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Characterization of Coal ash from a Captive Power plant for Potential End uses

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Abstract

Coal ash (fly ash and bottom ash) collected from a local captive power plant (Jamadoba Power Plant, Tisco, Dhanbad, India – production 10MW/day) was characterized in respect of their physico-chemical, morphological and mineralogical properties. Bulk densities, specific gravities, water holding capacities, pH were determined for as received, Fe enriched and Fe depleted fly ash and bottom ash. Mineralogical characteristics were obtained by carrying out XRD analysis (CoK_α and CuK_α) of all the above mentioned samples. Particle size analysis of all the samples was carried out using Laser based particle size analyzer (Fritsch). Morphological properties were assessed using Scanning Electron Microscope. FTIR spectra were determined for all the samples including the feed coal. In addition to the various instrumental analysis carried out for the ash samples, sieve analysis and flotation of selected size fraction (+212µm) were carried out. Analysis of the results obtained yielded some important information. The bottom ash samples were large fused masses (Fig.1) of irregular shape and much larger than those of fly ash samples. Mineralogical analysis revealed the presence of quartz as the principal phase. Additionally strong intensity bands were observed for hematite and magnetite in case of Fe-enriched fly ash fractions. Flotation experiments revealed that one particular fraction (+212 µm) has high carbon content, which indicates that this fraction can be enriched to a fraction which has the potential for reuse as domestic fuel. Other fractions having lower carbon content may have other utilization channels like road stabilization, brick making, in the making of cement etc.

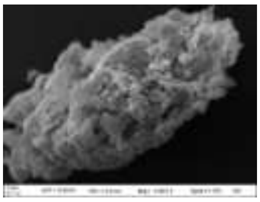

Fig. (a)Bottom ash

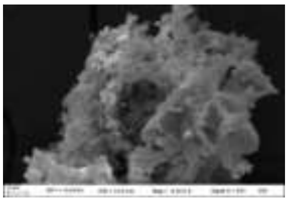

Fig.(b)Fly ash

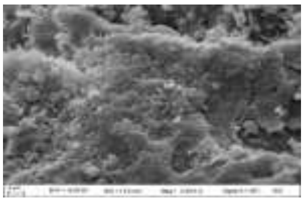

Fig.(c)Fe enriched ash

Keywords: Fly ash, Characterization, FBC coal ash, SEM, FTIR, XRD, flotation, End uses

INTRODUCTION

With the boom in population and industrial growth, the need for power has increased manifold. The electricity sector in India had an installed capacity of 199.87 Gigawatt (GW) as of March 2012, the world's fifth largest. Captive power plants generate an additional 31.5 GW. The power requirement has shot up exponentially and will continue to do so.

Coal based thermal power plants account to 55 % of the total power generated in India. In India, coal used for thermal power plants is usually high ash ROM coal as well as coal washery reject and middling coals. These coals have ash content as high as 65%. Hence the consumption of these coals generates huge amount of ash. The ash generated by these power plants is of two categories namely the fly ash and the bottom ash.

These fly ash particles are very fine and light. They do not settle on their own. They remain naturally suspended in air. These fine particles, due to their known hazards, cannot be let away by mixing in the air. Electrostatic Precipitators are used to collect a majority of these particles, some portion which do not get collected escape.

Some country rocks are also mined along with the coal when the mining is done at the interface of the coal seam and the country rock. The beneficiation of coal leads to these country rock reporting in the reject fraction of coal. These rocks, which are essentially incombustible material, when burnt leads to relatively coarse ash, known as bottom ash. Some of the bottom ash in an FBC power plant is also a result of limestone/sand used to control the bed temperature of the fluidized bed combustor, which is a normal practice in fluidized bed combustor.

A major challenge emerging is the effective handling and utilization of fly ash and bottom ash generated by these power plants. The understanding of the characteristics of ash particles and the transformation of coal minerals during combustion are essential to the development of value-added ash utilization technologies.

The present work involves the characterization of fly ash, bottom ash and feed coal collected from Jamadoba fluidized bed power plant for study of their potential for other application.

MATERIALS AND METHODS

100 Kg of feed coal, fly ash and bottom ash each were collected from the Jamadoba Fluidized bed combustion plant for the purpose of carrying out the experimental work.

The experiments conducted on FBC feed coal includes; sieve analysis, FTIR Spectroscopic analysis, Specific Gravity, Mineralogical analysis (XRD, Optical Microscopy), Morphological analysis (SEM), Ash Analysis and Calorific value determination.

The fly ash was subjected to various investigations which includes; Laser based Particle size Analysis, Sieve Analysis, FTIR Spectroscopic analysis, Bulk density, Specific Gravity, Mineralogical analysis (XRD), Morphological analysis (SEM), Magnetic portion removal using Davis Tube, Ash analysis of as received and different size fraction obtained during sieving, Determination of pH, Water Holding capacity, Conductivity, TDS and salt conc., Froth flotation of +212 µm size fraction and Calorific value determination.

The bottom ash samples were analysed for Sieve Analysis, FTIR Spectroscopic analysis, Bulk density, Specific Gravity, Mineralogical analysis(XRD), Morphological analysis (SEM), Ash analysis of different size fractions obtained after sieving, Determination of pH, Water Holding capacity, Conductivity, TDS and Salt concentration.

The experiments conducted on Magnetic enriched fly ash were Laser based Particle size Analysis, FTIR Spectroscopic analysis, Specific Gravity, Mineralogical analysis (XRD), Morphological analysis (SEM), Determination of pH, Water Holding capacity, Conductivity, TDS and salt concentration.

Finally the Non-magnetic fraction of fly ash were tested for FTIR Spectroscopic analysis, Bulk density, Specific Gravity, Mineralogical analysis (XRD), Morphological analysis (SEM), Determination of pH, Water Holding capacity, Conductivity, TDS and salt concentration.

The sieve analysis of fly ash was done after sampling and subsequently drying the sample in oven for two hours. This ensured that the sample was dry. Similarly the size analysis of bottom was done using the sieves of 425 µm, 600 µm, 710 µm, 1.4 mm and 1.7 mm, while the size analysis of coal was done with sieves of size 3 mesh, 4 mesh (BSS), 4 mm, 3.35 mm, 7 mesh (BSS), 2.36 mesh (BSS), 1.7 mm, 1.4 mm and 1mm.

Loss on ignition was determined by taking 1 gram of sample (pulverized to -75 µm) in a silica dish as per standard norms. Particle size analysis was carried out using LASER based model Fritsch Particle Sizer Analysette 22. Helium Neon Laser is used to obtain a series of diffraction pattern. The fundamental size distribution derived by this technique is volume based.

SEM analysis was done using scanning electron microscope model s – 440, LEO Electron Microscopy equipped with an Oxford link –Isis Energy dispersive X ray analyser (EDX). Mineralogical compositions were determined on X ray diffractometer Model PW – 1710 with Cu Kα radiation. The working voltage and current is 40 kV and 20 mA respectively. Scanning speed is 0.05 per °2θ per sec or 3° per min.

FTIR spectra of the samples were recorded on Perkin Elmer Model – System 2000 using KBr pellet method.

