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Beneficiation of iron and aluminium oxides from fly ash at lab scale

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ABSTRACT

During electricity production, the fly ash (FA) is generated from the combustion process and it is a pollutant. Present investigation was carried out for the recovery of iron oxide and alumina from fly ash. The fly ash was subjected to magnetic separation where part of iron oxide was separated. After this the fly ash was leached with hydrochloric acid followed by precipitating the leached liquor with ammonia hydroxide to get hydroxides of alumina and iron which are dehydrated at high temperature (1100 °C) to obtain iron and aluminium oxides as a mixed powder. Leaching of alumina and iron oxides from fly ash with hydrochloric acid, the effects of four parameters viz. acid concentration and acid to fly ash ratio, leaching temperature and time were investigated and optimum conditions were (i) concentration of HCl 6 N, (ii) fly ash (g) to HCl acid (ml) ratio 1:4, (iii) temperature of leaching 107 °C, and (iv) duration of leaching 5 h. The total recoveries of iron oxide and aluminium oxide were 63% and 73%, respectively.

1. Introduction

In India annual generation of coal fly ash (CFA) is over 130 million tons from thermal power plants (Burke, 2007) and this amount is likely to increase in time with coal consumption increase which is 2.2% (Ahmaruzzaman, 2010). In 2002, it was estimated that 25% of fly ash was used in cement production, construction of roads, brick manufacture (Bhattacharjee and Kandpal, 2002) and in 2009–10 approximately 32 MT of FA was estimated. Most of the fly ash which is produced is disposed as landfill, a practice which may affect the environment. The disposal of the large amount of fly ash has become a serious environmental problem. Since it has significant amounts of iron oxide (6–15%) and aluminium oxide (20–30%), it may find use in the extraction and production of aluminium and iron oxides, and subsequently in the recovery of aluminium and iron metals. The present investigation deals with separation of iron oxides first by magnetic separation from any CFA and followed by the leaching of the remaining fly ash after magnetic separation by using hydrochloric acid for the recovery of aluminium oxide from leach liquor with alkaline precipitation which will be beneficial economically and environmentally.

Some of the physical and chemical properties of fly ash are given below.

The physical properties of fly ash are reproduced from Ahmaruzzaman (2010). Fly ash is fine and powdery solid. Its particles are predominantly spherical in shape, either solid or hollow and mostly glassy (amorphous) in nature. The particle size distribution of most bituminous and sub-bituminous coal fly ash is generally similar to that of silt (≤0.075 mm or No. 200 sieve) and however, later it is generally slightly coarser than bituminous coal fly ash. The specific gravity of fly ash varies from 2.1 to 3.0, and specific surface area may range from 170 to 1000 m^2/kg. The color of fly ash can be gray to black, which depends on the unburned carbon content in the ash.

There are basically four types or ranks of coal, each vary in heating value, chemical composition, ash content and geological origin. Out of the four types of coal, which are anthracite, bituminous, subbituminous and lignite, in general only the last three are used in boiler. The chemical properties of fly ash produced are generally depending on the type of coal burnt and techniques used for handling and their storage.

On the basis of literature review, it is concluded that there are different techniques used for the recovery of aluminium and iron oxides from fly ash; they are (1) alkali leaching, in it only Al can be leached (Livingston et al., 1983; Gudyanga et al., 1992) (2) magnetic separation can be used to separate a part of iron that is magnetite (Russel and Zawadzki, 1963; Hurst and Styron, 1978; Brown, 1980; Dobbins and Murtha, 1983; Aldrich, 1984; Shcherban et al., 1995; Rao et al., 1999; Parkash et al., 2001; Sripriya et al., 2003; Shoumkova, 2011). However, Shcherban et al. (1995) also recovered alumina concentrate by hydro-alkaline treatment. (3) Desilication for only aluminium recovery (Shoumkova, 2011; Guanghui et al., 2010a, 2010b). Treatment of FA to form SiF_4 vapor and fluorides and fluorosilicate of Al and Fe, their separation and subsequently conversion of fluorides to oxides (Russ and Smith, 1985). (4) Fungal bioleaching for Al and Fe both (Siedel et al., 2001; Wu and Ting, 2006; Xu and Ting, 2009). (5) Leaching of Al and Fe with acid followed precipitation by alkali and dehydration of

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hydroxide. Leaching studies reported in literature are given in the next paragraph and (6) magnetic separation followed by leaching with mineral acid (Lisowyj, 1986).

Leaching of Al and Fe oxides from FA with sulphuric acid is reported by several workers (Gudyanga et al., 1992; Seidel et al., 1999; Blanco et al., 2005; Matjie et al., 2005). Gudyanga et al. (1992) used sintered pellets for leaching studies, however, few studies have been reported earlier on leaching of FA with hydrochloric acid (Ashworth et al., 1987, Berrya et al., 1987). Lisowyj (1986) carried out wet magnetic extraction followed by leaching with sulphuric acid. However, in the present work dry magnetic extraction of magnetite and maghematite from fly ash followed by its leaching with hydrochloric acid was carried out. Magnetic separation prior to leaching is beneficial as it reduces chemical required in leaching. In leaching with HCl, the Ti which is present in appreciable amount is not leached. Therefore, it reduces the need of separation of Ti from leachate. TiO_2 and SiO_2 are practically insoluble in hydrochloric acid and the leach liquor from such a treatment is expected to contain mainly iron and aluminium chlorides.

