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Physical Characteristics Of Fly Ashes From Three Thermal Power Plants In West Bengal, India: A Comparative Study

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Abstract: A comparative analysis on the physical nature of the fly ashes, obtained from three thermal power plants, situated in West Bengal, India is presented in this work. The particle size of the fly ash samples are estimated, and their morphology and compositional analysis are made. These samples are crystalline, and the major components are mullite and quartz. Quantitative magnetic characterizations of these fly ash samples were carried out too. Precisely, this report draws a comparison based on experimental data as regards physical aspects of the fly ash samples consisting of coarse, fine and ultrafine magnetic particulate materials (PMs) and provides an in-depth analysis.
Keywords: Fly Ash; Thermal Power Plant; Characterization; Particulate Matter.

Introduction

It is indeed inevitable that many countries depend on coal for electrical power generation[1], although there have been continuous efforts for exploring its viable alternatives across globe. As reported, combustion of coal in thermal power plants gives out potentially harmful by-products such as polycyclic aromatic hydrocarbons, oxides of sulphur and nitrogen and fly ash[2-4], although demineralization and desulphurization of coal are generally performed prior to use in thermal power plants. Fly ash of thermal power plants using coal is a fused residue of clay minerals present in it. These clay minerals in coal powder transform into a variety of fused fine particles as ultrafine (<100 nm) particulate matter (PM) with different minerals[5-7]. The primary constituents of fly ashes are SiO_2, Al_2O_3, Fe_2O_3, Fe_3O_4, TiO_2 and CaO. Besides, the fly ash may contain a small quantity of unburnt carbon with minute percentage of the oxides of Mg, Cr, Na, and K[8-13] and several potential toxic heavy metal elements like Pb, Zn, Cd, Ni, As and Co, which would pollute soils, surface water and ground water[14-16]. So, these are considered to be serious pollutants threatening the environment and thus the eco-system.

Fly ash contains significant amount of magnetic minerals of which iron oxides are in majority[17-20]. Among several iron oxides, Fe_3O_4 might be the most vital root of anthropogenic magnetic particles in soils, sediments and tree leaves in the fly ash affected areas[13]. Further, there are some reports that coals as available in India contains 1.8-6.0 ppm ^{238}U and 6.0-15.0 ppm of ^{232}Th[21] where 1 ppm ^{238}U and ^{232}Th corresponds to 12.3 and 4.0 Bq kg^{-1} , respectively, and it has a significant amount of radioactivity, which is a serious threat to the environment[22]. So, some attempts are warranted to find the constituents of the fly ash coming out of thermal power plants after coal combustion, for their proper disposal and treatment in view of safety aspects.

For last couple of years, the present authors have been carrying out a systematic study to identify the sources of different pollutants due to combustion, which might come in contact with air, water and soil[23,24]. The objective of the present work is to know the constituents of the products of three thermal power plants after coal combustion, which come directly out of the power plants and spread all over the surrounding habitats, and to understand their physical properties which may provide information on toxic contribution by the thermal power plants in the surrounding environment. Precisely, in the present study, we attempt to investigate the physical nature of the fly ashes from three thermal power plants situated in West Bengal, India and estimate the grain size of the particles to know the presence of very fine grains, which are likely to be inhaled into lungs causing diseases of various natures. This investigation also includes a systematic study of magnetic properties of the fly ashes in order to verify if fly ashes contain magnetic fraction and to identify its mineral sources. Further, this report includes a comparative study of fly ashes collected from three power plants.

Materials And Methods

The samples of fly ashes were collected from three thermal power plants located at Kolaghat, Bakreswar and Bandel in West Bengal, India, and these samples are hereafter called as KOL, BAK and BAN, respectively. For morphology and compositional analysis, we have used scanning electron microcopy (SEM, model: FEI Quanta 200F) coupled with energy dispersive X-ray spectrometry (EDX, model: EDAX). Crystalline structural aspects were investigated using X-ray powder diffraction techniques (Seifert XRD 3000 TT). The magnetic measurement of the samples was carried out by Quantum Design's MPMS XL SQUID magnetometer. ^{57}Fe Mössbauer spectra were recorded using a conventional constant-acceleration spectrometer with a $^{57}Co/Rh$ Mössbauer source.

