table 3 Chemical composition of fly ashes

Constituent	NFA (%)	MFA (%)
Silica (SiO_2)	50.97	56.88
Alumina (Al_2O_3)	18.81	27.65
Ferric (Fe_2O_3)	16.61	6.28
Calcium (CaO)	9.00	3.62
Magnesium (MgO)	1.41	0.34
Titanium (TiO_2)	0.28	0.31
Potassium (K_2O)	0.23	0.27
Sodium (Na_2O)	0.18	0.19
Loss on ignition	2.60	4.46

Table 4 Concentration of trace elements in fly ash (mg/kg)

Trace element	NFA	MFA
Pb	121.5	96.2
Zn	34.8	40.4
Mn	67.6	49.8
Cd	36.2	25.8
Cu	32.1	46.5
Cr	–	79.8

mixed with respective additives initially on weight basis. The samples were then compacted at their respective proctor densities at optimum moisture content values. The samples were then cured for 28 and 180 days separately. On completion of each curing period the samples were powdered and air dried before oven drying at 50°C for 2 h so as to avoid the loss of hydrated water of gypsum that occurs at 65°C. The leachability test was then performed on the sample, as earlier. XRD was carried out using the Bruker D8 Advance system on samples before they were subjected to the leachability test in order to identify the new compounds formed due to pozzolanic activity. Samples were scanned from 5° to 80° (2θ) using a 2.2-kW Cu anode long fine-focus ceramic X-ray tube at a scanning rate of 1°/min. XRD patterns of samples were compared with a standard pattern of possible types of compounds to identify the new compounds formed (JCPDS, Powder Diffraction File 1990).

Results and discussions

Effect of *L/S* ratio

Several studies have reported that the *L/S* ratio is a major factor dictating the leachability of trace metal ions from fly ash and other combustion residues. Figure 1 gives the leaching of Ca, Pb, Cd, Zn, Mn and Cu from NFA and Ca, Pb, Cd, Zn, Mn, Cu and Cr from MFA as determined for different *L/S* ratios of 5–100. It is a general observation that the leaching of any trace element increases with an increase in the L/S ratio. The increase is steep up to a *L/S* ratio of 20 and then is gradual beyond 20 up to 100. These results agree closely with those of Fytianos et al. (1998) who concluded that generally the leaching of most trace metals from fly ashes and combustion residues increases with the *L/S* ratios. The leaching order of trace elements for a given fly ash is not affected by an increase in the *L/S* ratio. The order of leachability for NFA (Fig. 1a) at any *L/S* ratio varies in the order of Ca > Pb > Cd > Zn > Mn > Cu. For MFA (Fig. 1b) the order is Ca > Cd > Pb > Mn > Cr > Zn > Cu. The variation in the order of leachabilities of trace elements for the fly ashes may be due to variations in pH at the given *L/S* ratio and the original concentrations of the elements present in the fly ashes. The leachability of any metal ions is found to increase with its initial concentration present in the fly ash. As the *L/S* ratio increases, generally the pH of the system increases, which will cause a reduction in the leachability of metal ions from the surface of fly ash particles. For both the fly ashes the leachability of calcium was found to be far more than those of the other elements at any given liquid to solid ratio. The leachability values of NFA are relatively more compared to MFA. This is due to differences in their particle size distribution characteristics. It is observed that MFA has a greater finer particle content than NFA. Similar observations have been observed by Sukandar et al. (2006) for metals leaching from medical waste incinerator fly ash.

Effect of pH

In studying the effect of the *L/S* ratio on the leachability of trace elements, no attempt was made to maintain a constant pH value. However, it is known that the leachability of most of the trace elements is affected by the pH of the leaching fluid. Leachability tests at constant pH values between 2 and 10 were performed at a fixed *L/S* ratio of 20.