In the present study the effect of three parameters affecting the extraction of Al and Fe, (i) conc. of HCl (ii) HCl to solid ratio and (iii) leaching time, have been carried out. The following were the specific objectives

i) To study the separation of iron oxide by magnetic separator from fly ash.
ii) To carry out the leaching of FA with hydrochloric acid for extraction of aluminium and iron chlorides and to optimize the parameters such as acid concentration, acid to fly ash ratio and leaching time.

2. Materials and methods

2.1. Materials

2.1.1. Fly ash

Fly ash was procured from PANKI POWER HOUSE (Kanpur).

2.1.2. Chemicals

Hydrochloric acid: analytical grade (AR) HCl (Merck; India) having 35 wt% of acid, diluted to the required concentrations and ammonium hydroxide: laboratory reagent were used. For filtration Whatman filter paper no. 40 & 41 were used.

2.2. Methods

2.2.1. Experimental procedures

2.2.1.1. General. The general properties of the fly ash were determined (i) pH: to determine pH, water soluble 1:20 (fly ash–water) extract was prepared. The pH of water extract was determined by a calibrated pH meter. (ii) Specific gravity: it was determined by specific gravity bottle using water media (iii) particle size of fly ash: particle size of fly ash was determined using particle size analyzer. (iv) LOI: loss on ignition was determined as usual procedure.

2.2.1.2. Magnetic separation of iron oxide from fly ash. The separation of iron oxide from fly ash was carried out through STEARNS Magnetic Inc. (magnetic separator) made by MILWAUKEE WISC, U.S.A. The instrument was set at current: 0.2 A and voltage: 50 V. The magnetic force field was 0.14 to 0.16 T. 300 g fly ash sample was separated in 8 passes, after this further pass of tailing does not yield appreciable amount of concentrate. On each pass the magnetic material was further concentrated with hand magnet, and tailings were used in next pass.

2.2.1.3. Experimental setup and procedure for HCl leaching of the fly ash. The leaching of the fly ash with hydrochloric acid was done using a 500 ml round bottomed flask, fitted with water cooled condenser, a magnetic stirrer for agitation, a thermometer for recording the temperature of the flask and a heating mantle. Thirty gram sample of the fly ash powder (tailing after magnetic separation, dried fly ash screened to +100 μm size) and the hydrochloric acid of pre-required quantity taken in the flask, kept in the mantle and the heated to attain the desired temperature. After the required time of digestion the flask was allowed to cool at room temperature. Liquid–solid separation was achieved by decant washing followed by filtration using the Whatman no. 40 filter paper. The leached liquor is settled for some time and it is taken for analysis. The effect of various parameters such as concentration, quantity of acid, and leaching time was studied to determine the conditions that are optimal to the extraction.

2.2.1.4. Procedure for NH_4OH digestion with the leached liquor. The leached liquor, obtained by treating the fly ash with HCl, was filtered through a filter paper. The filter paper was washed three times to ensure complete removal of iron and aluminium chlorides. The leached liquor was makeup to 500 ml. 3 g of ammonium chloride was added to the solution and 2 ml of methyl red was added to ensure the color change. Then add 1:1 ratio of ammonium hydroxide which reacted with iron and aluminium chlorides to form respective hydroxides, these were precipitated and filtered through a Whatman no. 41 filter paper. The precipitate was taken out and roasted in muffle furnace at about 1100 °C for 45 min to form aluminium and iron oxides.

2.2.2. Analytical procedures

X-ray diffraction analysis was used for phase identification of Al_2O_3 and Fe_2O_3. Energy Dispersive X-ray (EDX) analysis was used to know the elemental composition of fly ash sample and the aluminium and iron oxide mixture sample.

The concentration of iron and aluminium in the different samples was analyzed by atomic absorption spectroscopy. Elico SL-173 double beam Atomic Absorption Spectrophotometer (AAS) was used for the determination of the concentration of iron and aluminium in the leached liquor. Iron was determined at the wavelength 248.3 nm and fuel was air/acetylene and the nature of the flame was oxidizing. Aluminium was determined at the wavelength 396.2 nm and fuel was nitrous/acetylene and the nature of the flame was slightly reducing.

2.3. Summary of step by step procedures

(i) The separation of iron oxide from fly ash was carried through STEARNS Magnetic Inc. (magnetic separator) made by MILWAUKEE WISC, U.S.A. (ii) From step 1, 30 g tailings of fly ash was taken and it was reacted with hydrochloric acid. (iii) Then the leached liquor was taken out and the precipitate was washed thrice in order to remove total amount of iron present in it. (iv) Then make up the leached liquor to 500 ml. (v) Aliquot of the samples were taken to determine aluminium and iron oxides by AAS. From this the total amount of iron and aluminium oxides present in the 500 ml make up solution. (vi) Aliquot of the solution was precipitated with ammonium hydroxide (1:1 ammonia solution and water) and then ignite in Muffle furnace (1100 °C) to get a mixture of aluminium and iron oxides; content of this mixture were analyzed by EDX.

Table 1
Chemical composition of fly ash procured from Panaki, Kanpur, India.

Constituents	Wt.% obtained from EDAX	Wt.% after inclusion of LOI
SiO_2	46.77	45.79
Al_2O_3	28.83	28.22
Fe_2O_3	12.71	12.44
TiO_2	4.74	4.64
CaO	1.65	1.62
V_2O_5	1.07	1.05
K_2O	4.23	4.14
LOI (loss on ignition)		2.1