EXTRACTION OF ELEMENTS FROM COAL FLY ASH
USING THERMO-HYDROMETALLURGICAL METHOD

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Received 14 April 2016
Accepted 20 May 2016

ABSTRACT

The investigations integrate thermo and hydrometallurgical methods for fly ash treatment collected from thermal power Plants (TPP) in Bulgaria. The first stage of the autoclave treatment of the proposed process using sulfuric acid with concentration 20 and 30 mass % at thermal treatment temperature of 100, 120 and 140°C and time of 60, 90, 120, 240, 360 and 480 min, was studied. In the second stage, the obtained samples were dissolved with water at constant temperature and time. Based on the results from chemical analyses the extent of extraction of 12 elements was determined. The data obtained show that the method used can be considered successful for extraction efficiency of 88.8 % for Al, 88.5 % for Fe, over 80 % for Ca, Cu and Mn, over 70 % for Mg, Cr, Mo and nearly 100 % for other elements – Na, Zn and Ni. The extraction behaviour depends on all studied parameters.

Keywords: solid wastes, fly ash, bottom ash, acid solubilization, extraction, utilization.

INTRODUCTION

The combustion of coal for electricity generation in TPP produces solid wastes such as fly ash (FA), bottom ash (BA), boiler slag and Flue Gas Desulphurization (FGD) materials, which are commonly known as coal combustion by-products (CCPs) [1]. In a report by the International Energy Agency [2], a number of factors were identified as having significant impacts in respect to CCP quality and quantity. In Bulgaria, coal-fired power plants supply more than 40 % of the electricity generating about 5.5 - 6.5 millions tons of solid wastes annually [3]. Less than a quarter of them are being used, mainly in the concrete production.

The usage of lignite in power generation has led to increasing environmental problems associated both with gaseous emissions and also with the disposal of the ash residues. The conventional method for disposal of TPP solid wastes is the landfilling with impact to the environment [4]. Some elements, contained in the FA, are likely to be released from the storage/disposal application site when the ash comes in contact with water. The ecosystem appears to be an excellent filtering machine that retains contaminants such as metals in its soil profiles [5].

Alternatively, coal fly ash (CFA) can be pointed as an untapped resource with huge potential. There is therefore an expanding interest in developing other methods for commercial exploitation of CFA, particularly as a substitute for other resources. The initiatives undertaken by many researchers through technology development have turned around the perception of fly ash (FA) from a “waste material” to that of “resource material”. The application of FA in manufacture of bricks, cement, concrete, ceramic products, building materials composites, road base, mineral filter in asphalt mix, as well as, zeolites, geopolymers, has been proved to be beneficial in agriculture [6, 7].

The most widespread and powerful method to extract the metals from the TPP solid wastes includes acids because the high concentration of hydrogen ions induces the solubilization of inorganic components. Any mineral acid can be used for decomposing the aluminium – containing raw materials; however only sulfuric, hydrochloric, nitric, or hydrofluoric acids have practical significance. A low pH that will be obtained with the strong mineral acid will result in high leaching of many elements. The influence of the acid concentration, the temperature, the extraction time, the solid:liquid ratio,
The dissolution of alumina along with the other elements from FA using H₂SO₄ with different concentrations, in the range of 3 N to 36 N has been investigated [7]. The boiling temperature was within the range of 150 - 200°C, depending upon the concentration of the acid. The maximum of Al extraction was 84.17 % when 36 N H₂SO₄ and S/L ratio = 1:2 were used. The acid leaching is not selective for aluminum as Fe, Mg, K, Na and Ca generally dissolve simultaneously with aluminum.

The leaching of coal, and wood-ashes in sulfuric acid needed to maintain pH 1, temperature 25°C and solid: liquid ratio (S/L) = 1:10 as a function of time. The acid consumption and the extracted metal concentrations were also studied [12]. The kinetics of acid consumption in the case of ashes indicated an initially fast process, followed by a slower period, possibly extending longer than 10 days. Metals of the major elements (Fe, Mg, K, Na, Mn and P) were leached up to 80 - 100 %. Aluminium was leached to a lesser degree (68 %), possibly indicating the presence of stable ceramics, resulting from combustion at the high temperature.

