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Review Procedure

All submissions will undergo an abstract review and a double blind review on the full papers. The abstracts would be reviewed initially and the acceptance and rejection of the abstracts would be notified to the corresponding authors. Once the authors submit the full papers in accordance to the suggestions in the abstract review report, the papers would be forwarded for final review. The final selection of the papers would be based on the report of the review panel members.

Format for Citing Papers

Author surname, initial(s.) (2020). Title of paper. i-manager's Journal on Material Science, 8(3), xx-xx.

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CONTENTS

RESEARCH PAPERS

EDITORIAL

This volume of i-manager's Journal on Material Science (JMS), (October – December 2020: Volume-8, Issue-3) has five peer reviewed research papers that covers diverse topics in Material Science. The aim of the journal is to address the latest developments in the field to meet the challenges of the present world. The current issue focuses on fabrication of lanthanum strontium iron oxide (LSFO) films from (La, Sr, Fe)-EDTA solutions with atmospheric sintering for metal-air secondary batteries, sink fabrication from limestone-clay reinforced polyester composite, thermal energy storage device using paraffin wax as phase change material, analysis of leaching rate of heavy metals from fly ash at varying leachant pH and cumulative liquid to solid ratios, and elastic analysis of functionally graded rotating annular disk with varying properties using ANSYS.

Komatsu et al. have focused on the fabrication of lanthanum strontium iron oxide (LSFO) films from (La, Sr, Fe)-EDTA solutions with atmospheric sintering for metal-air secondary batteries. Perovskite-type lanthanum strontium iron oxide (LSFO) films were synthesized on Si substrate with metal-ethylenediaminetetraacetic (metal-EDTA) complex. A commercial spin coater and furnace were used for the fabrication. Unique solution-based synthesis with compositional accuracy for metal-air secondary battery has been proposed.

Bolaji and Samuel have presented a sink fabrication from limestone-clay reinforced polyester composite. In this study ceramic filled polyester composite material has been developed by adding micro-fine limestone and clay ceramic particulates with particle size of $<134\mu m$ to unsaturated polyester resin with a loading weight of 20% limestone and 30% limestone/clay and fixed amount of catalyst (1% weight). Resin Transfer Molding (RTM) technique has been used to fabricate a prototype sink using wooden and Plaster of Paris (POP) mold with a view to examine surface finish.

Omkar have made a thermal energy storage device using paraffin wax as phase change material. This paper demonstrates the thermal energy storage using phase change material (PCM), which is one of the effective methods. In this paper, a 'two-way heat exchanger system' is designed in which thermal energy is stored and hot water can be utilized during daytime as well as night. The performance of PCM is then compared with two different flow rates.

Agwe et al. investigated on the analysis of leaching rate of heavy metals from fly ash at varying leachant pH and cumulative liquid to solid ratios. Unutilized fly ash is dumped into the environment and this continued disposal of FA into the environment makes the heavy metals contained therein to move out in the leachate generated, polluting the soil, surface and ground water sources among others. This research shows some new ways in utilization of FA and focus on overall improvement in the current modes of FA utilization to minimize its negative impacts on the environment.

Jain and Mishra have presented an elastic analysis of functionally graded rotating annular disk with varying properties using ANSYS. Functionally graded materials are characterized as an anisotropic material whose physical properties varies continuously because the dimensions vary randomly or strategically to realize the specified characteristics. A functionally graded annular disc is analysed for different profiles like convex, concave, linearly varying and uniform with different angular velocity, and ceramic material considering exponentially varying material properties.

Ramarao has experimented on determination of input set through multi-objective optimization of PMEDM output parameters using modified TOPSIS. Input parameters identification and selection along with its range is an important task to proceed with the major experimentation in research work, especially when working with some specific methods in DOE. In this paper, the selection of range of values for 'Pulse ON Time' is discussed. Based on the results, better values for pulse ON time were identified which are proposed to be used in DOE.

We extend our sincere thanks to the authors for their contribution towards this issue and we are grateful to the reviewers for spending their quality time in reviewing these papers. Our special thanks to the Editor-in-Chief, Dr. Chithirai Pon Selvan for his continuous support and efforts in further improving the quality of the Journal.

Hope this issue imparts an enlightening reading experience! Enjoy reading!.