Results And Discussion

Fig. 1 represents SEM pictures of KOL, BAK and BAN samples showing surface morphology and particle nature. These images show the presence of spherical particles of various sizes in these fly ash samples. The spherical particles may be spherules usually observed in the combustion wastes[25]. The particle sizes lie within 0.16-5.50 m for KOL, 0.29-4.14 m for BAK and 0.18-5.90 m range for BAN. These spheres exhibit several morphology and textures on the surface of these particles. Though apparently the spheres appear with fine surfaces, rough surfaces too appear with small potholes.

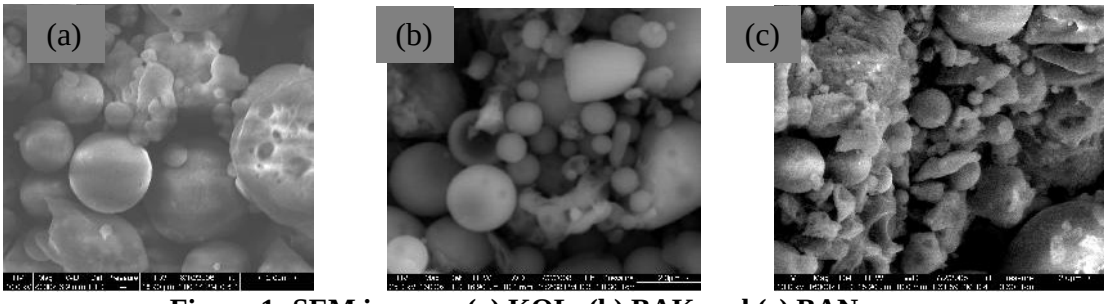

Figure 1: SEM images: (a) KOL, (b) BAK and (c) BAN.

Fig.1 also shows that these samples contain larger fraction of amorphous materials which usually emerge with asymmetrical shapes, than usually the spherical particles which correspond to magnetic fraction of any fly ash sample[13]. The non-magnetic components are generally irregular in shape and size. The magnetic

components have a general tendency to be spheroidal. With decrease in particle size the spheroidal nature of the magnetic particles increases. A reasonable mechanism for formation of such particles may be as follows: (a) the mineral matters present in coal (both intrinsic and extrinsic) undergo a series of transformations before fusion and melting[26], (b) subsequently the melts condense into smaller spheroidal particles, and (c) iron oxide (Fe^{+2}, Fe^{+3})/elemental Fe derived from iron carbonate present in fly ash[27] condense on the surfaces of such spheroidal particles[28].

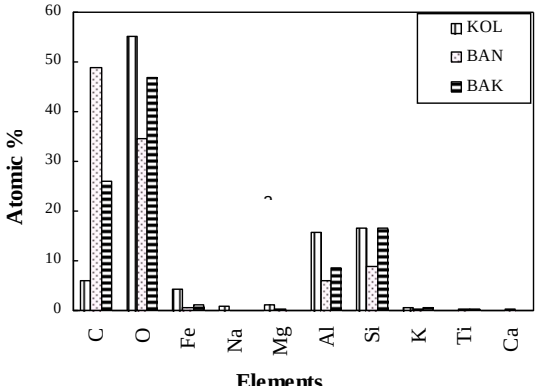

Figure 2: Comparison of different elements present in three fly ash samples

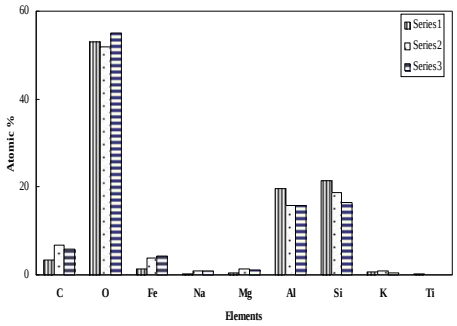

Figure 3: Elements present in KOL obtained from EDX spectra taken at different spot areas.