The fly ash sample of size 212 µm – 600 µm were subjected flotation experiment for the recovery of combustibles/carbon from fly ash. 310 g sample, thus obtained, was mixed with 2.7 liters of water. Diesel oil, used as collector, was added (dosage 0.8 kg/ton) and the sample was conditioned for 5 minutes. Pine oil, used as frother, was added, (dosage0.4 kg/ ton) and the sample was conditioned for 5 minutes. The air valve was opened and the samples were collected at regular intervals. The concentrates were kept in air oven and dried. The tailing was vacuum filtered and then oven dried. Subsequently Ash analysis and calorific value of the obtained fractions were determined. Water holding capacity of the samples was also calculated by keeping the sample in standard perforated cylinder over a filter paper. The cylinder was kept in water for four hours. The cylinder was then taken out and kept for one half an hour. The weight of the absorbed water was calculated per unit mass of the solid.

Davis tube experiment was carried out using standard Davis tube apparatus at the current value of 1.85 A, Magnetic Intensity 1200 Gauss.

The pH, TDS, salt concentration and electrical conductivity were determined by adding 80 g of material in 320 ml distilled water, keeping the samples in air tight containers for four hours and then measuring the properties of the solution after decantation.

RESULTS AND DISCUSSION

An examination of fig. 21 and fig. 26 reveals that the feed coal particles are large and irregularly shaped. It appears (fig 15) extrinsic mineral matters are present in these particles. Strips of such mineral matters could be observed. Also the coal particles appear dark whereas the mineral matters appear brighter due to higher atomic weight. As received fly ash particles are found to be irregularly shaped (Fig. 24 and Fig. 29). Scanning electron micrograph of a fly ash particle (as received) at higher magnification (6.38 KX) reveals that after combustion the coal particles have been converted to fused alumino silicates (fig. 19) Normally the fly ash particles are small regular spherical particles. From a critical examination of the mechanism of ash formation by coal combustion, the effect of the above mentioned factors on the shape and size of fly ash particles can be understood. At the combustion chamber the coal particles undergo pyrolysis. This is stage I. As a result of this char formation takes place. Further combustion of char takes place at this stage. The residual mineral matter now undergo coalescence and result in the formation of ash particles of medium size (0.2 -10 μm). The largest ash particles (10 – 90 μm) come from the transformed extrinsic mineral particles. Coal quality is an important factor. If the carbon present in coal is less oxydised and contains more of aromatic carbon then it will be readily converted to ash. Oxydised coal is less prone to condensation since the carbonyl/carboxyl groups present interacts with oxygen and forms hydro-peroxides, thus slowing down the oxygen uptake.[1] The nature of the mineral matters present also matters. High content of quarts particles also affects the shape and size of fly ash particles. Since quartz has very high melting point, Ash fusion temperature also increases with the increase in the content of quartz. As a result good melt may not be obtained. The form of silica and calcium plays very important role in their vaporization. Silica in the form of quartz is least reactive and in the form of illite, it will react readily. Calcium and magnesium present in Illite react rapidly but not so easily if it is present in carbonates (calcite and anchorite). Organically associated calcium readily reacts with alumina silicates and quartz within the coal matrix to form lower melting point phases $CaSiO_3$. Large veins of calcite leads to increased viscosity of the liquid phases. Combustion environment also plays an important role in the formation of ash particles in this respect, gas temperatures and excel air play the lead role. Particle temperature and excess oxygen are directly proportional. The greater the concentration of oxygen, higher will be the particle temperature. From the above discussion the reason for the fly ash particles being non spheroidal and irregular is quite clear. The coal particles are very large. On grinding such particles, the large sized coal particles are broken and mineral matters are exposed. The original feed coal cannot be converted to a uniform melt until and unless very high particle temperature is obtained which also depends upon the concentration of excess oxygen. Scanning electron microscopic analysis of non-magnetic fraction of fly ash reveals that there is distinct morphological difference between the coal particles and those of non-magnetic fraction. A perusal of fig. 15 and fig. 18 clearly shows this difference. Fig. 18 indicates that during combustion the coal particles undergo volatilization, char burning and mineral matter fusion. It is observed that agglomeration of the fused mineral matters has taken place. Scanning electron microscopic analysis of non-magnetic fraction of fly ash reveals that there is distinct morphological difference between the coal particles and those of non-magnetic fraction. A perusal of fig. 18 and fig. 15 clearly shows this difference. Fig. 18 indicates that during combustion the coal particles undergo volatilization, char burning and mineral matter fusion. It is observed that agglomeration of the fused mineral matters has taken place. Fe enriched fraction seems to be much more compact and consolidated.

FTIR analysis of the various samples gave useful information. Fig 5 – which represents FTIR spectrum of coal samples reveals various features of the coal samples. It is observed that both associated and free OH peaks are present (3693 cm^{-1} – 3434 cm^{-1}). The peak at 3693.57 cm^{-1} and 3620 cm^{-1} represent M –OH stretching, where M may be elements like M-Ca, M-Si etc. The broad peak at 3434 cm^{-1} is due to OH stretching of associated water. OH bending vibrations are observed in the region 1650 cm^{-1} - 1630 cm^{-1}. The coal sample contains substantial amount of aliphatic carbon. This is evidenced by the appearance of strong peak at 2924 cm^{-1}(Asymmetric CH_2 stretching) and 2854 cm^{-1}(Symmetric CH_2 Stretching).[2]

However the coal sample is partially oxidised which is evident by the sharp peak at 1742 cm^{-1} FIG. 5 (due to C=O). Considering high intensity peaks at 2924 cm^{-1}, 2854 cm^{-1} for aliphatic CH_2 stretching and presence of a peak at 1459 cm^{-1} (aliphatic CH_2 bending) the peak at 1742 cm^{-1} is less intense. Therefore it may be concluded that the coal sample is partially oxydised but it still has significant amount of aliphatic carbon. Various mineral peaks (1033 cm^{-1} Si – OH bending, 794 cm^{-1} Al/Si – O bending, and the peaks at 540 and 472 cm^{-1} Si – O –Si bending) are present.

FTIR spectrum of bottom ash Fig. 6 indicates that it consists of various types of mineral matters. The peak at 3468 cm^{-1} is due to associated H_2O.The peak in the region of 1634 cm^{-1} to1599 is due to H_2O

bending. Various mineral oxide peaks are observed (1083 cm^{-1} Si – OH bending -transformed from 1033 cm^{-1} in coal; 791 cm^{-1} Al/Si - O bending, transformed from 794 cm^{-1} in coal; and 461 cm^{-1} Si – O – Si , transformed from 472 cm^{-1} in coal). FTIR spectrum of the associated fly ash shows various M – OH stretching (3695 cm^{-1} – 3526 cm^{-1}). A peak at 3120 cm^{-1} is due to associated OH. One interesting feature of the FTIR spectrum is that in addition to the mineral clay/ Kaolinite etc the original mineral oxide peak at 1032 cm^{-1} which was present in the coal sample is also observed in this spectrum which clearly indicates that complete transformation of mineral matter has taken place.

Significant differences are observed between the spectrum of the magnetic and non-magnetic components of fly ash, whereas both show peaks for aliphatic CH_2 (2925 cm^{-1} and 2854 cm^{-1}). The non-magnetic Portion additionally has peaks 1544 cm^{-1} and 398 cm^{-1} which corresponds to various bending vibrations of the aliphatic CH_2 and CH_3. Both the fractions have high intensity peaks at 1085 cm^{-1}, 794 cm^{-1}, 469 cm^{-1} which correspond to the various mineral oxide bands as stated earlier.

