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USE OF UP-FLOW PERCOLATION TEST TO ASSESS THE ENVIRONMENTAL PROPERTIES OF RAW AND TREATED FLY ASH

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Abstract

Fly ash (FA) is a by-product created from the burning of coal in thermal power plants. FA mainly consists of mineral compounds that make them naturally caustic. Despite this, research has shown that FA may be utilized as an alternative material in civil engineering, hydrotechnics and agronomy. Leaching tests may provide theoretical validation that FA may be used as an alternative sorbent or additive in construction material. In this paper, the results of up-flow percolation tests performed on raw FA as well as lime and cement modified samples (MFA) are presented. This method was chosen as it yields the most reliable assessment of releasing heavy metals under long-term exploitation. The research has focused on the leaching behaviour of Sb, As and Se in raw, lime and cement MFA samples. Raw FA was found to leach Se, As and Sb metal ions, cement modification did not prevent Sb and Se leachates, while lime-modified MFA was found to be the most stable material.

Key words: fly ash, heavy metals, leaching, up-flow percolation

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1. Introduction

Thermal power plants (TPP) in Serbia annually produce seven millions of tons of fly ash (FA). This by-product can be used as an adsorbent for the removal of heavy-metal pollutants, as a soil remediation agent or a medium for the reduction of carbon emissions (Abdelhadi et al., 2018; Karanac et al., 2018a, 2018b; Yao et al., 2015).

The environmental acceptability of fly ash as an alternative material may be determined through leaching tests (Izquierdo and Querol, 2012; Tiwari et al., 2015). It is crucial to evaluate FA leaching

behaviour in order to prevent the transferring pollutants into surrounding media.

For this reason, column tests are best-suited to resemble exploitation conditions and imitate leaching behaviour under field conditions, despite their being costly in terms of funding and time compared to others possible (Naka et al., 2016). Accordingly, up flow percolation tests were used on raw FA and lime and cement-MFA (Fatoba et al., 2013; Naka et al., 2016). Fig. 1 displays the potential environmental impacts that TPP may cause. In order to mitigate these risks, viable uses of FA must be found in which their application may end their cycle as a stored pollutant

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and their valorisation as a source material (Ciocan *et al.*, 2018; Jayaranjan *et al.*, 2014).

2. Pollutants

In the combustion of coal, emissions including resultant ashes are transported through a flue gas system (electrostatic filters and chimney) that have a general removal efficiency rate of 98.0 to 99.83 %. Larger particle sizes are easily captured through these systems, but smaller particles are emitted into the Earth’s atmosphere. The FA collected is removed and transported by a hydraulic system as a mixture of water to dump sites for slag and ash. Currently, efficient electrostatic filters represent the best available technology for collection FA from flue gas (Jaworek *et al.*, 2019). From the seven million tons of fly ash generated annually by Serbian TPPs, the FA can be used as efficiency adsorbent for heavy metal removal from water (Karanac *et al.*, 2018b).

Research focused on coal combustion and the relative composition of FA has established that each element has its own mechanism of transformation and

incorporation into FA during combustion (Koshy and Singh, 2016; Meawad *et al.*, 2010). The relative distribution between solids of fly ash, calcifications, flying particles and volatiles, is illustrated in Fig. 2.

Speciation analysis is crucial in order to define the dominant form and valence of each element and FA compound. Some elements that may also evaporate during combustion may otherwise still stay solid in ash due to the calcification that can occur in heavily calcified coal or in non-volatile minerals when alumina or silicates are present (Jayaranjan *et al.*, 2014).

3. Material and methods

The leaching test for the FA sample and the samples modified with 5% of lime or cement were carried out in accordance with the leaching behaviour test SIST-TS-CEN/TS 14405:2004. The sampling procedure was conducted by collecting a composite sample of FA from the Morava TPP’s FA dump site from October 2014 to April 2015 at intervals of once per week.