The EDX spectra of KOL, BAK and BAN were taken from different spot areas of the homogeneous regions of the SEM images of the specimen sample to get reliable information as regards the presence of elements in the specimens along with their relative abundance. The elements revealed by the EDX spectra are O, Al, Si, C, Fe, K, Ti, Mg and Na for KOL, whereas O, Al, Si, C, Fe, K and Ti for BAK and BAN (Fig. 2). This result demonstrates that nearly same type of coal was used in two power plants at Bakreswar and Bandel but a different type was used in the other plant at Kolaghat as KOL sample contains two additional elements – Mg and Na. The samples detected in KOL are in general agreement with as seen earlier[29]. Interestingly, there is no signal for S in the present fly ash samples of our study, which is usually observed in many fly ash samples. Thus, in case of these fly ashes, there is no chance of sulfur-contamination of the environment due to air-borne/dumping of fly ash. The relatively weak signal intensity for iron indicates the presence of much lower concentration of iron in these spherical particles. Notably, no radioactive element in these samples could be detected as was reported earlier[21]. It is noticed that the EDX spectra taken at different spot areas for any sample are in general agreement with each other. A representative diagram of the quantitative estimation of the elements based on the EDX spectra obtained over three spot areas for KOL as a representative one is shown in Fig. 3. This figure indicates that the spherical particles of any particular sample are made-up of the same elements and the elements are nearly homogeneously distributed.

The XRD study of the fly ash samples was carried out at room temperature. Fig. 4 represents the powder XRD pattern for BAN as a representative one. The diffraction peaks/lines are compared with those of possible minerals as references. The major components present in the three fly ash samples as detected by XRD are presented in Table 1. From the XRD peak broadening, the mean crystallite size (D) of the major components was estimated using Scherer formula[29]. The major components in KOL, BAK and BAN samples are mullite ($Al_6Si_2O_{13}$) and quartz (SiO_2). Some additional small Bragg peaks can also be detected, and those are due to the presence of hematite (JCPDF no. 24-72 and 13-534) and microcline (Al, K, Si) (JCPDF PDF 3-471). From accurate estimation from the XRD profile, it is also possible that these samples possess magnetite / maghemite (ICDD PDF 19-629) and as well as free iron (ICDD PDF 18-877) in small fractions.

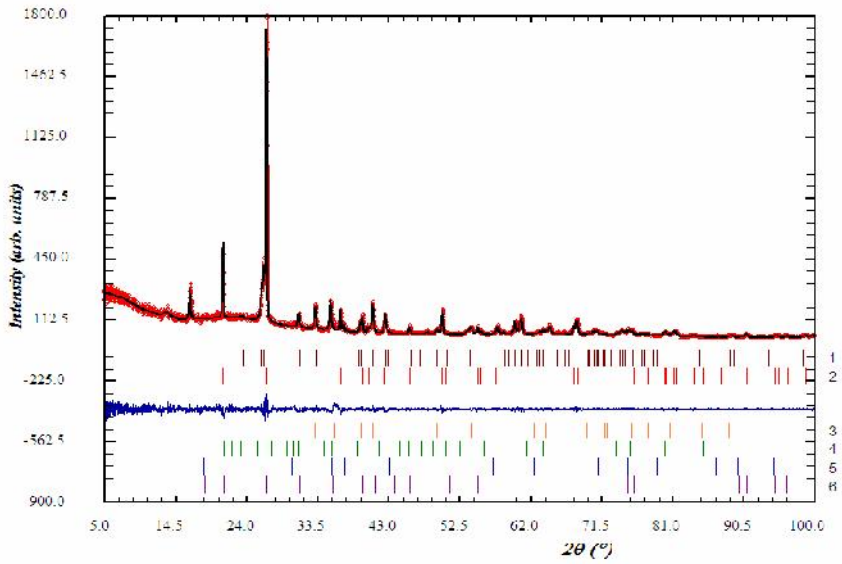

Figure 4: Powder XRD pattern of BAN at 300 K.