From the XRD graph of coal (FIG.10), the various minerals identified are Quartz, Mullite, Magnetite, Hematite and Anatase (TiO_2). The minerals present in bottom ash are Hematite, Quartz, Anatase and Albite($NaAlSi_3O_8$). The minerals present in fly ash are quartz hematite and magnetite. The minerals present in magnetically enriched fraction contain a strong peak for magnetite. Quartz and Anatase have also been identified. Quartz and hematite are found principally in Fe depleted fly ash. [3]

Mullite present in coal completely vanishes in the end products. Anatase (TiO_2), which does not undergo any chemical transformation mostly ends up in bottom ash. Albite ($NaAlSi_3O_8$) is only found in bottom ash. This may be due to the reaction between Halite (NaCl) and the alumino silicates present [4]. It seems that most of the minerals which undergo chemical transformation are mostly on the surface of the coal particles and they subsequently report to the fly ash, while those who did not undergo chemical transformation mostly report to the bottom ash. As the bottom ash particles are coarse, it is likely that most of the particles therein do not get a good environment for chemical transformation (due to the low specific surface area of coarser particles). This must due to the fact that proper contact might not have been established between the reacting phases. The presence of a higher quantity of Anatase in bottom ash while its absence in fly ash may also be due to the fact that these particles do not undergo any chemical change and have a lower probability to be elutriated due to their high molecular mass.

Another inference that can be made is that the magnetic particles are not well liberated, as the magnetically enriched fraction contains non-magnetic materials. But the magnetic particles are free from carbonaceous and charred material (Distinct morphological difference is observed between the fly ash and the magnetic particles). Hence it can be inferred that Fe estimation cannot solely be done by magnetic enrichment and the analysis of this fraction. This fact can be substantiated by the fact that the LOI of the magnetically enriched fraction was found to be 11.2 % and also, the FTIR result of the magnetically enriched fraction suggesting the presence of aliphatic CH_2 and CH_3.

Ash analysis reveals that the sized fractions lower than 45 µm have a very poor value of LOI. This means that the mineral matter is concentrated in the smaller sizes. The smaller carbon particles are easily burnt probably due to high surface area per unit mass. Hence the LOI value in the smaller fraction is low. The bottom ash particles have a very low LOI value. Since the bottom ash particles have a higher residence time in the furnace than the fly ash particles, the bottom ash particles are likely to lose more carbon and this results in a lower LOI value for bottom ash. The finer and the larger size fraction of the bottom ash have a relatively higher LOI than the other fractions. This may be because some amount of fly ash reports to the bottom ash section. As the fly ash particles have higher carbon content specially in its coarse range, this justifies the fine bottom ash particles having high LOI. The coarse fly ash fraction has a high carbon content possibly because the bigger particles have a poor specific surface area.

Optical microscopy of the coal sample revealed the presence of pyrite in a reasonably good quantity. The pyrite seems get transformed to magnetite and hematite. This is the reason of good quantity of magnetic particles in the fly ash. Since the pyrite is finely disseminated and undergoes a chemical reaction, the magnetite had hematite formed by this process[5] is likely to be found in fly ash.

The flotation and calorific value of the fly ash sample reveal that the fly ash sample could be beneficiated to a level which has a good calorific value. The overall recovery of the combustible material is also acceptable. The fly ash fraction obtained after sieving at 212 micron were around eight percent. The yield in hundred seconds is fifty percent (of the feed value) at a carbon grade of 49.95 percent. The calorific value of the concentrates is higher that the feed coal to the FBC. This is quite encouraging. Davis tube experiment revealed that the 7.4 % particles could be removed by magnetic separation.

Conclusion

From the forgoing discussion the following conclusions can be made (a) Fly ash and bottom ash collected from the captive power plant are in general irregular in shape and appears to be fused masses. (b) The Predominant mineral phase is quartz and some Fe bearing phase like hematite and magnetite. (c) Particle size analysis reveals that the particles are of larger size (D_{80p} μm) (d) The combustion is incomplete leading to the formation of large irregularly sized particles (e) Some fractions have a high carbon content and may be beneficiated and used as fuel (f) In general the fly ash may be used for pavement making, land filling etc.

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Table 1: Optical microscopy of feed coal

Mineral / Macerals	Volume %
Vitrinite	19.69
Liptinite	Traces
Inertinite	26.06
Clay	49.39
Quartz	2.72
Pyrite	2.14

Table 2: Specific gravity of feed coal, fly ash and bottom ash

Material	Specific gravity	Bulk Density, g/ml
Coal	1.87	-
Fly ash	2.049	0.635
Fe enriched fly ash	2.769	-
Fe depleted fly ash	1.907	-
Completely burnt ash	2.45	-
Bottom ash	2.636	1.01

Table 3: LOI of different size fraction of bottom ash and fly ash

Bottom ash		Fly ash	
Size (mm)	LOI %	Size (µm)	LOI %
+1.7	3.7	+212	27.04
+1.4	2.07	+125	24.58
+1	2.1	+90	23.26
+0.71	1.31	+63	22.02
+0.6	1.06	+45	18.4
+1.7	3.7	+38	8.7
+0.425	1.39	-38	8.5
-0.425	3.04		

Table 4: Physico chemical properties of different ash

Ash → Properties ↓	Bottom Ash	Fly Ash	Fe Enriched Fly ash	Fe Depleted Fly ash
pH	7.65	7.99	7.9	8.04
Salt Concentration	204 ppm	221	191	229
Water holding capacity (water/solid)	0.4758	0.9824	1.101	0.9956
Conductivity (µS)	377	387	343	410
TDS	263	280	429	292

Table 5:Flotation results

Time	(Yield) Wt %	Carbon %
50	38.48	52.60
100	11.47	46.14
150	10.73	44.89
200	8.64	43.90
250	1.97	32.68
300	5.91	39.20
Tailings	22.80	17.40

Table 6:Calorific values

Material	Calorific value (cal/g)
+212 micron Fly ash	2609.0
Cleans (50 s – flotation)	3529.4
Cleans (100 s – flotation)	3208.7
Tailings (Flotation)	1137.2
FBC feed coal	3035.0
Fly ash (as received)	1071.8