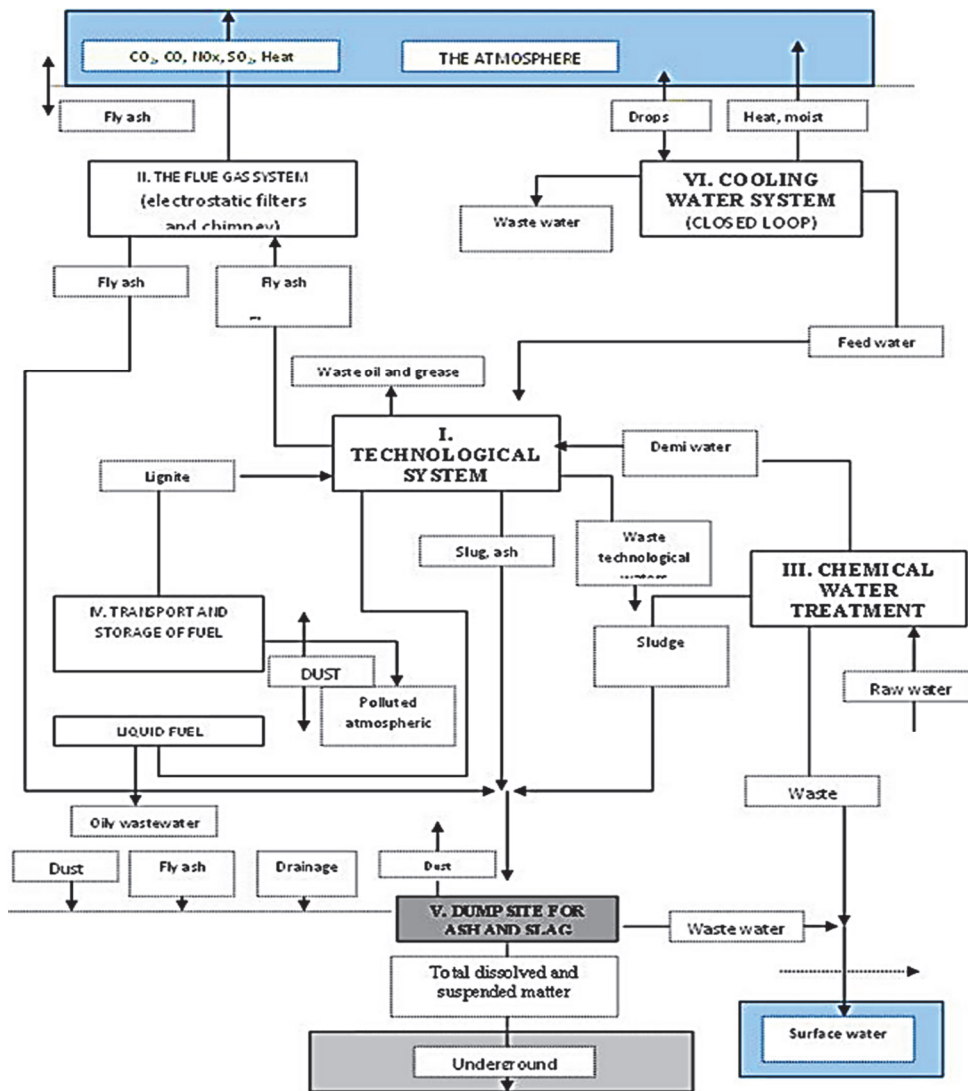


Fig. 1. Potential impacts of TPP on the environment

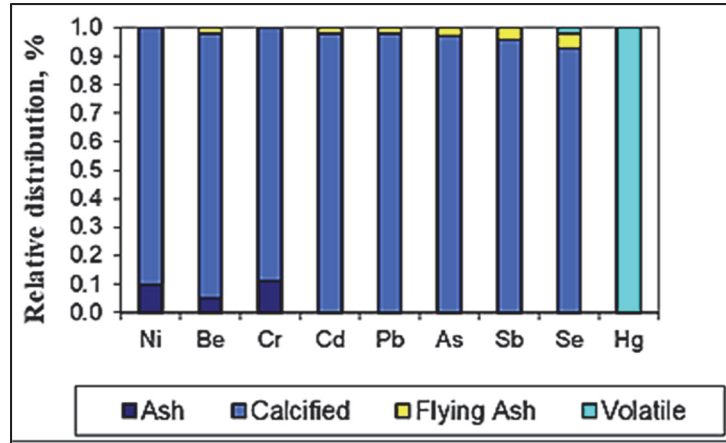


Fig. 2. The relative distribution of elements of FA in coal combustion (Visa, 2016)