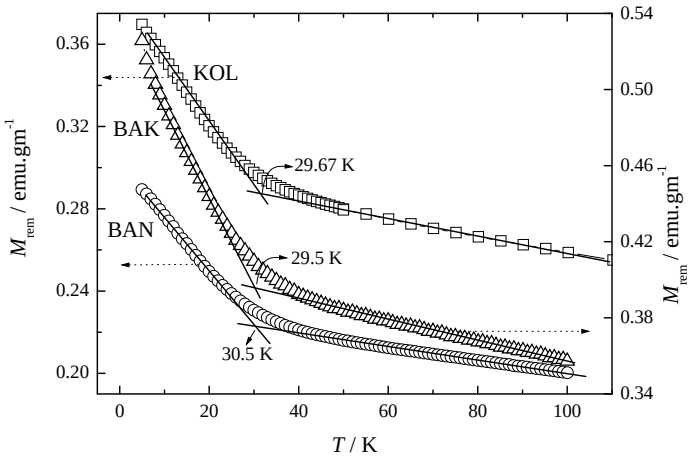

Figure 5: M_{rem} vs. T plots for fly ash samples. Dotted arrows show the

Table 1: Results of the XRD study of the fly ash samples.

Sample	Major components	Lattice parameters				
		a / Å	b / Å	c / Å	V / Å^3	D / Å
KOL	mullite ($Al_6Si_2O_{13}$) (ICDD PDF 15-776)	7.564	7.699	2.888	168.239	419.42
	quartz (SiO_2) (ICDD PDF 5-490, 33-1161)	4.919	4.919	5.408	113.352	742.61
BAK	mullite (ICDD PDF 15-776)	7.553	7.687	2.884	167.488	325.03
	quartz (ICDD PDF 5-490, 33-1161)	4.912	4.912	5.402	112.897	617.26
BAN	mullite (ICDD PDF 15-776)	7.557	7.695	2.885	167.806	272.24
	quartz (ICDD PDF 5-490, 33-1161)	4.914	4.914	5.402	112.985	814.61

In order to investigate the magnetism of the fly ash samples, we attempted to study the remanent magnetization. To estimate any remanent magnetization (M_{rem}) of the fly ash samples, each sample was initially cooled down to 5 K from room temperature under 10 kOe magnetic field. After reaching 5 K the magnetic field was rapidly switched to 50 Oe. Then M_{rem} was measured while heating. Fig. 5 represents the M_{rem} vs. T plots for three fly ash samples. The value of M_{rem} is found to decrease rapidly with increasing temperature above 5K, while beyond an inflection point (~30 K, ~29.5 K and 27.7 K for KOL, BAK and BAN, respectively) the temperature variation of M_{rem} becomes gradual and decreases linearly with further increase of temperature. The inflection point is usually considered the magnetic transition temperature for conventional magnetic materials. However, it is difficult to assign any transition temperature as these samples may be mixtures of two or more metallic magnetic oxides as evidenced by the EDX and XRD studies. From Fig. 5, it is certain that the fly ash samples are magnetic (either ferri- or ferromagnetic). It is also clear from the present M_{rem} vs. T data that the M_{rem} values are different for the three fly ash samples in 5 – 100 K range as well as at the lowest working temperature (M_{rem} at 5 K: 0.37, 0.53 and 0.29 emu/gm for KOL, BAK and BAN, respectively). So, it may be concluded that the different extent of magnetic remanence observed in these samples are owing to the various magnetic minerals present in these samples in different amounts. Temperature dependent magnetization (M) measurement was carried out in 5-300K range under 10 kOe magnetic field. The observed $M(T)$ data were used to estimate the magnetic susceptibility ($\chi = M/H$). The $\chi(T)$ data for all fly ash samples, in general, obey the Curie-Weiss law [$\chi = C/(T-\theta)$] at $T >$ 100 K with negative θ values. The negative θ values indicate antiferromagnetic interactions present in these fly ash materials.