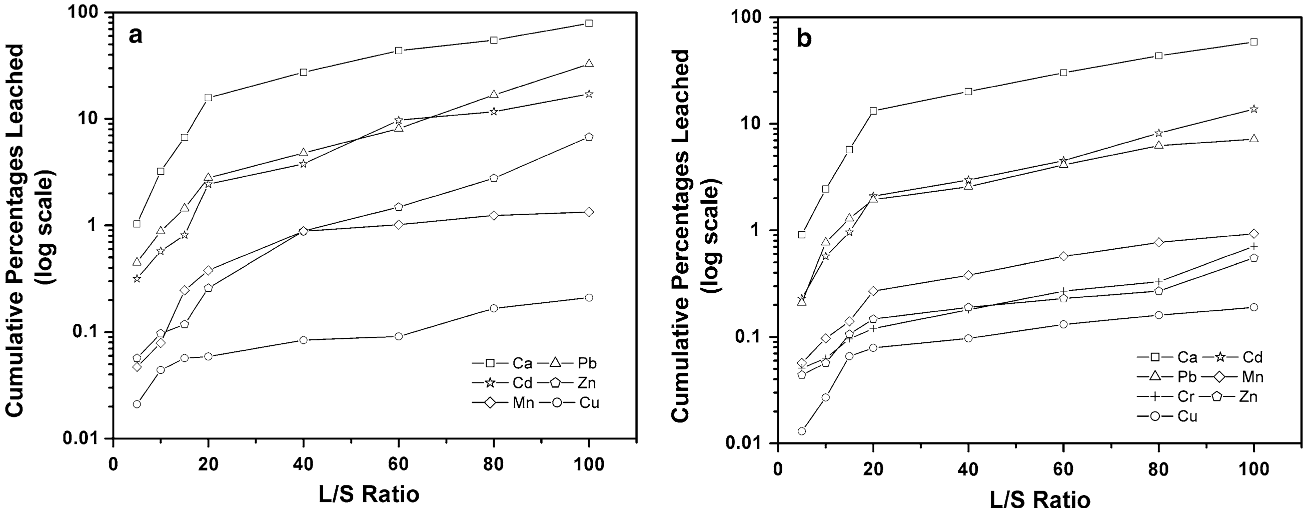

Fig. 1 Cumulative percentage of various elements leached from **a** NFA and **b** MFA with *L/S* ratio

The pH of the slurry was maintained at a constant value by adding small amounts of 0.1 M HCl and NaOH solutions. The sensitivity of the pH adjustment was around ±0.2 in all the experiments. All the trace elements considered in the present study showed a characteristic maximum leachability at lower pH than at higher pH values. It can be attributed to an increase in the attack on the mineral phases that contain these trace metals by hydrogen ions (Praharaj et al. 2002). Also at lower pHs the metals usually exist in an ionic state as they do not precipitate. For NFA the leaching order of metals from pH 2 to 10 (Fig. 2) is seen to be Pb > Cd > Zn > Mn > Cu, whereas that for MFA (Fig. 3) is Pb > Cr > Cd > Mn > Zn > Cu. The order of leachability, by and large, is not affected by the pH of the leaching fluid. But the amounts of element leached are greatly influenced by the pH of the system. The amount of element leached depends to a certain extent on the amount of trace element present in the fly ash. This also, to a large extent, controls the order of leachability of elements from fly ashes.