The samples were prepared for the leaching test by drying at 105 C° for 2 hours, afterwards they were homogenized in a pestle and mortar. According to the particle size distribution, the FA samples were composed of silty and powdery sand, predominantly of an average diameter of $d < 75 \mu\text{m}$, where 60-71 % of the particle sizes ranged from fine sand to fine pebbly gravel. As stated in Standard for leaching tests, the performance of the test, meaning the repeatability and reproducibility is dependent on the material and experimental conditions. At the time when standard was adopted by European Committee for Standardization it was still not validated and the data on the repeatability and reproducibility, as well as on robustness were not available. According to standards set out by the American Society for Testing and Materials (ASTM), the investigation of raw FA

pertains to class F (Komljenovic et al., 2010). The FA sample in this study is defined as such (ASTM C618-08a, 2008). The MFA was produced by adding hydrated lime (calcium hydroxide, $\text{Ca}(\text{OH})_2$) or cement to mixtures of fly or bottom ash, which was then subjected to further experimental testing. On the basis of a stable FA composition (CaO (stable) 62-67%, SiO_2 19-25%, Al_2O_3 2-8%, Fe_2O_3 1-5%, SO_3 3-4,5%, CaO (free) 2%, MgO 5%, Na_2O and K_2O 0,5-1,3%) laboratory mixtures were prepared using 5% of hydrated lime ($\text{Ca}(\text{OH})_2$) for the MFA/lime, and, for the MFA/cement, 5% of cement. The mixtures were statically compacted and aged under controlled conditions for 28 days. Aged samples were installed into the apparatus, presented in Fig. 3, for the up-flow percolation test according to SIST-TS CEN/TS 14405:2004.

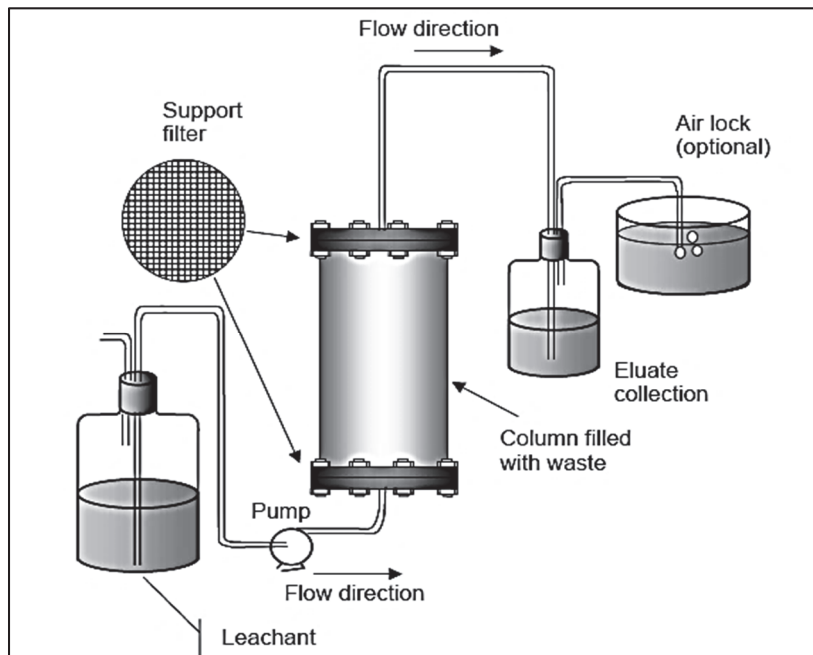


Fig. 3. Equipment for the up-flow percolation test

The apparatus for the leaching test consists of a column with an internal diameter of 100 mm and a filling height of 30 cm. Each specimen was of the following dimensions: diameter 100 mm and height 300 mm. The extraction solution was demineralized water, where electrical conductivity was up to 0.1 mS/cm. Eluates were taken in 7 separate samplings until a liquid to solid ratio (L/S) 10 was achieved.