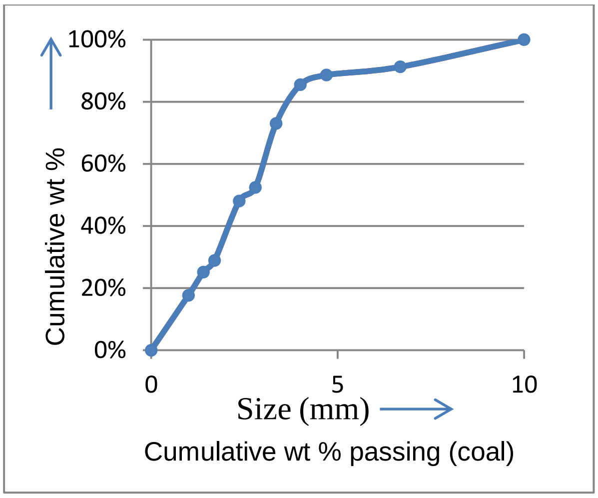

Fig. 1 Size distribution – Coal

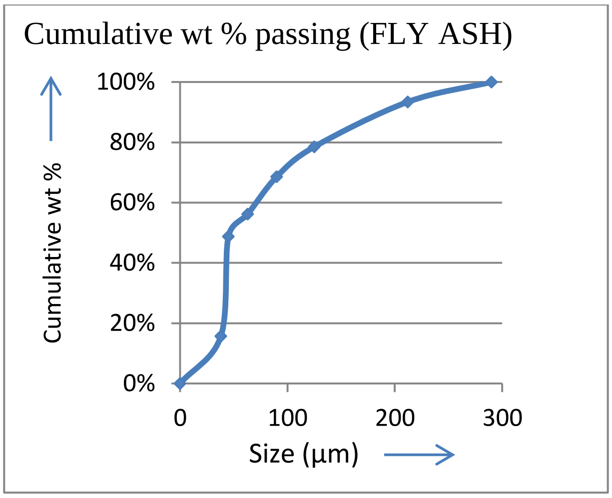

Fig. 2 Size distribution – Fly ash

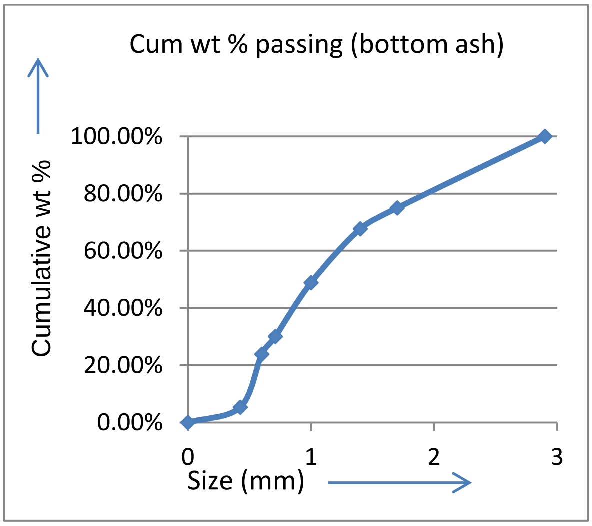

Fig. 3 Size distribution – Bottom ash

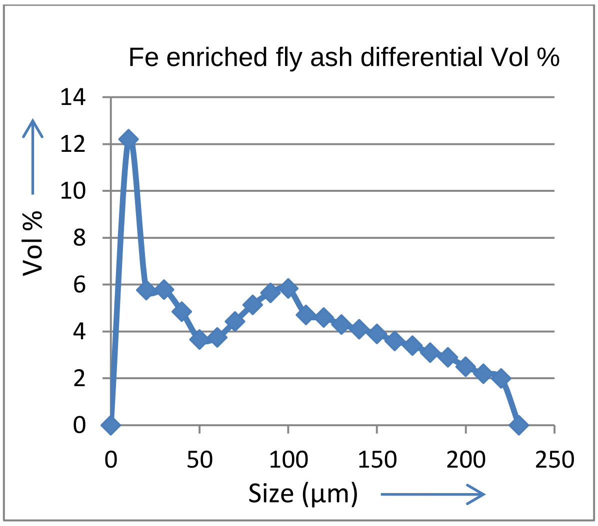

Fig. 4 Particle size analysis – Fe enriched fly ash

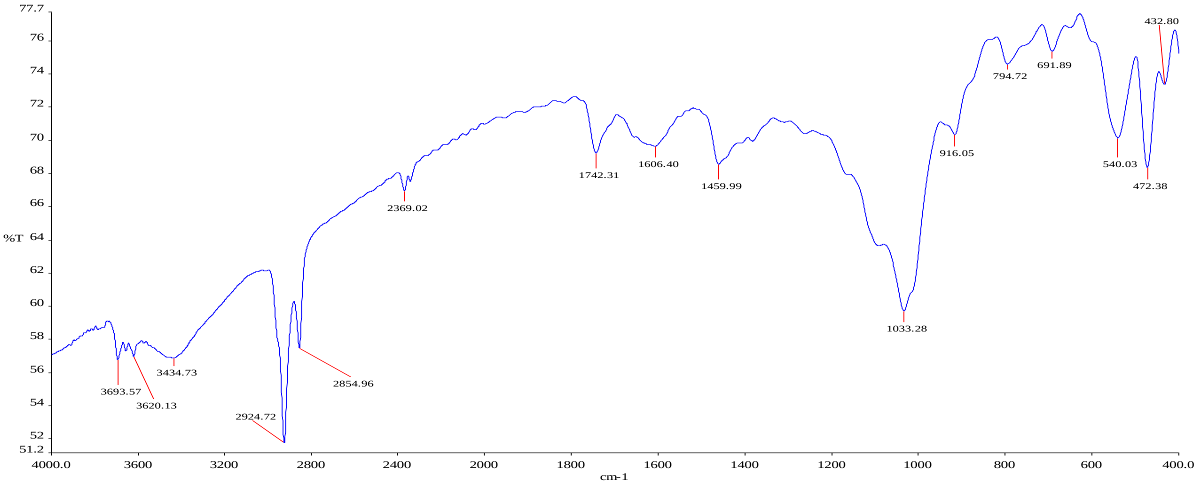

Fig. 5 FTIR spectrum of coal sample

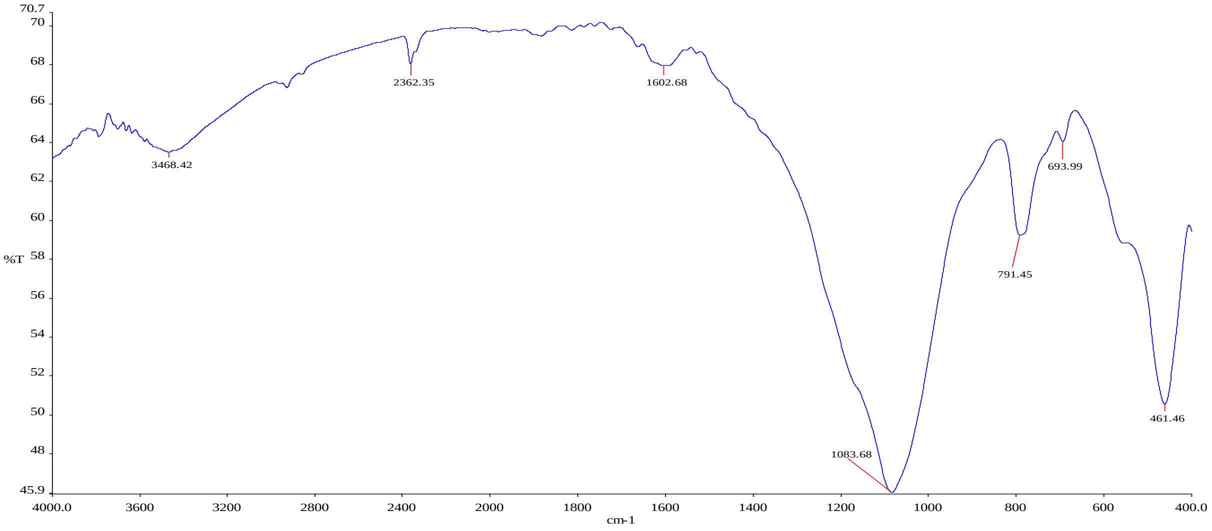

Fig. 6 – FTIR spectrum of Bottom ash sample