Warm regards,

Christal K.
Technical Editor
i-manager Publications

ANALYSIS OF LEACHING RATE OF HEAVY METALS FROM FLY ASH AT VARYING LEACHANT pH & CUMULATIVE LIQUID TO SOLID RATIOS

By

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ABSTRACT

Fly ash (FA) is a particulate matter consisting of finely divided, non-combustible particles obtained from the flue gases arising from combustion of coal, accounts for over 80% of the total ash produced during coal combustion. In 2018 alone, about 780 million tons of FA has been generated globally, of which voluminous quantity remained unutilized, hence dumped into the environment. This continued disposal of FA into the environment makes the heavy metals contained therein to move out in the leachate generated, polluting the soil, surface and ground water sources among others. In this study, 5 sets of leaching test columns were packed with an equal quantity of air dried fly ash samples and each of them leached with leachant of pH 5.87, 6.08, 6.41, 6.46 and 7.01 and eluate from each column collected at cumulative liquid to solid (L/S) ratios in l/kg of 0.1, 0.2, 0.5, 1.0 and 2.0. Analysis of the eluate for Copper (Cu), Selenium (Se), Zinc (Zn), Cadmium (Cd), Lead (Pb), Nickel (Ni), Chromium (Cr) and Arsenic (As), revealed that the concentrations of Se at Selenium L/S of 0.1 for leachant pH of 5.87, 6.41 and 7.01, exceeded the allowable limits for non-hazardous wastes disposal into the landfills.

Keywords: Eluate, Fly Ash, Heavy Metals, Leaching, Leachant, Liquid to Solid Ratio, pH.

INTRODUCTION

Peavy et al. (2017) defines fly ash (FA) as a particulate matter consisting of finely divided, non-combustible particles obtained from the flue gases arising from combustion of coal and its particle size ranges from 1-1000 µm. According to Tripathy et al. (2019), the world wide annual coal FA generation stood at 780 million tons in 2017. In the same period, India alone generated 196.44 million tons of FA, contributing about 25% of the annual coal FA generation. Lokeshappa and Dikshit (2012) argues that disposing fly ash in ponds, dykes and landfills provides moist condition which favors the metals contained therein to leach out into the environment. Leaching in this context refers to the removal of soluble components from a solid matrix (Kim, 2003). The material being leached is called a Leachee, the liquid used to leach as a leachant and the soluble component coming out from the solid matrix during the field and laboratory leaching processes are called the leachate and eluate respectively. Mathematically, leaching process can be expressed as,

$$\text{Leachee} + \text{Leachant} \quad \rightarrow \quad \text{Leachate/eluate}$$

According to Kim (2005), leaching of FA is an area of serious environmental concern. Praharaj et al. (2002) and Gong et al. (2010) and many other researchers state that leachate coming out from FA during leaching process contains potentially toxic substances which pollutes the soils, surface and groundwater once it comes into contact with them.

The above threats posed by leaching of FA calls for a comprehensive study on its leaching behavior to give impetus for its effective management and idea of devising new ways of utilization of FA and thrust on overall improvement in the current modes of FA utilization to minimize its negative impacts on the environment.

1. Literature Review

1.1 Leaching Test Methods

Kim (2005) stated that there are more than 100 leaching tests methods with no particular single accepted as a universal method. These methods can be broadly categorized as monolithic and bulk, batch, column, standard test methods and pH dependence leaching test methods among others. Leaching tests can also be classified based on whether the leaching fluid is a single addition (static extraction tests) or is renewed (dynamic tests), as per Kim (2005), Leiva et al. (2018), Tiwari et al. (2015) and also as suggested in many other literatures.

The column leaching test method which has been used in this study, simulates the flow of percolating groundwater through a porous bed of materials which are granular in nature. It assumes that the leachant is uniformly distributed and therefore all the areas of the test portion of the granular material get exposed to the leachant. According to Grathwohl (2014), Gong et al. (2010) and Kim (2005), column leaching test method is the one which is the closest to the natural leaching systems than those of the batch shake systems and has been embraced in the past years to conduct leaching studies on contaminated soils, wastes such as fly ash, recycling and construction materials. Tiwari et al. (2015) further alludes that despite being more expensive and operationally more complex than the batch leaching method, the column leaching test method still yields better results which is almost the same as those obtained from the real leaching systems. This is subjected to fluid flow and solute transport and it further helps to reveal the temporal variations in the leaching behavior of the metals/elements/compounds of concern which is a major limitation in batch leaching. The advantages of the column leaching test method over the others, offered a firm foundation upon which it has been employed in the current study.