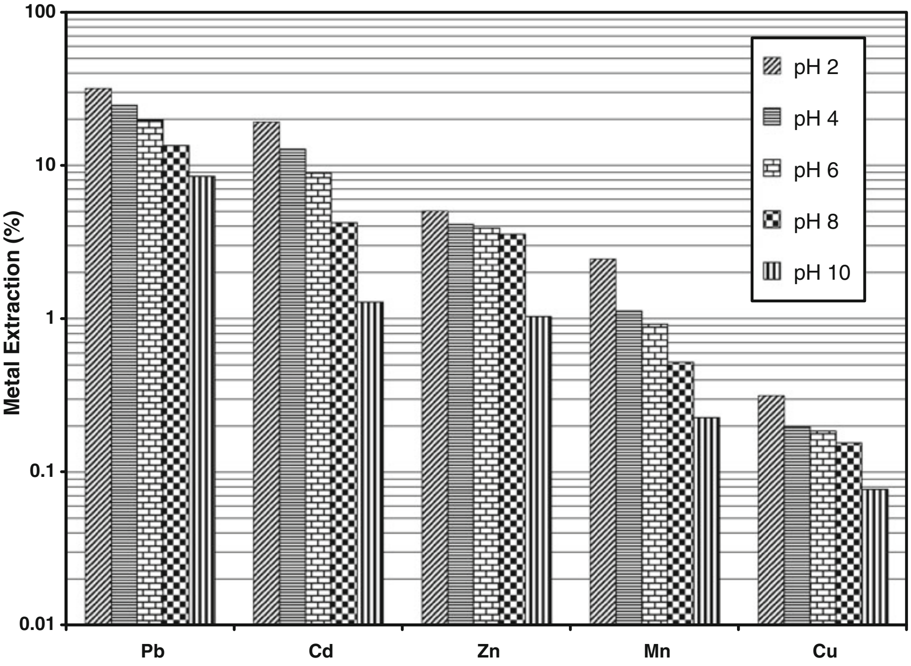

Fig. 2 Leachability of metal ions at various pH conditions from NFA

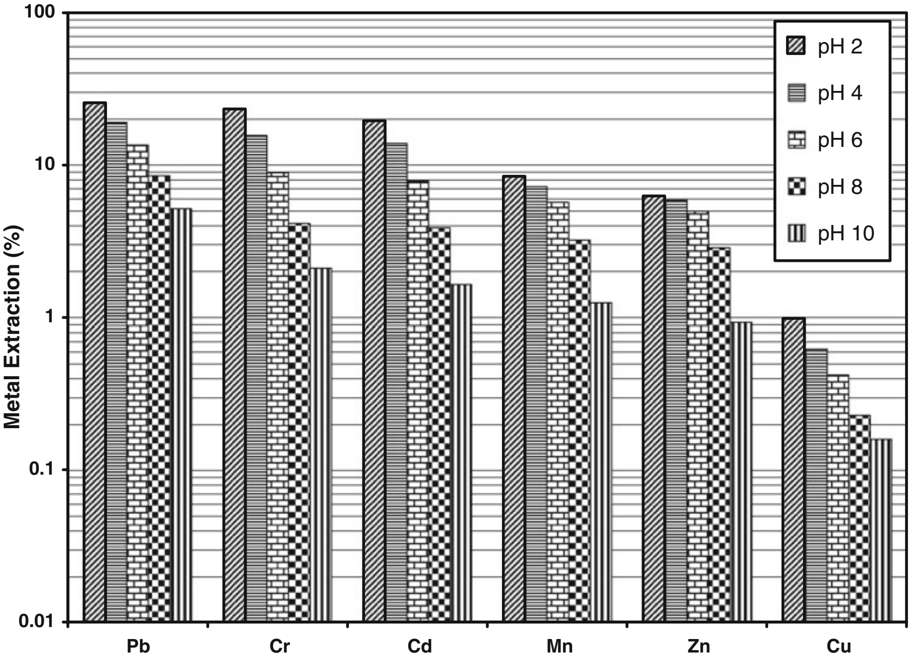

Fig. 3 Leachability of metal ions at various pH conditions from MFA

Effect of additives at different pH

It is an established fact that fly ashes to some extent produce cementitious compounds with curing time. The amount of compounds produced that increase with curing period basically varies with the composition of fly ash, namely the reactive silica and lime present. To increase the pozzolanic reactivity, lime and gypsum are amended at about 2.5% of the weight of fly ash. The effect of the compounds formed on the leachability of fly ashes is not well known. For this purpose the leachability values were also obtained for samples cured at both 28 and 180 days. The leachability of the samples after curing was studied at a *L/S* ratio of 20. During leaching tests the pH of the suspension was controlled and maintained constant at 2, 4, 6, 8 or 10 with the addition of 0.1 M HCl solution. The leachability of metal ions at various pH conditions from gypsum (2.5%) treated lime (2.5%) stabilized NFA and MFA after 28 and 180 days of curing period is presented in Figs. 4, 5, 6, 7 and 8. Every element showed a reduced leaching value at any constant