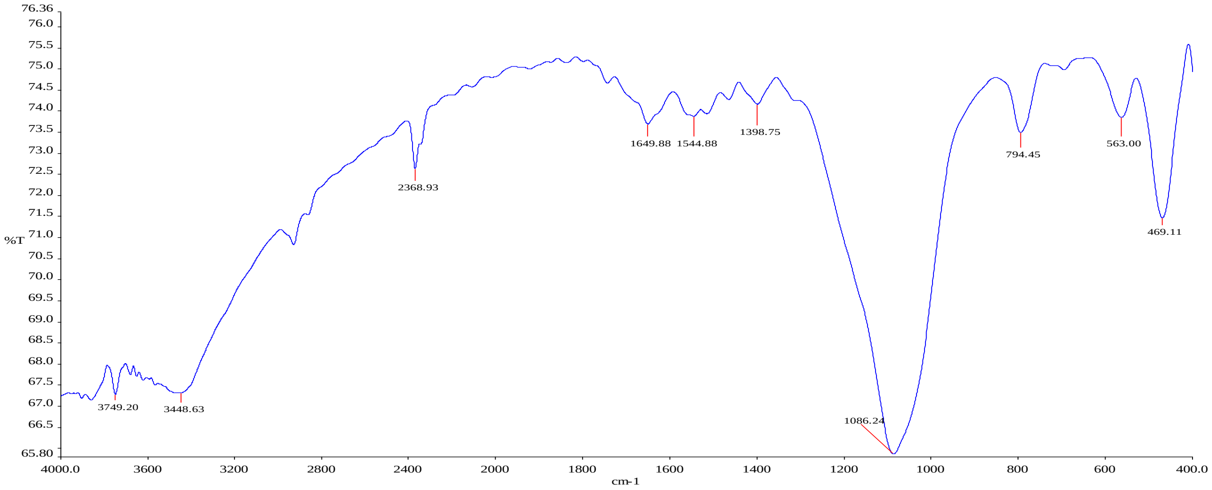

Fig. 7 FTIR spectrum of Fly ash sample

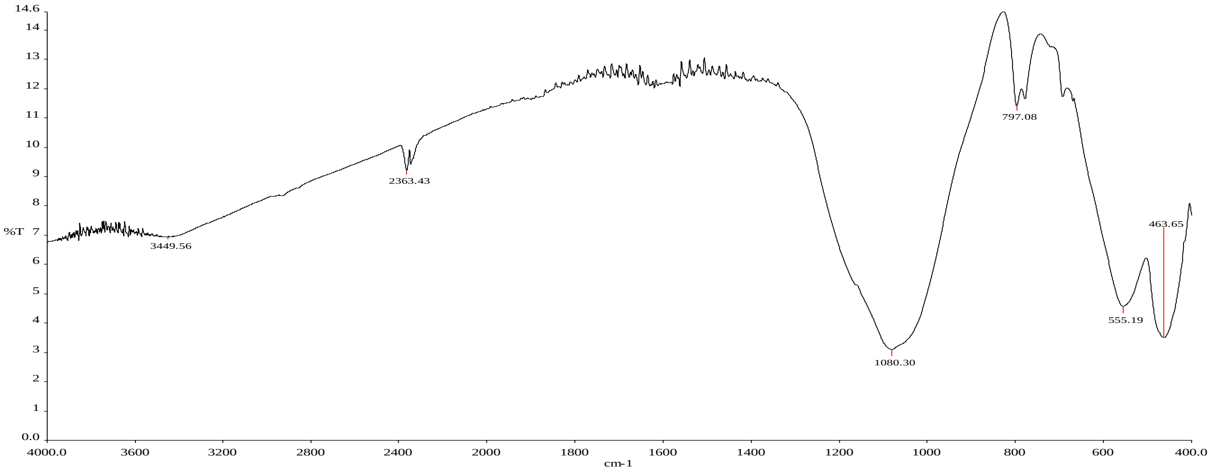

Fig. 8 FTIR spectrum of Fe enriched fly ash

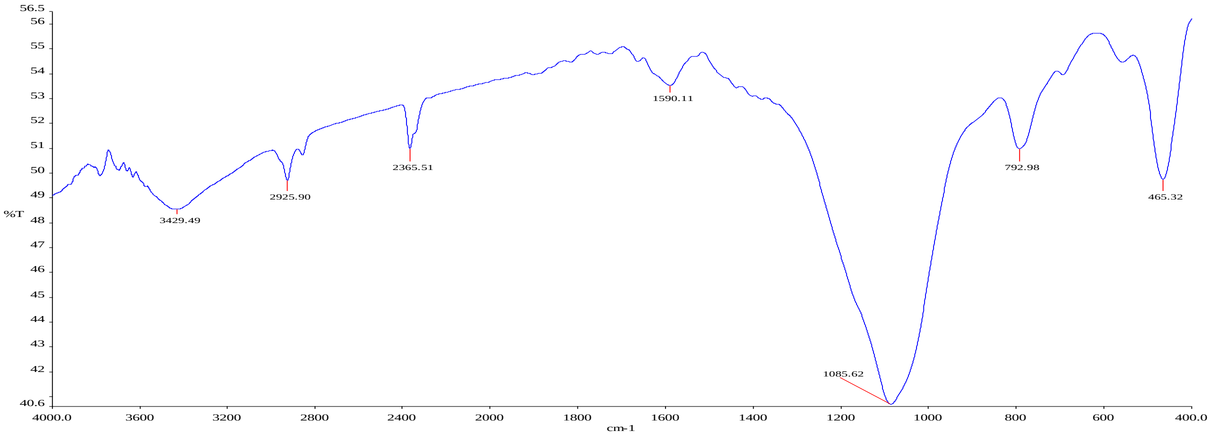

Fig. 9 – FTIR spectrum of Fe depleted fly ash

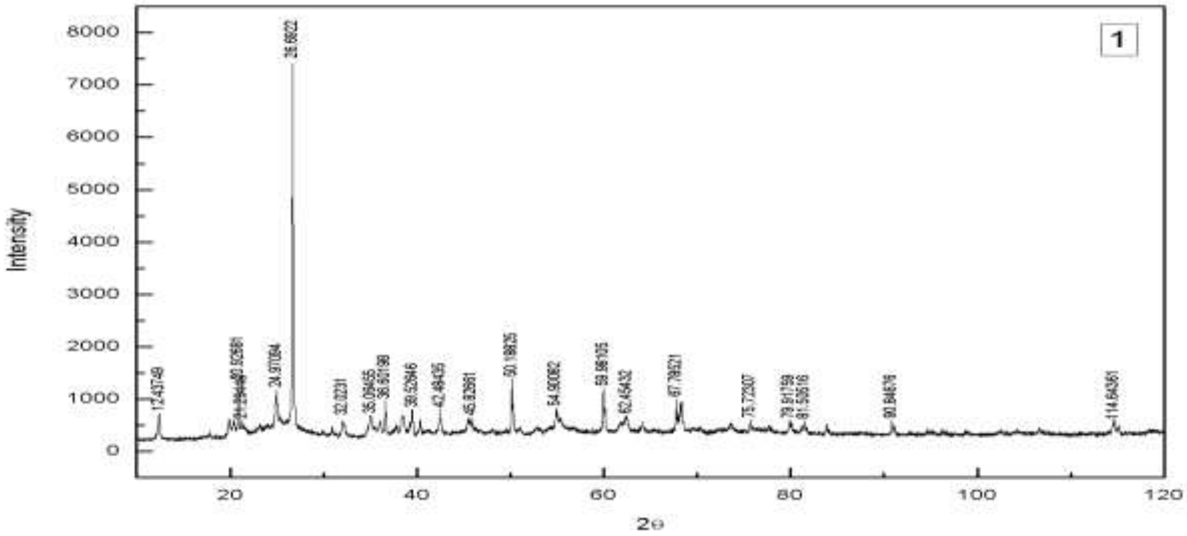

Fig. 10 X Ray diffractogram of coal sample

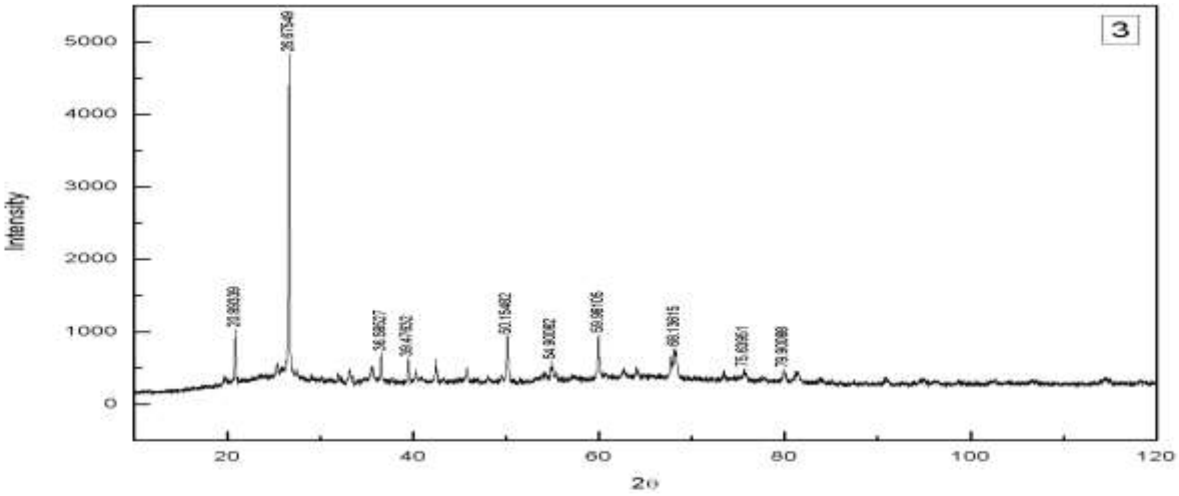

Fig. 11 X Ray diffractogram of Fly ash sample

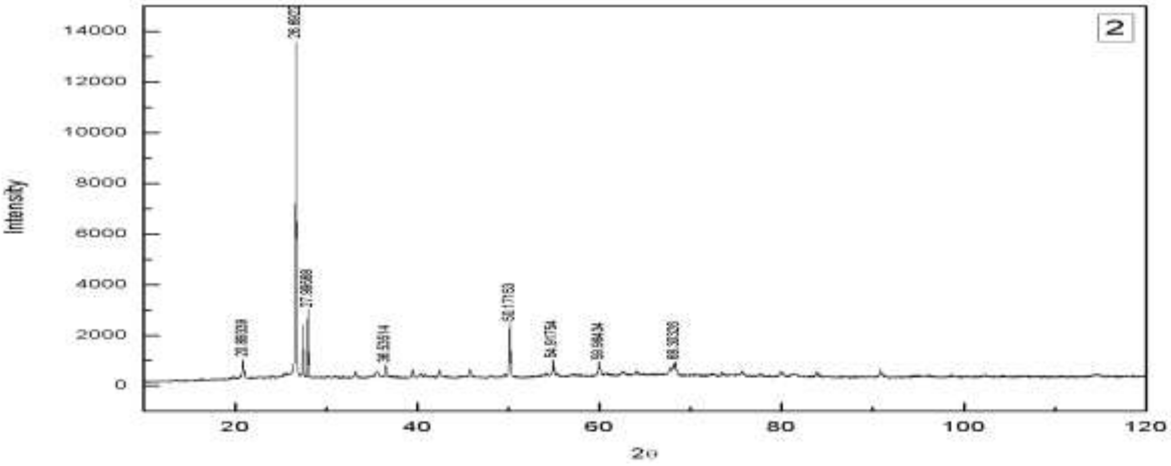