1.2 Leaching Behavior of FA from Previous Studies

According to Praharaj et al. (2002), concentrations of metals is higher at lower L/S ratios and the concentrations decreases with increasing L/S ratios. In particular, they asserted that the cumulative concentrations of Arsenic, Manganese and Molybdenum in the eluate all exceeds the limits recommended by the World Health Organization. Their concentration plus that of iron also exceeds the allowable concentrations for drinking water as outlined in the United States Environmental Protection Agency guidelines for the same.

In the leaching process of fly ash and other coal utilization by-products, heavy metals leach more in acidic environment. Other elements form oxy-anions leach under alkaline environment while trace elements are only slightly soluble under all pH conditions. In general the chemical or mineralogical and physical properties of fly ash plus the leachant composition is heavily influenced by pH which affect the rate of leaching of different elements/compounds (Kim, 2005).

Kim (2005) suggested that out of the main compositions of fly ash, silicates and alumino-silicates are comparatively insoluble, oxides slightly soluble and sulfates highly soluble in water. Choi et al. (2002) also revealed that elements like K, Na, Cu and Ca leach out faster in the initial stages of leaching, but metals/elements still remains detectable in the leachate even after washing it several times with distilled water giving indication of a slow and long term leaching behavior of certain elements from fly ash. This study also reveals a positive influence of fly ash leachate from fly ash disposal mound on the quality of groundwater where the levels of Cr (VI) of some fly ash samples exceeded the limits recommended by EC directives for non-hazardous wastes.

2. Methodology

The fly ash sample used in this study has been collected in a dry state, from the electrostatic precipitators of NTPC, Thermal Power Station, Bongaigaon (Assam). It has been stored in an airtight plastic bag and brought to the laboratory of the Central Soil and Materials Research Station (CSMRS), New Delhi, India, from where the tests were conducted. The sampling and preservation of the sample has been done in accordance with the provisions of Bureau of Indian Standards (2005). A total of 0.86 kg dry mass (M_0) of the sample has been packed into 5 sets of a 5 cm diameter glass columns in accordance with the procedures presented in European Committee for Standardization (2004), sections 7.2 and 7.3 and the packed columns were saturated in accordance with annex B, (3) part (2) of European Committee for Standardization (2004), each with stock solutions (leachant) of different pH. The dry mass (M_0) obtained using the mentioned Procedure is very important in the calculation of crucial parameters used in this study such as the eluate fraction volumes and the sampling duration of each of these eluate fraction volumes, has been calculated in accordance with equation (2) in European Committee for Standardization (2004) part 7.3.

Another very important parameter, the dry residue (W_{rd}), which is very important in the calculations of the dry mass of the sample packed in the up-flow leaching columns and the eluate volume, Equation (1) in European Committee for Standardization (2004) part 6.4 has been used to calculate this important parameter.

Equation 3 of European Committee for Standardization (2004) part 7.4 has been used in the calculation of the flow rate (Φ) of the leachant through the columns. The flow rate is very important in determining the collection time of each eluate fraction volume (European Committee for Standardization, 2004).

In determining the number of eluate fractions and respective volumes to be collected from each column, the procedures given in the Table 2 of European Committee for Standardization (2004), and in particular Note No. 2. Therefore, five fractions of eluate were collected from cumulative L/S ratio of 0.1 to cumulative L/S ratio of 2 for each of the fly ash packed column leached with leachant of different pH. The assumption made here is that the unutilized quantity of fly ash is disposed into the landfills with fill material laid on top of it to reduce the rate of leaching. This should be the actual practice, since wet disposal of fly ash is the common method of disposal, poses more threats to the environment than the dry disposal method. (Note: The procedures, equations and technical specifications can be found by referring to CEN/TS 14405:2004 European Committee for standardization (2004) and are not reproduce in this article).

After packing the sample into the columns, saturating it and calculating the other important parameters as mentioned. The volumes of eluate fractions calculated were each collected within the time schedule and each analyzed for Copper (Cu), Selenium (Se), Zinc (Zn), Cadmium (Cd), Lead (Pb), Nickel, total Chromium as (Cr) and total Arsenic as (As) in accordance with the provisions of American Public Health Association (2017). These parameters (trace metals) analyzed are under the scope of American Public Health Association (2017), and International Organization for Standardization (2005).