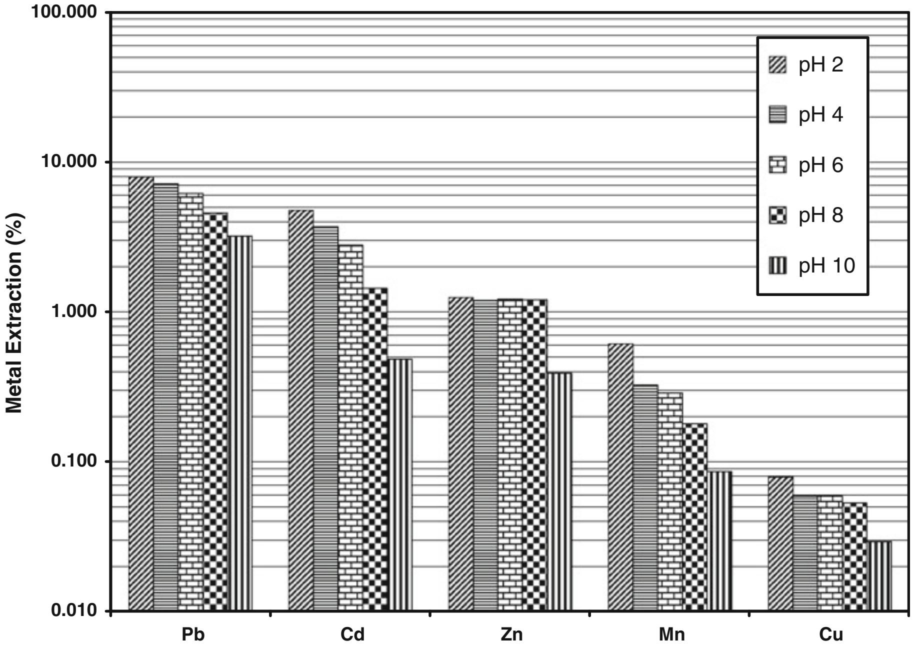

Fig. 4 Leachability of metal ions at various pH conditions from gypsum (2.5%) treated lime (2.5%) stabilized NFA after 28 days of curing period

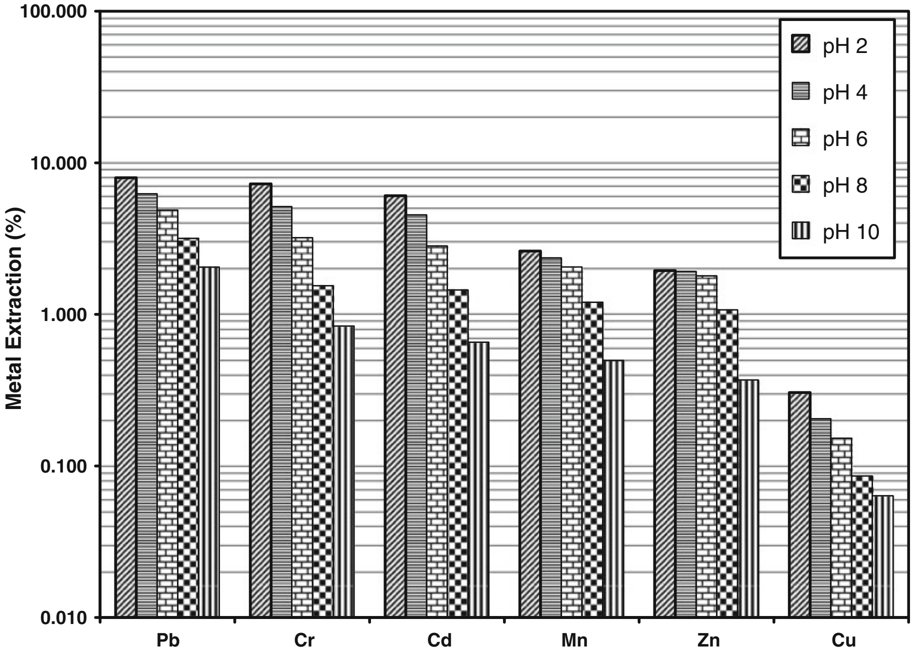

Fig. 5 Leachability of metal ions at various pH conditions from gypsum (2.5%) treated lime (2.5%) stabilized MFA after 28 days of curing period