3.1. Determination of dry residue

The dry residue of the analysed samples is determined when the L/S ratio is calculated. Dry residue (w_{dr}) requires treatment at 105 ± 5 °C. To calculate the dry residue, the (Eq. 1) has been used:

$$\omega_{dr} = m_d \times \frac{100}{m_r}, \% \quad (1)$$

where: w_{dr} represents the dry residue of the material, in %, m_r represents the mass of the undried test portion, in g, m_d represents the dry mass of the test portion in g.

To determine the dry mass (m_0) of the analyzed material, (Eq. 2) has been used:

$$m_0 = \frac{(m \times \omega_{dr})}{100} \quad (2)$$

where: m_0 represents the dry mass of the analyzed material, in kg, m represents the mass of the water content, in kg, w_{dr} represents the dry residue in %.

For the flow rate calculation, Eq.3 has been used:

$$\phi = V_L \times \pi \times d^2 \times 0.0104 \quad (3)$$

where: ϕ represents the leachate flow rate, in mL/h, V_L represents the linear velocity of the leachant through the empty column, in cm/day, d represents diameter of the column, in cm.

For every component, the quantities released in all eluate fractions were calculated as shown by Eq.4:

$$U_i = \frac{(V_i \times c_i)}{m_0} \quad (4)$$

where: i is the index of the eluate fraction (1, 2, ..., 7), U_i represents the released quantity of the component per quantity of the sample for analysis in the eluate fraction i , in mg/kg of dry matter, V_i represents the volume of the eluate fraction i , in L, c_i is the concentration of the component concerned in the eluate fraction i , in mg/L, m_0 is the dry mass of the test portion in the column, in kg.

For every component, the cumulatively released quantity (ΣU_i) shall be calculated by accumulating the released quantities of the specific component, measured in the different eluate fractions. When the concentration of a component in one or more eluate fractions is below the lower detection limit, two

calculations shall be carried out for this component as to indicate both the upper limit and the lower limit of ΣU_i (SIST-TS-CEN/TS 14405:2004, leaching behaviour test).

3.2. Chemical analysis

Analysis of the chemical ions in each fraction of the eluate was carried out using several different techniques: 1) inductively coupled plasma-optical emission spectroscopy (ICP-OES) for Zn, Ba and Se; 2) atomic absorption spectroscopy coupled with hydride generation (AAS-HG) for As^{5+} ; 3) graphite furnace atomic absorption spectroscopy (GF-AAS) for Pb^{2+} , Cd^{2+} , Ni^{2+} , $Cr^{3,6+}$ and Sb^{5+} ; and liquid chromatography (LC) for F^- and SO_4^{2-} . The apparatus used for inductively coupled plasma-optical emission spectroscopy (ICP-OES) was a Thermo iCAP 6500 system, equipped with Thermo iTEVA software, a concentric nebulizer and a Cyclonic Spray Chamber. External calibration was done according to Certipur Merck 1000 mg/L in single standard solutions. Calibration standards and samples were acidified with 65 % nitric acid (Trace Select from Sigma–Aldrich) to a final concentration of 2 % nitric acid as a calibration blank. The AAS apparatus used was AAS Perkin Elmer 4100ZL. For fluoride and sulphate concentrations, liquid chromatography (LC) was used in accordance with the ISO 10304:2007 method. The liquid chromatograph used to measure fluoride and sulphate concentration was the Dionex DX 100 IC system (Dionex, Sunnyvale CA, USA) possessing an IP20 isocratic pump and a CD20 conductivity detector. PeakNet 5.1 was used for instrument control, data collection and processing. Dionex IonPac AS14 (250×4 mm I.D.) and AG14 (50×4 mm I.D.) were used as columns.