Fig. 12 X Ray diffractogram of Bottom ash sample

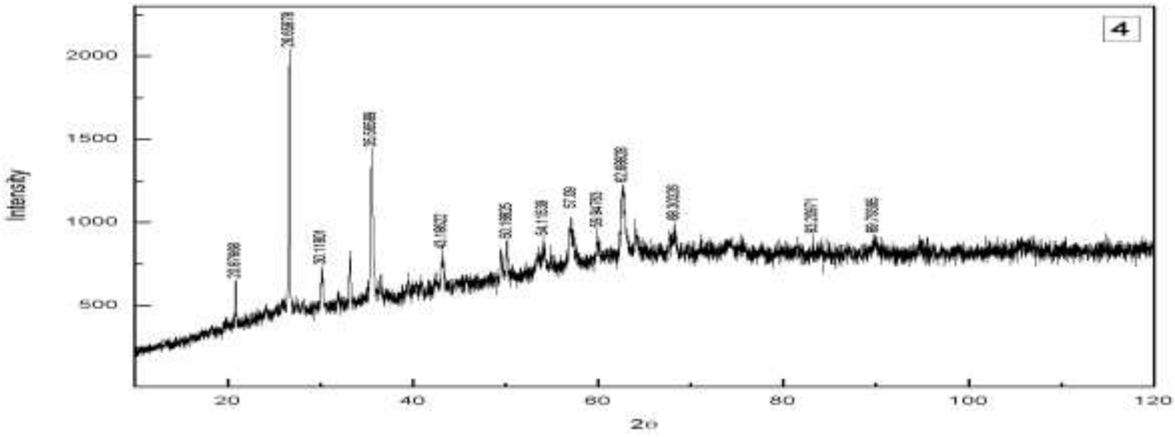

Fig. 13 X Ray diffractogram of Fe enriched fly ash sample

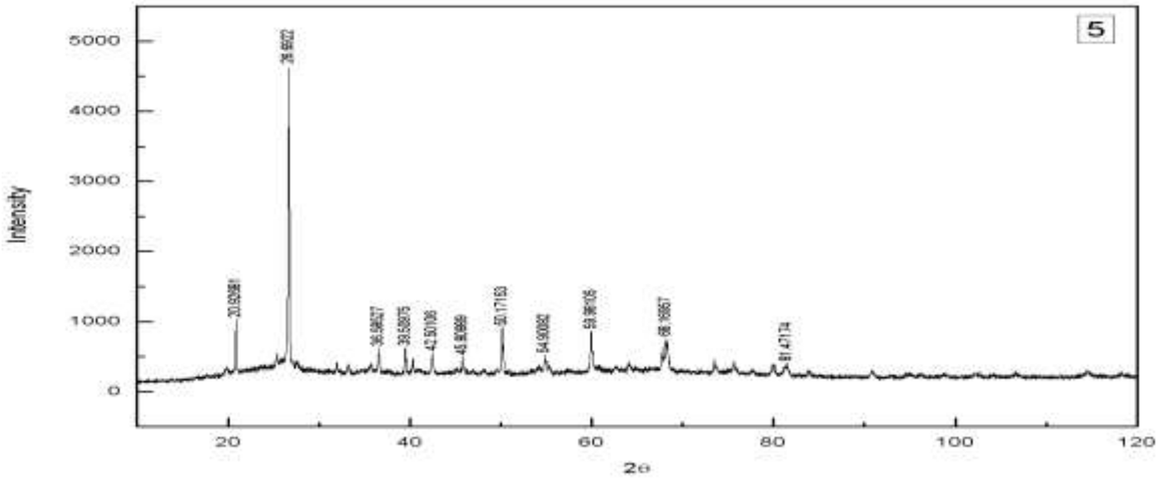

Fig. 14 X Ray diffractogram of Fe depleted fly ash

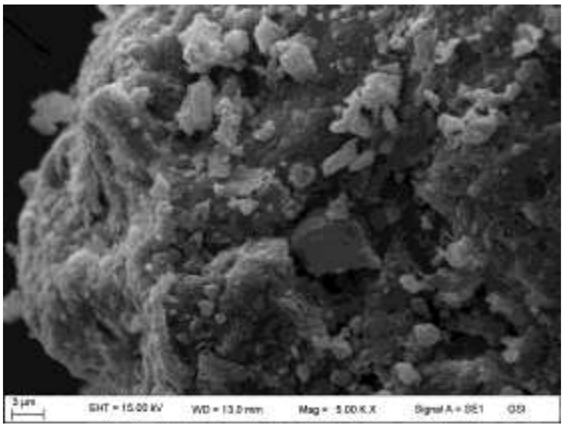

Fig. 15 – SEM image of Coal at 5 KX

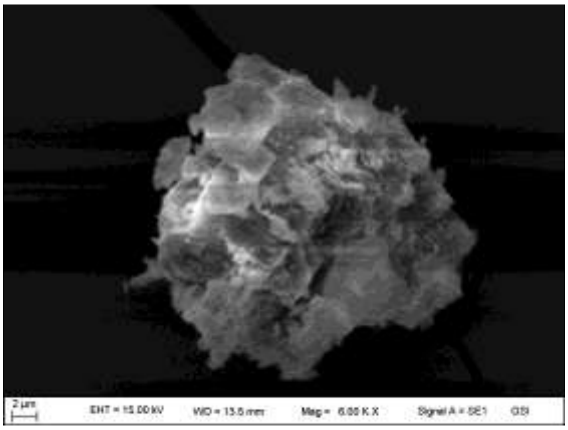

Fig. 16 – SEM image of Bottom Ash at 6 K X

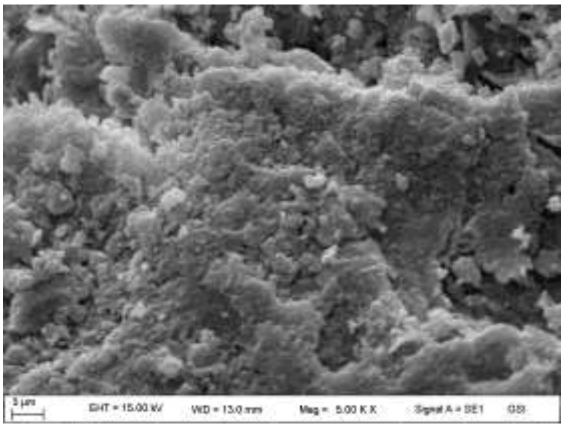

Fig. 17-SEM image Fe enriched Fly ash 5 KX

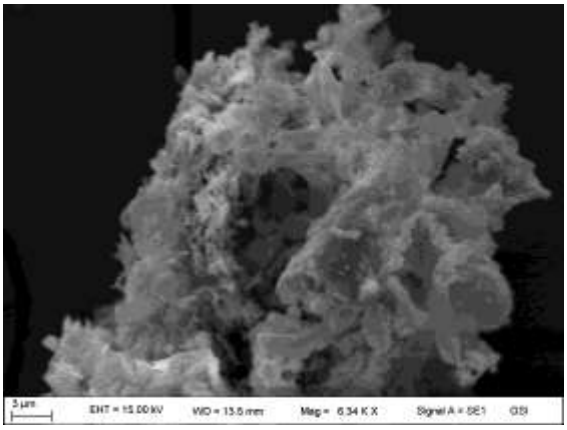