The method of statistical design was used to optimize the factors affecting the extraction efficiency of the alumina from oil shale ash which is a solid byproduct of oil shale processing. The interactions between the reaction temperature, reaction time and sulfuric acid concentration have been studied. An efficiency of 89.70 % can be obtained at the following conditions: temperature of 100°C, the reaction time of 4 h and the H₂SO₄ concentration of 50 mass % [13].

A new method for recycling of the aluminium and the iron from FA and the boiler slag (BS), wasted from a coal combustion plant, integrates two processes: an obtaining of sintered pellets and acid extraction of Al and Fe from the sintered pellets [11, 15]. The main objective of the sintering step is to provide strong materials and also to break the crystalline mullite phase (3Al₂O₃·2SiO₂) rendering free Al₂O₃ that is available to be leached [14]. Fly ash was palletized with fine coal and calcium oxide using a ratio of 5:4:1 to produce pellets. The subsequent calcinations were performed at a temperature ranging from 1000 to 1200°C [11]. The boiler slag was palletized together with a washed coal and a lime at a ratio of 50:40:10 and 40:40:20, 60:30:10 and 70:20:10, respectively, and then pellets for the leaching test were sintered [15]. The effect of the temperature, the mixing time, the concentration of sulfuric acid and the solid to liquid ratio of sintered pellets and H₂SO₄ on the recovery of Al and Fe was studied. An aluminium and iron efficiencies of 81 and 71 %, respectively, have been obtained when the sintered pellets reacted with 6.12 mol dm⁻³ sulfuric acid, a solid: liquid ratio of 1:5, at 80°C for 4 h [10]. The maximum extraction efficiency of Al from boiler slag was 86.55% and 94.6% for Fe, respectively, using sintered pellets with ratio of slag, coal and lime 50:40:10 and 4 mol/L H₂SO₄, at solid: liquid ratio of 1:5 at 80°C for 24 h [15].

The aim of the present research is to investigate the removal of metals and other valuable elements from TPP fly ash using combined extraction techniques. The investigations integrate a thermo-hydrometallurgical treatment of FA, generated from TPP in Bulgaria. As a first stage in the proposed process an autoclave FA treatment is used. Sulfuric acid with different concentrations, various temperatures and reaction time were studied. The second stage is the solubilization of samples, obtained at constant value of water, temperature and time.

**EXPERIMENTAL**

**Materials**

The fly ash sample is collected from Enel Maritza East 3 TPP, which is one of the three large power plants in the Maritza East power complex, all of which are supplied with locally mined lignite from the Maritza East Mines. The power plant is situated in south central
Bulgaria.

A composite sample of FA was randomly taken from a landfill and re-sampled by the quarter method. The sample obtained was first dried at 100°C to the water content of 1 mass %, grinded and sieved to obtain a fraction below 0.63 mm and stored in closed desiccators for further studies.

As a leaching agent a sulfuric acid solution was used. The solutions with different concentration were prepared by mixing different volumes of reagent grade H₂SO₄ (Merck, Poole, Dorset, UK) with distilled water.

**Methods**

**Chemical analysis**

Fluorescence spectrometry (XRF) was used for chemical composition analysis and ICP-MS (Inductively Coupled Plasma-mass spectrometry) was used for elemental analysis.

**XRD and SEM-EDX Analysis**

The mineral composition and morphology of the fly ash were studied using X-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD analysis was performed on the apparatus “TUR-M62” with Cu-Kα radiation. Step-scanning data were collected from 10 to 70° 2θ, with a fixed time of 3s per 0.05° 2θ. Scanning electron microscope combined with energy dispersive X-ray (EDX) spectroscopy (Hitachi SU-70, Ultra-High-Resolution Analytical FE-SEM equipped with Bruker s X Flash Silicon Drift Detector) is also used during the experiments.

**FT-IR spectrometry and DTA-TG Analysis**

Fourier transformed infrared (FT-IR) transmission spectra were measured on a Mattson 7000 FTIR spectrometer in the range of 400 – 4000 cm⁻¹ using KBr pellets. The thermal behaviour of the FA was investigated with differential thermal analysis (DTA) and thermal gravimetric analysis (TGA). TGA and DTA were performed with a Labgys TG-DTA 16 setaram instruments. An experiment was run in air atmosphere and the sample was heated with a heating rate of 5°C min⁻¹.