The suppressor employed was an anion auto self-regenerating model (ASRS, 4 mm I.D.) operating in a recycle mode. A sample loop of a volume of 1000 μ L was made from a poly(ether ether ketone) (PEEK) tubing of length of 210 cm and I.D. 0.750 mm, whose volume was verified by measuring the mass difference between the sample loop filled with deionized water and the empty loop. A pH meter produced by WTW, type 340i was used to measure the pH values, according to the electronic method (Standard methods for the examination of water and wastewater, 22nd edition, 4500-H⁺-B).

4. Results and discussion

4.1. Heavy metal concentrations

The results obtained from analyzing the heavy metal leaching concentrations from the raw FA sample in fractions of eluate are presented in Table 1. As the raw FA exhibited excessive leachates of Sb, As and Se (Table 2), lime and cement were used to modify the FA in order to study possible reduction in leaching. The results of the MFA are provided in Table 2.

Table 1. Heavy metal concentrations in eluates from the raw FA sample

	<i>FRACTION</i>	<i>1</i>	<i>2</i>	<i>3</i>	<i>4</i>	<i>5</i>	<i>6</i>	<i>7</i>	<i>Calculated values</i>
	<i>L/S</i>	<i>0.15</i>	<i>0.27</i>	<i>0.58</i>	<i>1.10</i>	<i>2.12</i>	<i>5.22</i>	<i>10.06</i>	
<i>The parameter</i>	<i>Unit</i>	<i>Up-flow percolation test</i>							
pH	-	7.60	7.80	7.80	7.70	7.90	9.80	10.90	-
Sb	mg/L	0.01	0.01	0.01	0.01	0.01	0.01	0.05	0.32
As		0.07	0.08	0.08	0.11	0.10	0.15	0.28	2.16
Cu		0.10	0.10	0.10	0.10	0.10	0.10	0.10	1.05
Ba		0.07	0.04	0.04	0.04	0.05	0.05	0.02	0.36
Cd		0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.01
Ni		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.11
Pb		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.11
Se		0.05	0.04	0.03	0.03	0.03	0.03	0.03	0.32
Cr		0.06	0.06	0.02	0.01	0.01	0.01	0.01	0.12
Zn		0.99	0.31	0.13	0.05	0.10	0.05	0.05	0.74
SO ₄ ²⁻		1290	1190	1350	1490	882	134	53.	3212
F ⁻		0.05	0.05	2.0	0.05	0.05	1.2	0.30	5.88

Table 2. Heavy metal concentrations in raw FA and MFA after up-flow percolation tests

<i>Parameter</i>	<i>Unit</i>	<i>Sample</i>			<i>Percolation limit values associated with environmental characterization in mg/kg of dry matter</i>	
		<i>FA</i>	<i>MFA, 5% lime</i>	<i>MFA, 5% cement</i>		
<i>Measured values in mg/L</i>						
					<i>Underlying courses of capped pavement or shoulder sublayer</i>	<i>Covered engineering embankment or shoulder</i>
Sb	mg/L	0.32	0.10	0.53	0.4	0.2
As		2.16	0.30	0.23	0.8	0.05
Cu		1.05	0.10	0.11	50	50
Cd		0.01	0.00	0.05	0.32	0.16
Ni		0.11	0.20	0.11	1.6	0.8
Pb		0.11	0.20	0.21	0.8	0.5
Se		0.32	0.10	1.18	0.5	0.4
Cr		0.12	0.50	0.19	4	2
Zn		0.74	0.50	0.53	50	50
SO ₄ ²⁻		3212.54	293.30	354.64	10000	5000
F ⁻		5.88	0.10	4.56	60	30

As well as the maximum permissible values for the acceptability of alternative materials in road construction (Acceptability of alternative materials in road constructions, based on environmental assessment, 1207A-ISRN: EQ-SETRA—12-ED03—FR+ENG, 2012). These guidelines serve as reference to the potential of the reuse of fly ash in construction materials according to leaching limits permitted by law, particularly in road construction. The results indicate that MFA may significantly reduce the total leaching of heavy metals such as Sb, Se and As, (Chen et al., 2009, Sun et al., 2017) which can lead to its potential safe valorization as a re-used source material (Chen et al., 2018; Ungureanu et al., 2015).