Fig. 18 - SEM image Fe depleted fly ash 6.34 K X

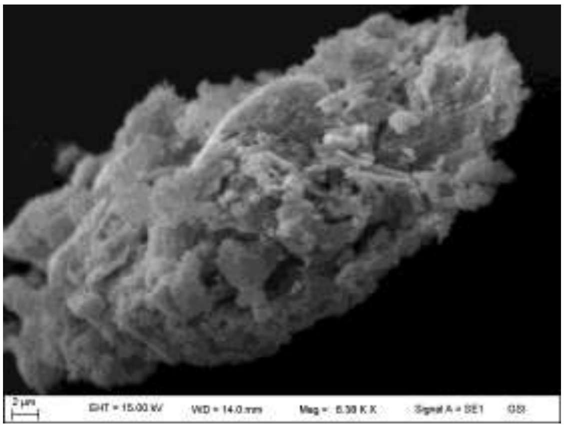

Fig. 19 - SEM image of Fly ash 6.38 K X

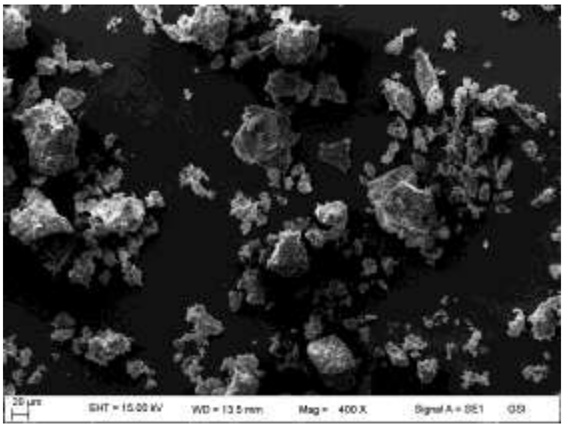

Fig. 20 - SEM image of Bottom ash 400 X

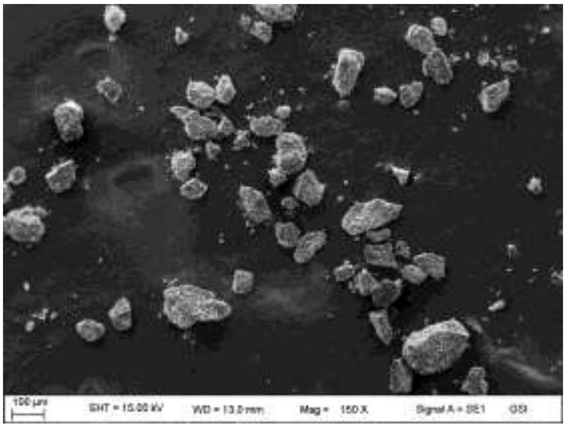

Fig. 21 - SEM image of Coal 150 X

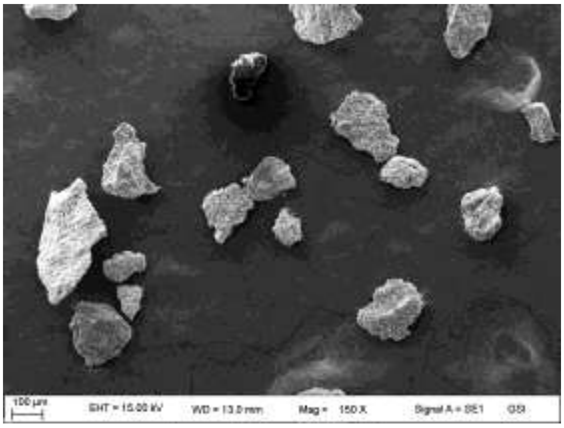

Fig. 22 - SEM image of Fe enriched fly ash 150 X

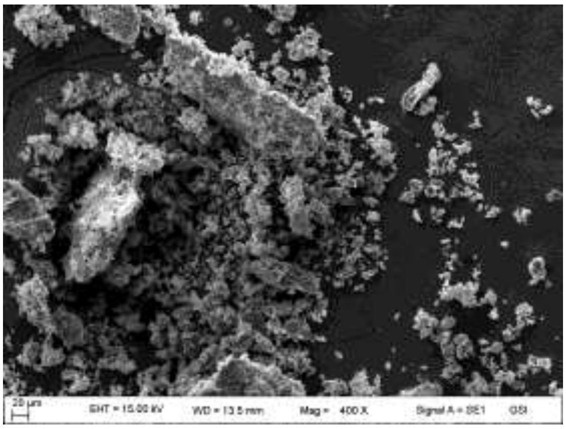

Fig. 23 - SEM image of Fe depleted Fly ash 400 X