**Experimental procedure**

The recovery of valuable elements from FA under thermo-hydrothermal conditions was examined. A 1g representative sample of FA was added into a 100 ml Teflon reactor. A 26 cm³ of H₂SO₄ with different concentration (20 and 30 mass %), was transferred to the reactor containing ash. The amounts of H₂SO₄ used are calculated on the basis of chemical content of oxides in the sample and at a constant value of liquid to solid ratio. The resulting slurry was autoclaved at 100, 120 and 140°C in the sealed reactor for different time of treatment (60, 90, 120, 240, 360 and 480 min), respectively. The leachate and the leached residual ash, obtained after thermo-hydrothermal treating were transferred with a constant value of distilled water in a glass reactor and stirred at 50°C for 1 h. The leached residual ash was separated from the solution by filtration. Distilled water was used to remove all of the residual leachate that was absorbed on the leached ash. The leachate liquor and the wash solution completed with distilled water to certain volume. The content of major and trace elements in the liquid phase were determined by means of ICP-OEP (Varian Vista-MPK).

**RESULTS AND DISCUSSION**

**Chemical analysis**

The chemical composition of the main elements in the fly ash sample is presented in Table 1. In general, the results indicate that the fly ash is composed mainly of SiO₂, Al₂O₃, and Fe₂O₃.

**Physical chemical analyses**

**XRD and SEM analyses**

XRD analysis indicated that the fly ash is a mixture of inorganic compounds and the detected phases (Fig. 1) include minerals such as SiO₂ (quartz), NaCa₄Al₃Si₅O₁₉.

Table 1. Chemical composition of the fly ash sample.
(plagioclase), KAlSi₃O₈ (orthoclase), NaAlSi₃O₈ (albite), CaAl₂Si₂O₈ (anortite), Fe₂O₃ (hematite) and Fe₃O₄ (magnetite).

The SEM images in Fig. 2 (a, b) show an availability of spherical and prismatic crystals and aggregates with irregular shape. Prismatic crystals are with size of up to 6 μm, which are associated in aggregates (Fig. 2a). Spherical crystals are with size nearly 30 μm (Fig. 2b). Some of these crystals are hollow and more of them are covered with lamellar crystals length of 7 μm and height 2 μm. The difference in the crystals morphology does not lead to chemical content difference of the crystals, which is proved by EDX analysis (Fig. 3). The results show the highest content of Si (18 %), Al (11 %) and Fe (6 %). An availability of small quantities of Ca, K, Mg is also observed.

**FT-IR spectrometry and DTA-TG Analysis**

Fig. 4 shows the FT-IR spectrum of the fly ash sample. The bands observed at 3436 and 1629 cm⁻¹ are attributed to stretching vibration and bending vibrations of H-O-H, respectively. Asymmetric stretching vibration of T-O-Si (T= Si or Al at 1068 cm⁻¹ and symmetric
stretched vibration of Si-O-Si at 792 cm⁻¹ are observed. Absorption bands in the region 750 - 560 cm⁻¹ are assigned to symmetric stretching vibration of Si-O-Si and Al-O-Si. The band at 460 cm⁻¹ is attributed to bending vibration of Si-O-Si and O-Si-O.

Fig. 5 represents differential thermal analysis/thermo-gravimetric results. The pore water evaporation starts from the room temperature nearly to 150°C. Apart from the loss of moisture, losses of volatile elements may also occur. A little activity can be observed in DTA curve from 500°C to about 760°C, with a mass loss probably due to the volatilization and destruction of various organic pollutants in the ash.

Results from Extraction Process

Extraction efficiencies of major elements

The influence of the concentration of sulfuric acid, the temperature and autoclaving time are considered to find the optimal extraction efficiency of the valuable elements. On the basis of the results from the chemical analyses obtained, the extent of extraction (α) of the major and other elements in the liquid were estimated.

\[
\alpha = \frac{G'}{G^i}, 100 \ % \ \text{w/w}, \quad (1)
\]

where:
G’ - the mass of the element in the liquid phase after
treatment according to the used method, g;

\[ G'' \] - the mass of the element in the initial sold sample, before treatment according to the used method, g.