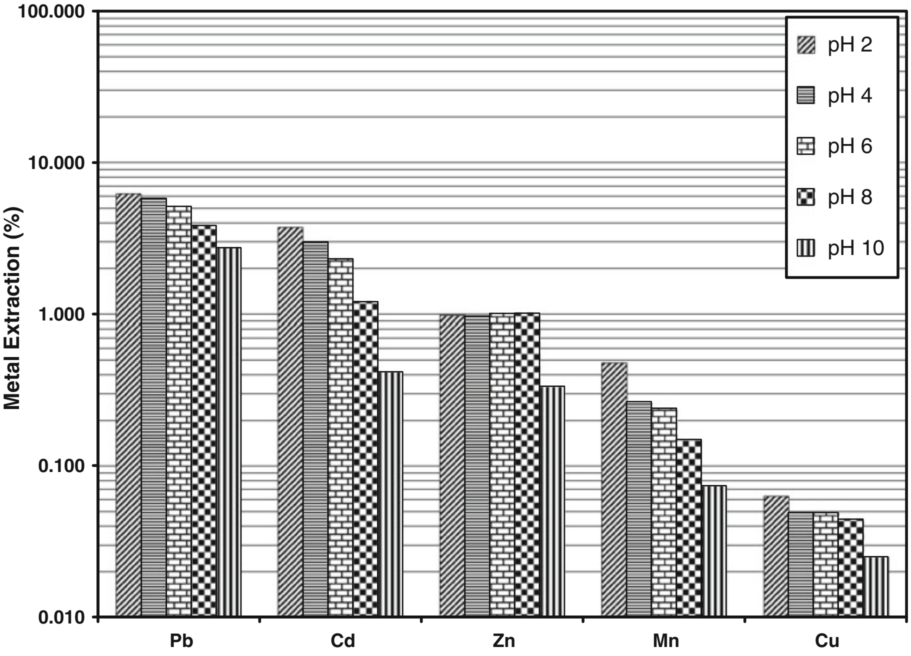

Fig. 6 Leachability of metal ions at various pH conditions from gypsum (2.5%) treated lime (2.5%) stabilized NFA after 180 days of curing period

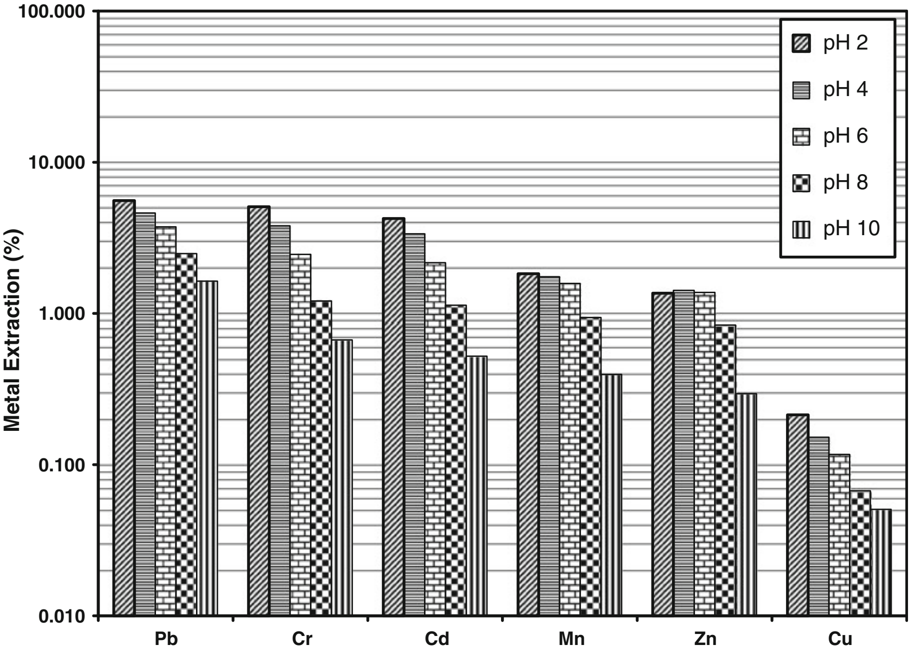

Fig. 7 Leachability of metal ions at various pH conditions from gypsum (2.5%) treated lime (2.5%) stabilized MFA after 28 days of curing period