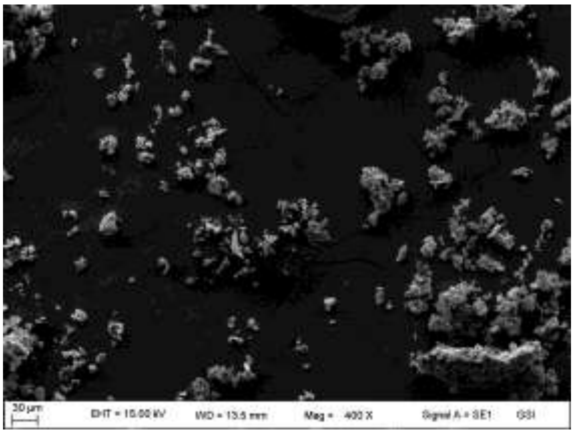

Fig. 24 - SEM image of Fly ash 400 X

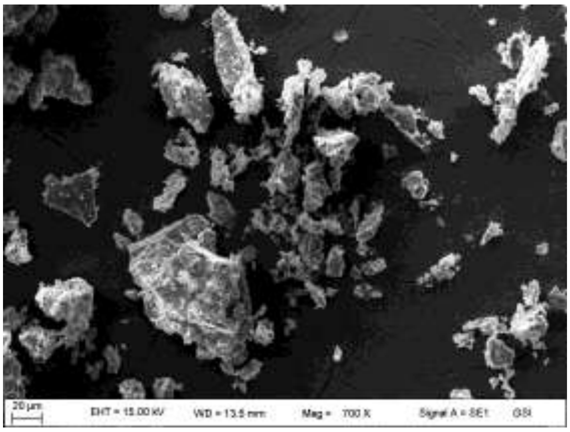

Fig. 25 - SEM image of Bottom ash 700 X

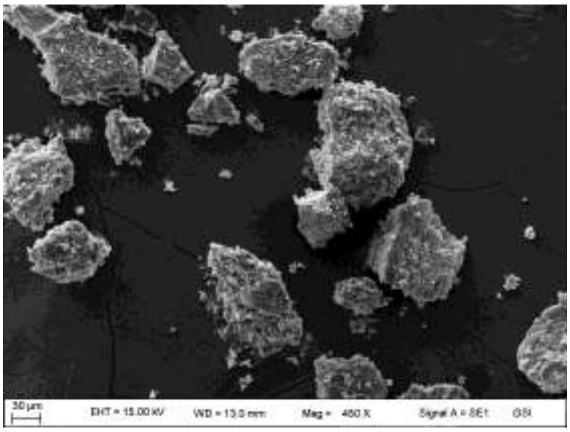

Fig. 26 - SEM image of Coal 450 X

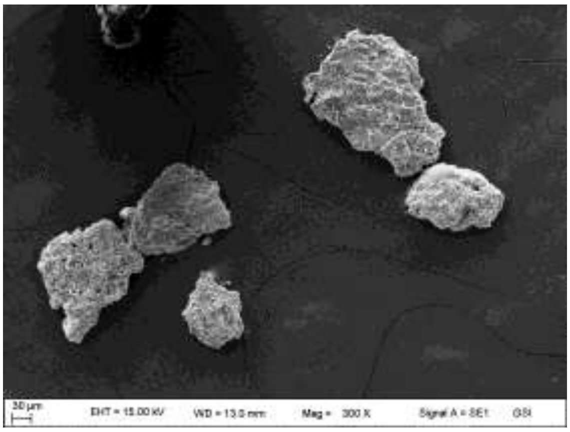

Fig. 27 SEM image of Fe enriched Fly ash 300X

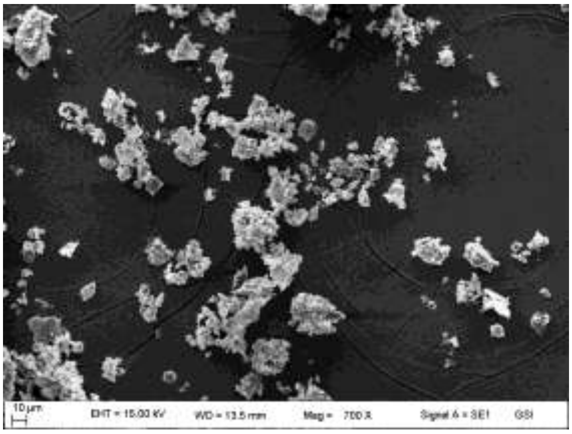

Fig. 28 - SEM image of Fe depleted FA 700 X

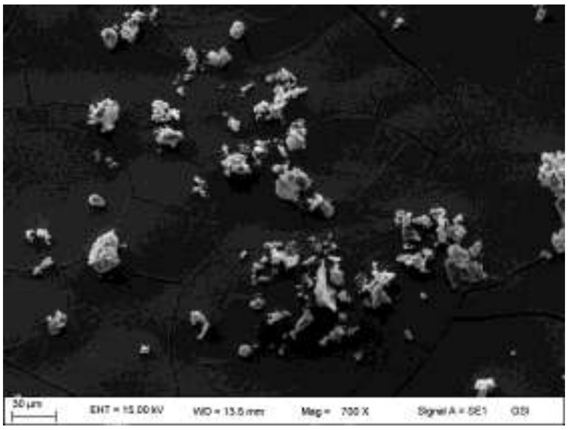

Fig. 29 - SEM image of fly ash at 700 X