The dissolution behaviour of the major studied elements (Al, Fe, Ca, Mg, K and Na) as a function of the temperature, the time of thermal treatment (TTT) and the \( \text{H}_2\text{SO}_4 \) concentration are presented on Figs. 6 - 11. As seen from the figures all studied parameters influence the
extraction of these elements. $\alpha$ value for Al, Fe, Mg and K increases essentially for all investigated temperatures with increasing of the time, at a constant value of $H_2SO_4$ concentration. The largest variation of $\alpha$ value depending on the change of the temperature, time of thermal treatment and acid concentration, is observed for Al, Fe, Mg and K. The $\alpha$ values for Al vary from 34.6 to 88.8 % (Fig. 6), between 30 and 85.5 % for Fe (Fig. 7), between 27 and 72.8 % for Mg (Fig. 8) and in the interval from 23 to 64.6 % for K (Fig. 9). It is observed that there is a gradual increase of $\alpha$ for these elements with an increasing of the TTT by constant values of the TMTT and $H_2SO_4$ concentration. As seen from the data (Fig. 6, 8) the higher acid concentration has positive effect on $\alpha_{Al}$ and $\alpha_{Mg}$ at constant other conditions. A maximum of 72.8 % $\alpha_{Mg}$ was achieved at the longest time of thermal treatment (360 and 480 min), independently of the TMTT. When the acid concentration and the duration of thermal treatment are constant with increasing of the temperature, $\alpha_{Mg}$ increases continuously for the experiments with 20 % $H_2SO_4$. Equalization in the values of the extraction extent for Mg was obtained with 30 % $H_2SO_4$ at 100°C and treatment time 360 and 480 min. The same effect was observed at the higher temperature of 120 and 140°C, by the same acid concentration after 240 min treatment time. The highest extent of aluminum 88.8 % was achieved at 120°C and 140°C using 30 % $H_2SO_4$ at the longest time of thermal treatment of 480 min.

Fig. 7 illustrates lower $\alpha_{Fe}$ at 100°C for lower acid until 120 min, equivalent or higher values at 120°C in the interval of 60 to 240 min and higher values after 120 min at 140°C, comparing the results with 20 and 30 % $H_2SO_4$. 

Fig. 12. Zn extraction efficiency ($\alpha$) as a function of the acid concentration, the temperature and the time of thermal treatment.

Fig. 13. Ni extraction efficiency ($\alpha$) as a function of the acid concentration, the temperature and the time of thermal treatment.

Fig. 14. Cu extraction efficiency ($\alpha$) as a function of the acid concentration, the temperature and the time of thermal treatment.

Fig. 15. Mn extraction efficiency ($\alpha$) as a function of the acid concentration, the temperature and the time of thermal treatment.
The extent of K extraction (Fig. 9) has lower values in comparison with the extent of Na extraction (Fig. 11). The increasing of the TTT has a positive effect on α_k at constant values of the temperature and acid concentration. In the experiments with 20 % H_2SO_4, α_k is nearly constant after 120 min TTT at 140°C. When 30 % H_2SO_4 was used the same effect has been observed, but the α_k has lower values compared with the data with 20 % H_2SO_4 at the same other conditions.

For other elements (Ca and Na) with increasing of the thermal time treatment by a constant values of other parameters the difference between α values decreases and at definite conditions has constant values (Figs. 10, 11). This tendency can be seen for α_Ca at temperature of 140°C and acid concentration 20 % and 30 % after 90 min of thermal treatment. The data for α_Ca show that values of nearly 100 % can be achieved at all investigated temperatures and times with all H_2SO_4 concentrations. One can see that the full extraction of Na can be obtained at 100°C after 120 min with both investigated H_2SO_4 concentrations. At the higher temperature (120 and 140°C) the same effect is observed after 90 min and after 60 min TTT for 20 % and 30 % concentration of acid, respectively.

The decrease in aluminium extraction at higher acid concentration is probably due to the low mass transfer rates of reactants and products caused by the increase in the quantity of CaSO_4 formation in the slurry mixture [16]. A similar phenomenon was also observed by Seidel and Zimmels [10], which postulated that increasing acid concentration produced two opposing effects simultaneously. An increase in the hydrogen ion enhances the dissolution of aluminium, whereas the increase in the concentration of the sulphate ions and dissolved calcium ions intensify the formation of calcium precipitates. The precipitates hinder mass transfer across the ash particle, because they form barrier on the surface and within the pores of the fly ash particles during the metal dissolution thus causing a resistance to mass transfer. Increase of the sulphate ions in the reaction mixture with the increase of acid concentration and super saturation of calcium facilitate the precipitation of CaSO_4 [8, 18].