pH value with an increase in curing period. Leaching values are observed to further reduce with increase in pH. However, the effect of pH could still be observed even after 180 days of curing. These results are summarized in Table 5. From the table it can further be seen that at pH 2 the maximum amount of leachability of Pb, which is 38.6 mg/g, reduced to 7.53 mg/g upon curing for 180 days for NFA, whereas from MFA it reduced from 24.72 to 5.36 mg/g after 180 days for Pb. The decrease in leachability values with an increase in pH is higher when additives are present. Thus, for Pb from NFA without lime and gypsum, while the leachability decreased from 38.60 to 10.26 mg/g, with additives it decreased from 7.53 to 3.33 mg/g as the pH was increased from 2 to 10. The order of leachability of metal ions for NFA at any given pH is Pb > Cd > Zn > Mn > Cu, which remains unchanged, whereas for MFA the leaching order that also remains unchanged is Pb > Cr > Cd > Mn > Zn > Cu.

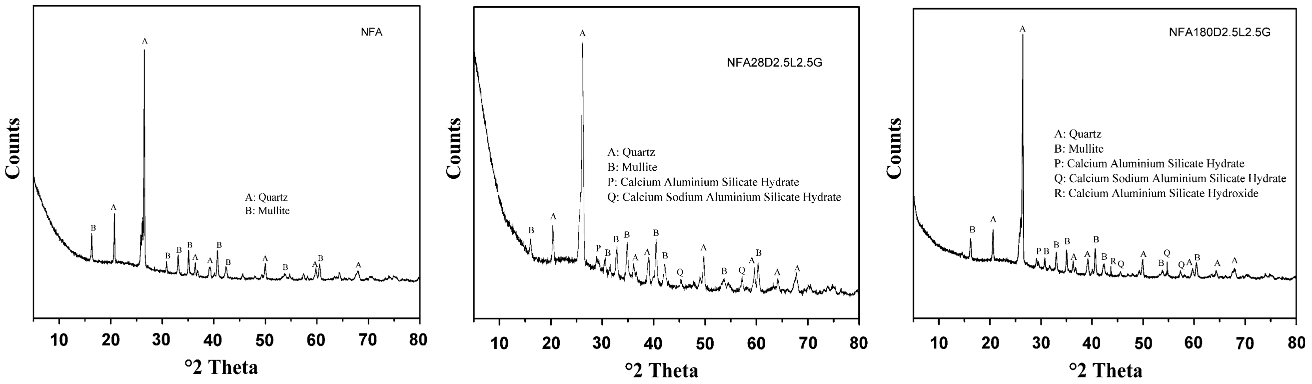

Fig. 8 X-ray diffractograms of NFA before and after amending with lime and gypsum at different curing periods

Role of improvement in strength

It is observed that the leachability of trace elements from both the fly ashes decreases upon curing because of the formation of cementitious compounds because of the addition of lime and gypsum, which enhances the retention of trace elements. The nature and amount of these compounds formed change with time. The dependence of leachability of trace elements on these changes has been attempted. The variation of unconfined compressive strength of both the fly ashes with lime and gypsum after curing for 28 and 180 days is given in Table 5. The unconfined compressive strength of NFA, which is 4,607 kPa after 28 days of curing period, increases to 5,181 kPa when cured for 180 days. For MFA the strength, which is 2,625 kPa at 28 days, increases to 3,496 kPa when cured for 180 days (Table 6).

The leachability of trace elements from both the fly ashes decreases with similar curing period. Thus, prima face it appears that as more and more compounds are formed, not only the strength increases, but also the leachability decreases. It is observed that the compressive strength increases with increase in curing period from 28 to 180 days, causing marginal decrease in leachability. This may be due to the amount of compounds formed: calcium aluminium silicate hydrate [d spacing of 3.09 Å, JCPDS (1999) 21-0133 for NFA (P); 3.06 Å, 42-1451 (P) and 2.83 Å, 46-1405 (P′) for MFA], calcium aluminium silicate hydroxide [2.07 Å, 29-0290 (R) for NFA] and calcium sodium aluminium silicate hydrate [1.99, 1.67 and 1.61 Å, 83-1655 (Q), for NFA]; initially and with a further increase in curing period, the gelatinous compounds transform to relatively more crystalline form. Thus, the peaks of these compounds are not sharp in samples cured for 28 days, unlike the peaks in samples cured for 180 days (Figs. 8, 9). The gelatinous compounds formed retain the elements better, and their release is inhibited.