3. Results and Discussions

The Table 1 and Table 2 show the results for the dry residue (W_{rd}), dry mass of fly ash sample packed in the leaching column (M_0), leachant flow rate (Φ), eluate liquid to solid ratio fraction of dry mass of fly ash (L/S) fractions,

Parameters	Value Obtained	Remarks
Dry residue (W_{rd})	99.7 % by weight	Value obtained used in the calculation of dry mass of fly ash packed into the column
Dry mass of fly ash packed in the column (M_0)	0.86 kg	The mass of air dried sample packed in the column (M_0) has been 0. 86 kg. The value of M_0 obtained has been used in the calculation of eluate fraction volume and collection time required for each fraction.
Leachant flow rate (Φ)	12 ml/hour	The value obtained is the same as that predetermined in section 7.4 of European Committee for Standardization (2004), when a similar column size is being used in the leaching study.

Table 1. Parameters and Values of Leaching Study

Parameters		Value Obtained		Remarks
L/S fractions of dry mass of FA (1/kg)	Cum. L/S fractions of dry mass of FA (1/kg)	Calculated Eluate volume (1)	Collection period from start of leaching (days)	
0.1	0.1	0.086	0.3	The eluate volume for each column leached with leachant of different pH were collected within the calculated collection period and analyzed for pH, electrical conductivities and trace metals and results analyzed.
0.1	0.2	0.086	0.6	
0.3	0.5	0.258	1.5	
0.5	1.0	0.430	3.0	
1.0	2.0	0.860	6.0	

Table 2. Parameters and Values of Fractions of Eulate

cumulative L/S fractions, eluate volume for each cumulative L/S fractions and eluate collection time from start of the leaching process for each cumulative L/S fractions of the fly ash test portion packed in the column and subjected to an up flow column leaching studies.

These results have been discussed under the remarks column. The results of the impact of the leaching behaviors of leachant of pH 5.87, 6.08, 6.41, 6.46, and 7.01 is shown in Figure 1, 2, 3, 4, and 5 respectively.

The following analysis have been made on the impact of leachant pH and cumulative liquid to solid (L/S) of dry mass of fly ash on the leaching rates of the said heavy metals.

- Regardless of the pH of the leachant, cadmium has been below detectable limits at all L/S fractions.
- Total chromium as Cr has been detectable only when the pH of the leachant has been more acidic (5.87) than the rest and this has been observed at cumulative L/S of 0.3 with the value being 0.03 mg/l. This therefore implies that leaching of Cr occurs at a later stage of the leaching process and it's more pronounced when the leaching fluid is more acidic. The Cr content detected when the pH of the leachant has been 6.46 at cumulative L/S of 0.1 and 0.2 with values of 0.004 mg/l and 0.001 mg/l respectively seems to have come from the stock solution as it has also been detected in the stock solution itself. Further decrease in its concentration with time further concretes the above argument. The maximum value of 0.03 mg/l observed above still falls below the maximum allowable of 0.05 mg/l for drinking water as prescribed in Bureau of Indian Standard (2012).
- Traces of copper (Cu) could be detected under all leachant pH conditions with more concentrations at

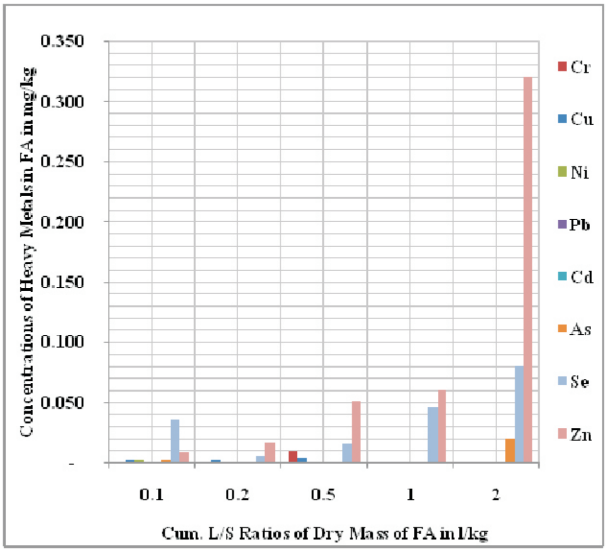

Figure 1. Leaching Behaviors of Trace Metals when FA Is Leached By Leachant of pH 5.87