**Extraction efficiencies of the trace elements**

The dissolution behaviour of the trace elements studied (Zn, Ni, Cu, Mn, Cr, Mo) as a function of the studied parameters is presented on Figs. 12 - 17. For all investigated temperatures with increasing the time of thermal treatment and constant value of sulfuric acid concentration, α_Zn (Fig. 12) and α_Ni (Fig. 13) increase. The data indicate that the extraction extent of Zn and Ni has higher values at the lower H_2SO_4 concentration, independently of the temperature at the same time of thermal treatment. α_zn has values over 80 % if the time is prolonged more than 120 min and the concentration of H_2SO_4 is 20 % for all investigated temperatures. As can be seen complete Zn extraction was achieved using 20 % H_2SO_4 at the longest time of thermal treatment at 100°C, and for time of 360 and 480 min for the samples obtained at 120 and 140°C. The same tendency for changing of extraction degree can be seen by comparing the experimental data obtained for Zn (Fig. 12) and those for Ni (Fig. 13). The difference is that an equality of the Ni extraction degree was achieved when the temperature was raised to 120°C and 140°C, and the leaching period was 240, 360 and 480 min, respectively, using 30 % H_2SO_4. At these conditions α_Ni varied between 80.5 and 81.9 % and between 83 and 84.2 % at temperature 120 and 140°C, respectively. The achieved maximum value of 100 % for Ni was obtained when solubilization was made with 20 % H_2SO_4 at 120°C and reaction time over 240 min, and at temperature of 140°C and time over 90 min, respectively.

The dependence of Cu extraction as a function of temperature and time of thermal treatment as well acid concentration is shown on Fig. 14. For the extraction procedure the higher extraction extent appear to be related primary to the H_2SO_4 concentration. Data show heigher values of α_Cu using 20 % H_2SO_4 at an equivalent conditions of solubilization. A significant influence of the thermal time treatment can be observed for experiments at temperature of 100°C by the two acid concentration used (20 and 30 %). When the temperature was increased up to 120°C and 140°C, the α_Cu obtained have nearly similar values if the time was increased by a constant value of acid concentration. The achieved maximum is 89.45 % in the conditions of thermal treatment at 100°C for time of 480 min with 20 % sulfuric acid.

The dependence of Mn extraction on thermal time treatment at different temperature and acid concentration is shown on Fig. 15. The increasing of the temperature has visible possitive effect on the solubility for the experiments with 20 % sulfuric acid in the studied interval times of thermal treatment. When H_2SO_4 concentration is
20 % for time over 90 min an equalization of values can be seen \((\alpha_{\text{Mn}}, \text{nearly 81\%})\) at temperature 140°C, which is the achieved maximum for this element. When the experiments were carried out with 30 % sulfuric acid the extraction degree of Mn increases with increasing of the time and reaches nearly constant values for time over 120 min at all temperatures, studied. As a result of this equalization the maximum of \(\alpha_{\text{Mn}}\) achieved is 75 %, 74 % and 72.5 % at temperature 100, 120 and 140°C, respectively.

The dissolution of Cr as a function of the studied parameters is shown on Fig. 16. The \(\alpha_{\text{Cr}}\) values vary in a large interval, from 23.6 to 81.2 %, depending on the change of the temperature, time of thermal treatment and acid concentration. The duration of the time has a positive effect on the extraction for the temperatures studied, independently from the acid concentration. In the conditions of 20 % acid concentration, \(\alpha_{\text{Cr}}\) has higher values compared to the results when 30 % \(\text{H}_2\text{SO}_4\) was used at a constant temperature. The extent of Cr extraction reaches a maximum of 85 % at 140°C, for time of thermal treatment 480 min with 20 % \(\text{H}_2\text{SO}_4\).

The extent of Mo extraction shows higher values when the investigations were made with lower concentration of sulfuric acid (20 %) compared with the results obtained with 30 %, at similar other conditions (Fig. 17). As can be seen from the figure a maximum of \(\alpha_{\text{Mo}}\) can be observed if the time of thermal treatment was prolonged to 120 min for nearly constant values at all temperatures and with two concentrations of the \(\text{H}_2\text{SO}_4\) used in the study. The extent of Mo extraction reaches the highest value of 70 % at 100°C for 120 min with 20 % sulfuric acid.