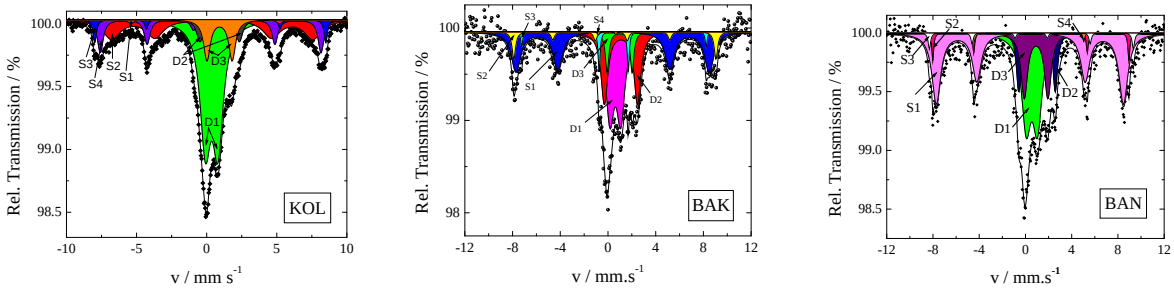

Figure 6: ^{57}Fe Mössbauer spectra of fly ash samples.

By using ^{57}Fe Mössbauer spectroscopy[30,31] one can identify different iron compounds present in a solid mixture. In this light ^{57}Fe Mössbauer spectra of KOL at 300 K, BAK and BAN at 80 K were recorded (Fig. 6). The spectra were fitted to Lorentzian lines with isomer shift ¢ (relative to α-iron), quadrupole interaction aE_Q, hyperfine field H_n and the line width as the variable parameters for any given component in the spectral fits. The Mössbauer spectra of each sample can be well fitted with three doublets (say, D1, D2 and D3) and four sextets (say, S1, S2, S3 and S4). Thus, the samples have paramagnetic as well as magnetic fractions. The corresponding hyperfine parameters estimated from the best-fitted spectra are presented in Table 2. The site population has been calculated from the related area of the various subspectra with respect to the total area of spectra.

The paramagnetic part of the Mössbauer spectra can be significantly fitted with one Fe^{3+} and two Fe^{2+} doublets. In Fig. 6 the doublet D1 can readily be assigned to Fe^{3+} which may correspond to an average spectrum of mullite along with some contribution from superparamagnetic hematite or maghemite[32] . The doublets D2 and D3 are typical for Fe^{2+}. The origin of this Fe^{2+} is unidentified, as was seen by Vandenberghe et al[32]. There are several reports on the investigation of the decomposition of coal[33-37]. The ferrous iron in silicate has high-spin configuration. During combustion the high spin silicate compounds are transformed to high spin coal ash in two different Fe^{+2} and Fe^{+3} states. However, due to the change of their ionization state, the isomer shift as well as their quadrupole splitting is changed. This perhaps reflects the observation of two different Fe^{2+} species.

The magnetic part of the Mössbauer spectrum consists of four sextets S1, S2, S3 and S4. The hyperfine parameters observed for sextets S1 and S2 correspond to tetrahedral sites and octahedral sites in magnetite, respectively, while those for sextet S3 indicate the presence of small amount of hematite. This spectrum shows the very small presence of -Fe in the fly ash sample (sextet S4). The ignition of the unidentified Fe^{2+} compound in air at high temperatures may be giving rise to the -Fe, magnetite as well as some hematite[32]. From literature it is known that coal contains szomolnokite ($FeSO_4 \cdot H_2O$)[38] which contributes to Fe^{2+} doublet in the Mössbauer spectra of unburnt coal. However, the present XRD study could not detect this material in the fly