The modifying agents are shown to possess adverse effects on the ionic adsorption of Sb, Se and As. Lime MFA best limited the leaching behavior of Se and Sb, while cement MFA was best at limiting As leaching. Nevertheless, there was no significant improvement when cement MFA samples were used, compared to raw sample of FA for Sb. In Fig. 4, 5, and 6, the removal efficiency for Se, As and Sb, respectively is presented for the raw FA, as well as lime and cement MFA samples. These results are in

accordance with erstwhile studies on the same leaching behaviors (Tian et al., 2019; van der Hoek et al., 1994).

4.2. Se removal

As already demonstrated by previous studies focused on Se behavior, when Ca is added to particles of ash, the reaction between Se and Ca will result in the production of a stable CaSeO₃ (Huggins et al., 2007a; López-Antón et al., 2006). Se and its species behavior are highly pH dependent, where, for values lower than 2.5, the dominant species are molecular forms of H₂SeO₃. As the pH value increases, ionic species (HSeO₃⁻ and SeO₃²⁻) become dominant that can be adsorbed and removed from the leachate (Otero-Rey et al., 2005). The most efficient sorption occurs when the pH value is neutral, approximate to a pH of 7.0 (van der Hoek et al., 1994). As the pH value rises, sorption decreases, whereby the desorption process occurs and the Se is released. Due to this phenomenon, the traceability and mobility of Se becomes even more significant for further study (Huggins et al., 2007b). The most recommendable

option for Se is the use of lime MFA, as to achieve more alkaline conditions (Fig. 4).

4.3. As removal

As with Se, the leaching of As is also complex (Hartuti *et al.*, 2017). pH value is again a key factor in its leaching behaviour. As releases from acidic FA as the latter's pH increases, whereas the trend is reversed in alkaline FA (van der Hoek *et al.*, 1994). When Ca is added to the matrix, the pH value becomes alkaline whereby the precipitation of Ca-arsenate occurs (Karanac *et al.*, 2018b). Fe and Al oxides are able to efficiently hold onto As if the pH value of the matrix is within 3.0 to 8.0 (Camacho *et al.*, 2009, Lata and Samadder, 2016). The affinity of arsenate for metal oxides decreases when the pH value is higher than 8.0, which must be taken into account for all leaching studies. As removal through co-precipitation by phosphates and carbonates, if present, have also been studied as a possible solution (Camacho *et al.*, 2009, Lata and Samadder, 2016). The most recommendable option for As is the use of cement MFA (Fig. 5).

4.4. Sb removal

Elevated concentrations of Sb in leachate can

be removed using several methods when applying FA (Ungureanu *et al.*, 2015). Sb often correlates to As due to their binding properties in naturally occurring geological formations. Inorganic Sb compounds are more toxic than organic, however, Sb(III) are far more toxic than Sb(V) species.

In order to correctly remove Sb present within samples, the exact type of Sb present in the matrix must be determined. While sorption has been applied to antimony removal from water, numerous factors also dictate sorption efficiency. Much like with Se and As, pH value is the greatest determinant of sorption efficiency; however, the presence of other ions in the matrix strongly affects Sb removal (Ungureanu *et al.*, 2015). The most recommendable option for Sb is the use of lime MFA (Fig. 6). Through these experiments it was established that pH value is key influencing factor when projecting efficiencies of heavy metal removal. Their tendency to increase the pH value determines overall removal (Herath *et al.*, 2017). FA itself can be a good material to prohibit Sb leaching due to its complex oxide structure, as has been shown within these tests. Raw FA, therefore, made be used an adsorbent for the removal of certain heavy metals, whose non-leaching nature made be applicable to its use within construction materials.