Table 5 Comparison of leachability values at pH 2 before and after pozzolanic reactivity

Combination	UCC (kPa)	Leachability (mg/kg)					
		Pb	Zn	Mn	Cd	Cu	Cr
NFA	–	38.60	1.75	1.65	6.93	0.10	–
MFA	–	24.72	2.53	4.20	5.04	0.46	18.67
NFA after 28 days	4,607	9.65	0.44	0.41	1.73	0.03	–
MFA after 28 days	2,625	7.66	0.79	1.30	1.56	0.14	5.78
NFA after 180 days	5,181	7.53	0.34	0.32	1.35	0.02	–
MFA after 180 days	3,496	5.36	0.55	0.91	1.09	0.09	4.05

Table 6 Comparison of leachability values at pH 10 before and after pozzolanic reactivity

Combination	UCC (kPa)	Leachability (mg/kg)					
		Pb	Zn	Mn	Cd	Cu	Cr
NFA	–	10.26	0.36	0.15	0.46	0.02	–
MFA	–	4.97	0.37	0.62	0.42	0.07	1.68
NFA after 28 days	4,607	3.90	0.14	0.06	0.17	0.01	–
MFA after 28 days	2,625	1.97	0.15	0.25	0.17	0.03	0.67
NFA after 180 days	5,181	3.33	0.12	0.05	0.15	0.01	–
MFA after 180 days	3,496	1.57	0.12	0.19	0.13	0.02	0.53

Conclusions

Based on this study, it was concluded that the leachability of fly ashes increases with a decrease in pH of the fly ash liquid system. It also increases with an increase in *L*/*S* ratio. Fineness of particles has a decreasing effect on the leachability of the fly ashes. The leachability of trace elements decreases with an increase in the cementation of the

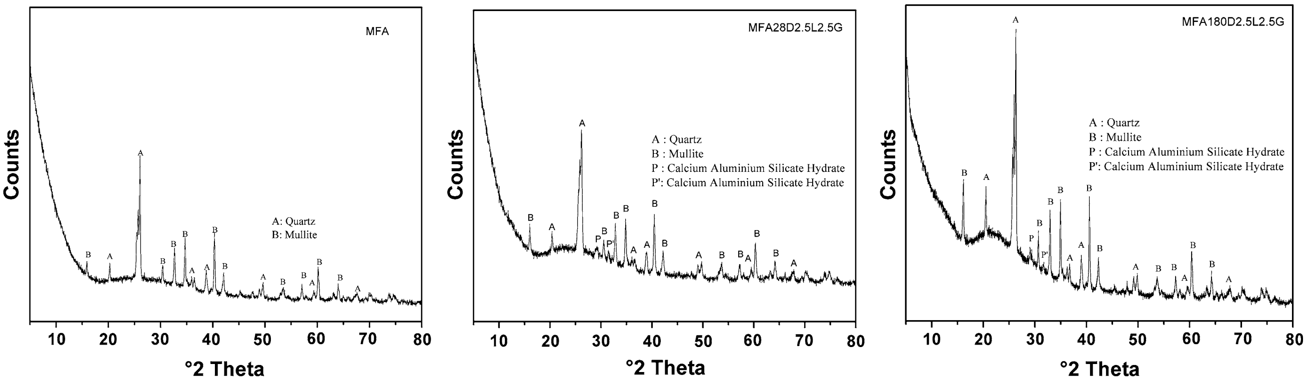

Fig. 9 X-ray diffractograms of MFA before and after amending with lime and gypsum at different curing periods

particles due to pozzolanic reactivity. The effect of a decrease in the cementation of particles with curing is seen even after leaching at low pHs, though at reduced levels, than at higher pHs.

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