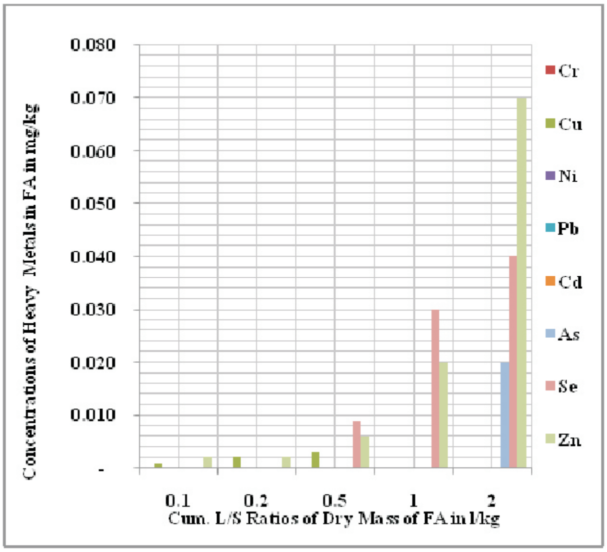

Figure 2. Leaching Behaviors of Trace Metals when FA is Leached by Leachant of pH 6.08

acidic conditions and at the start of the leaching process followed by a general decrease in its

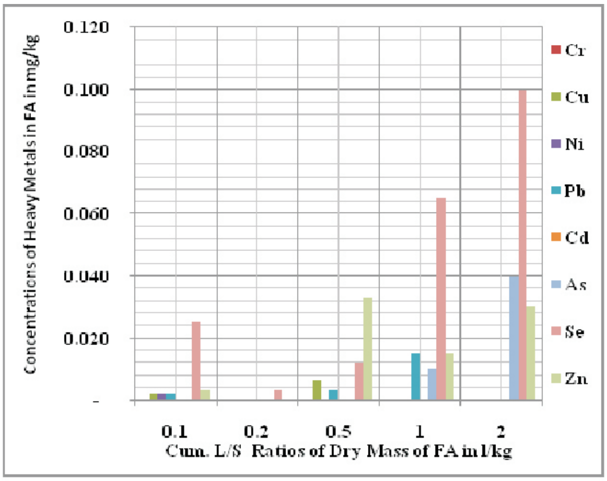

Figure 3. Leaching Behaviors of Trace Metals when FA is Leached by Leachant of pH 6.41

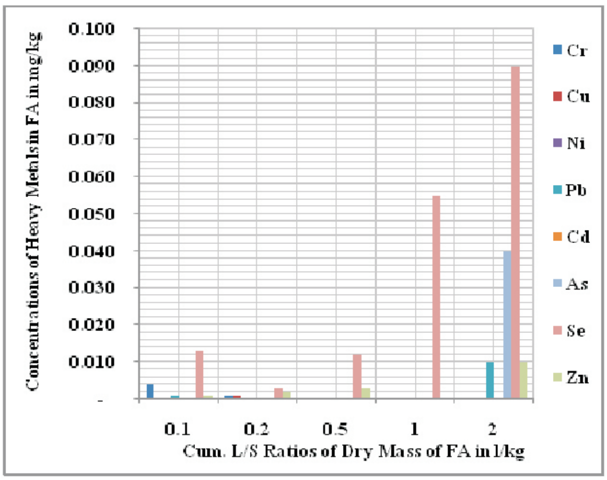

Figure 4. Leaching Behaviors of Trace Metals when FA is Leached by Leachant of pH 6.46

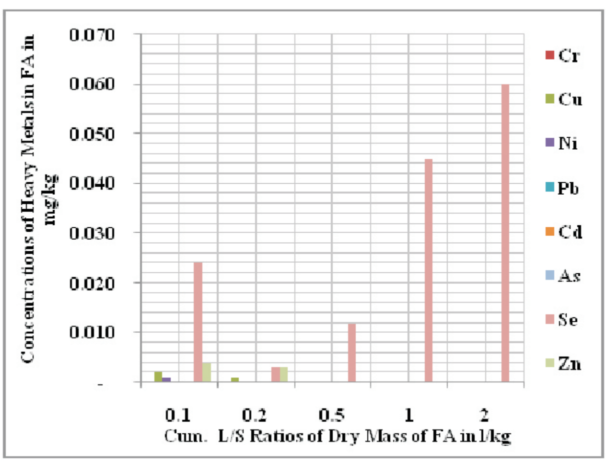

Figure 5. Leaching Behaviors of Trace Metals when FA is Leached by Leachant of pH 7.01

concentration with time. The maximum concentration of copper leached out has been 0.02mg/l, this value is below the maximum value of 0.05 mg/l provided in Bureau of Indian Standard (2012).