The recovery of the valuable elements from different types of fly ash is related to several factors, including specific element solubility and availability or release potential. A powerful method to extract the metals and other elements from TPP solid wastes includes acids, as the high concentration of hydrogen ions induces the solubilization of inorganic components. For the extraction of aluminium from alumina containing minerals the acid leaching processes are generally preferred. Such leaching process is also dependent on the type of acid used and on the type of the fly ash studied. The low value of the pH obtained with the strong mineral acids, resulted in high leaching of many elements. From an economical point of view, the usage of sulfuric acid as a lixiviant for the leaching processes is preferable. However different factors were investigated in the sulfuric acid leaching process. The basic studied factors are: acid concentration, the liquid:solid ratio, the fly ash particle size, the time of acid treatment, the temperature of acid solubilization and others.

Several investigations were made using direct and indirect leaching methods to extract aluminium. The direct extraction of aluminium and iron was studied at ambient temperature [11,15]. The results indicate poor extraction efficiency of alumina (10 – 12 % \(\text{Al}_2\text{O}_3\)) if the experiments were performed with 4M or 6M \(\text{H}_2\text{SO}_4\) at a solid:liquid ratio of 1:4 even after 24 hour of leaching. Indirect leaching of pellets of boiler slag mixture with coal and lime, obtained at sintering temperature and sintering time about 1000°C and 120 min, respectively, has a significant effect on Al efficiency in acid leaching [15].
The results from the experiments carried out with municipal solid wastes [16] and 1.5 M sulfuric acid, liquid:solid ratio 10:1, and time of 24 hours at a room temperature, are similar to our results. The data show high leaching extent of many elements (Al, Fe, Mg, Zn, Mn and Cu) corresponding with our results obtained.

A combined effect of temperature and time on leaching of coal fly ash in 6 M sulfuric acid at a solid to liquid ratio 1:4 has given an extraction extent of 23.5% at 75°C after 8 h 45 min of leaching time [17].

The experimental design based on the statistical modeling used to evaluate the relationship between sulfuric acid concentration, reaction time and reaction temperature was studied to determine the alumina extraction from oil shale [13]. The optimal extraction efficiency of 89.71% can be obtained when extraction is performed with 50% mass sulfuric acid at temperature of 100°C and time of 4 hours. The extraction process was carried out with liquid:solid ratio 3:1 at constant stirring.

The extent of leaching obtained for more of the investigated major and minor ash components can be explained also with mineralogical and chemical composition of the fly ash waste [7, 11-13, 15, 16, 18, 21]. A high extraction value of aluminium and other elements can be a result of the preliminary thermal treatment of the fly ash wastes.

Our results show high extraction extent of the major elements (Al, Ca, Na), over 80% and over 60% for Mg and K. This fact indicates that in the conditions of the experiments used, the transformation of the stable aluminium silicate (orthoclase, albite, anorthite) and plagioclase in acid soluble compounds occurs. The availability of the amorphous glassy matter in the fly ash structure appears to be the most likely source of Al, Ca, K and Na after extraction [19, 20]. The structure and composition of the glass may enhance the reactivity and leachability of the Al. Those glasses modified with alkaline earth elements show a more open structure that is susceptible to chemical attack and enhanced leaching [9]. Our results are in accordance with this assumption. The lower of Ca extent of extraction is probably due to the low mass transfer rates of reactants and products caused by the increase of the quantity of CaSO₄ formation in the slurry mixture [9, 17].

The combination of autoclave thermal treatment with sulfuric acid and wet extraction can be an effective alternative for the fly ash treatment. The data obtained show that the used combined method can be considered like successful for extraction of Al together with other valuable elements. The results from experiments indicate, that this method is not selective for Al extraction, as the other elements like Fe, Ca, Mg, Na, Ni, Mn, Cu and Zn generally dissolve simultaneously with aluminium. The extraction behaviour of all investigated elements depends on the all studied parameters. Obviously stable minerals, like orthoclase, albite, anorthite and plagioclase which contain in fly ash can be destroyed and as a result the mobility of the elements increased and the most of the valuable elements can be almost completely extracted.

CONCLUSIONS

REFERENCES