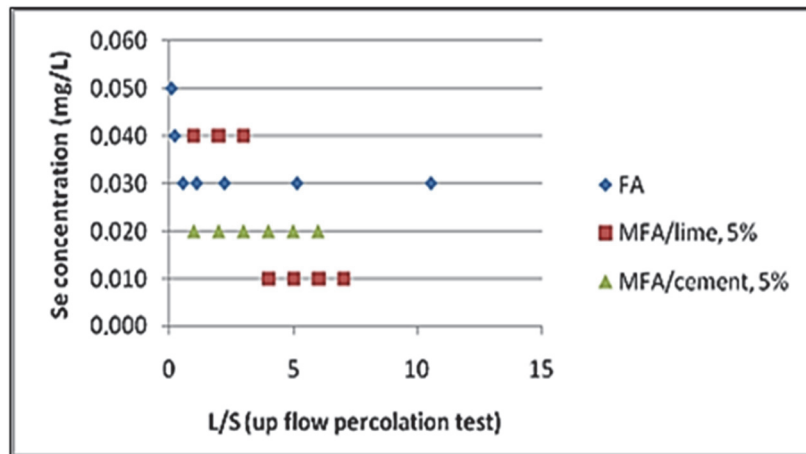


Fig.4. The efficiency of Se removal with raw FA, MFA/lime and MFA/cement

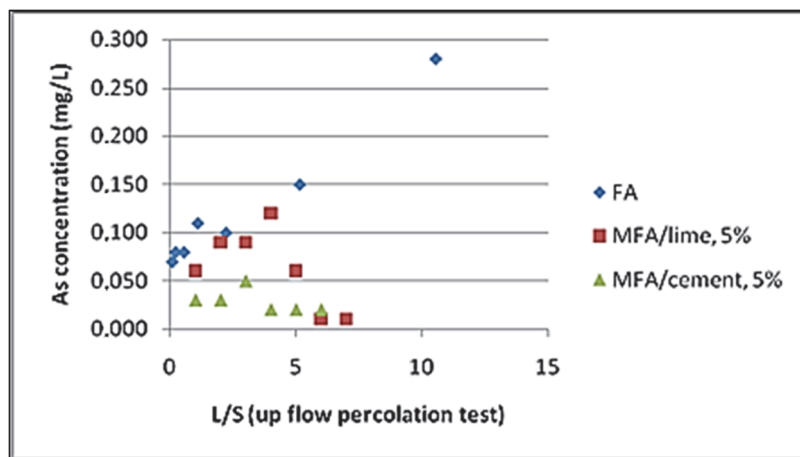


Fig.5. As removal efficiency with raw FA and MFA

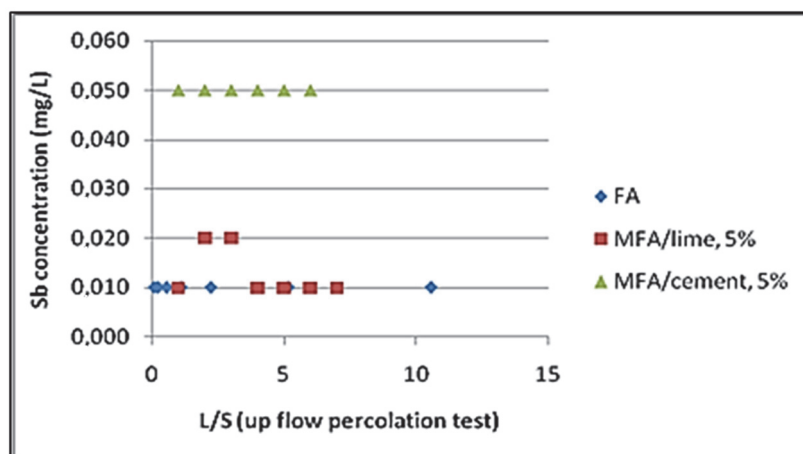


Fig.6. The removal efficiency of Sb with raw, and modified samples of fly ash

5. Conclusions

The environmental acceptability of FA as an alternative sorbent and potential additive used in construction materials has been validated through leaching tests reflecting varied mass/liquid ratios. For the leaching of Se, As and Sb metal ions, raw FA and modified MFA/lime and MFA/cement were found to demonstrate varying affects.

Regarding target/selected ions, both lime and cement modifications have obtained the best results of As (the lowest leaching performance). In general, MFA/lime has shown as the best leaching impregnation for selected ions, while MFA/cement was not efficient for Se and Sb. Cement MFA did not result in material stabilization, which may be attributed to the complex chemical composition of the cement itself.

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