- Nickel could only be detected at cumulative L/S of 0.1 with higher concentrations when the leachant pH has been below neutral and these values were just detected at the start of the leaching process. This could therefore inform that more Ni leaches under acidic medium at the start of the leaching process. The maximum value of 0.02 mg/l is equals to the maximum value of 0.02 mg/l provided in Bureau of Indian Standard (2012).
- The pH range at which Lead leaches more seems to be narrow as there has been more leaching at leachant pH of 6.41.

  At this pH there has been a general increase in the concentrations of lead with the maximum value being 0.03 mg/l. All detected values of lead at leachant pH of 6.46, 6.41, 7.01 and 6.08 were all either equal to or higher than the maximum allowable (0.01 mg/l) as provided in Bureau of Indian Standard (2012). No lead has been detected under a more acidic condition (pH of 5.87) perhaps implying higher acidity impedes the leaching of lead.
- Arsenic leaches at a later stage of the leaching process with an increasing rate as time passes by and also more leaching occurring at an averagely acidic condition of pH of 6.46 and 6.41. No arsenic (As) has been detected when the fly ash leached by a neutral leachant. The maximum value of 0.04 mg/l obtained at leachant pH of 6.41 and 6.46 and at cumulative L/S ratio fraction of 2 l/kg has been much higher than the maximum allowable of 0.01 mg/l as provided in Bureau of Indian Standard (2012). This value has also exceeded at leachant pH of 5.87 at cumulative L/S of0.1 l/kg and 2 l/kg and at leachant pH of 6.08 at L/S of 2 l/kg and lastly at leachant pH of 6.41 at L/S of 1 l/kg.
- Selenium (Se) leaches more at the start of the leaching process with more leaching at a more acidic medium. This leaching rate decreases with time and it's the most

prominent problem as leaching of fly ash is concerned as at all pH of the leaching fluid. At all L/S, it leached at a much higher concentrations as compared to the maximum allowable concentrations of 0.01 mg/l. The maximum leaching occurred at the most acidic condition (leachant pH of 5.87) and at cumulative L/S 0.1 l/kg. This maximum concentration (0.35 mg/l) is 35 times higher than the maximum concentrations of 0.01 mg/l as provided in Bureau of Indian Standard (2012).

- Leaching rate of Zinc (Zn) progressively increases with time with more leaching taking place when the leachant is more acidic. All the values obtained with the maximum being 0.32 mg/l at leachant pH of 5.87 (the most acidic condition) and at cumulative L/S of 2 l/kg were all below the maximum allowable concentrations of 5 mg/l as provided in Bureau of Indian Standard (2012).

Conclusion and Recommendations

The following conclusions and recommendations have been drawn by the authors.

- Comparing the concentrations of the heavy metals analyzed, it can be deduced that under all pH conditions of leachant, the concentrations of Selenium exceeded the Bureau of Indian Standard (2012) acceptable limits of 0.01 mg/l. The concentrations of Pb also at L/S of 0.1 and 0.5 when leachant pH has been 6.41 also exceeded the acceptable limits of 0.01 mg/l as prescribed under Bureau of Indian Standard (2012). Arsenic also exceeded the acceptable limits of 0.01 mg/l when pH has been 5.87 at cumulative L/S of 0.1 and 1, when pH of the leachant has been 6.41 at L/S of 0.5 and 1.0 and when the pH of 6.46 at L/S of 1.0.
- The Selenium concentrations of 0.35 mg/l, 0.24 mg/l and 0.25 mg/l at L/S of 0.1 and at leachant pH of 5.87, 7.01 and 6.41 respectively all exceeded the allowable limits of Article 16 of Annex II to Directive 1999/31/EC Decision, Council of 19 December 2002 No. (2003/33/EC) for nonhazardous wastes requiring disposal into the landfills. On this note therefore, the authors recommend that no fly ash should be disposed into the environment, instead many more noble application areas should be sought of to achieve its 100% utilization (EUR – Lex, 2002).
- Since the concentrations of Selenium, Lead and Arsenic in the eluate analyzed exceeded the allowable limits prescribed in Bureau of Indian Standard (2012), water samples around mounds with this fly ash is being damped regularly and examined for the same.
- The future studies should be extended to analyze the spatial extent of pollution of environmental parameters such as soil, surface and ground water from within the fly ash disposal areas by these heavy metals/elements and other compounds contained therein the leachate. Concentrations of these heavy metals in the biota within such areas should also be analyzed on how they spatially vary.

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