ash samples studied. The occurrence of magnetite / hematite may be due to the transformation of pyrite and szomolnokite during coal combustion[39]. Comparing the paramagnetic components owing to various Fe species in KOL, BAK and BAN samples, it is evident from Table 2 that the proportion of mullite and other silicates is large in general, but significantly large in the KOL sample. It is clear that the most dominating magnetic phase present in these samples is magnetite, and the ratio of population in magnetite and hematite phases are 1:0.11, 1:0.20 and 1:0.07 for KOL, BAK and BAN, respectively. It has to be noted that the total population ratio of paramagnetic and magnetic phases in these three samples are interestingly nearly similar (1:0.7, 1:0.8 and 1:1.2 for KOL, BAK and BAN, respectively). Comparing the population of the different Fe species, mullite with Fe^{3+} has the highest share among the paramagnetic components, whereas magnetite leads among the magnetic components. Thus, the Mössbauer spectroscopic observations are in complete agreement with the observations made from the XRD study.

Table 2: Hyperfine parameters estimated from the ^{57}Fe Mössbauer spectra

Sample	Sub spectra	δ(mm.s^{-1})	ΔE$_Q$(mm.s^{-1})	H$_{hf}$(kOe)	A (%)	Origin
	D1	0.36	0.90	-	44	Fe^{3+} in mullite / other silicates
	D2	0.93	2.80	-	05	Fe^{2+} in silicates
	D3	0.92	1.80	-	10	,,
KOL#	S1	0.30	-0.01	488	14	Tetrahedral sites in Magnetite
	S2	0.43	-0.04	445	22	Octahedral sites in Magnetite
	S3	0.36	-0.10	511	4	Hematite
	S4	0.06	0	328	1	α-Fe
	D1	0.62	0.91	-	29	Fe^{3+} in mullite / other silicates
	D2	1.08	2.77	-	21	Fe^{2+} in silicates
	D3	0.98	2.10	-	6	,,
BAK*	S1	0.49	0.01	480	5	Tetrahedral sites in Magnetite
	S2	0.44	-0.05	502	31	Octahedral sites in Magnetite
	S3	0.43	0.15	526	7	Hematite
	S4	0.08	0	290	1	α-Fe
	D1	0.55	0.96	-	30	Fe^{3+} in mullite / other silicates
	D2	1.00	3.16	-	10	Fe^{2+} in silicates
	D3	0.92	2.04	-	15	,,
BAN*	S1	0.50	0.01	547	3	Tetrahedral sites in Magnetite
	S2	0.43	-0.05	502	38	Octahedral sites in Magnetite
	S3	0.45	0.01	526	3	Hematite
	S4	0.33	0	292	1	α-Fe

at 300 K; * at 80 K; ¢ = isomer shift relative to α-Fe at room temperature; aE_Q = quadrupole shift; H_{hf} = magnetic hyperfine field at ^{57}Fe nuclei; A = area of the spectrum component

In general, comparing the hyperfine parameters obtained from Mössbauer spectroscopy for KOL, BAK and BAN fly ash samples as well as the observations of other physical characterizations (viz. EDX, magnetic and XRD) discussed here, it may be concluded that the BAK and BAN samples are of similar elemental composition but different from that of the KOL, whereas all these fly ash samples are in general of similar magnetic nature as far as the iron-dependent mineralogy is concerned. A long heat treatment period or heat treatment at higher temperature has been usually held the root cause of different iron oxide formations [40]. At this point, one should note that the interpretation of different observations made in the present study with different fly ash samples may lie in the actual combustion mechanism taken place in the power plants. The use of coals from different sources may be one origin of this difference. Moreover, the presence of other metal(s), as detected by EDX study, in KOL, besides those common to BAK and BAN, may affect the magnetic structure of KOL.

Conclusion

The paper sheds light on the physical nature of the fly ashes as collected from the thermal power plants. This work encompasses various measurements that include particle size, nature of the minerals, structures,

morphology, elemental analysis, physical nature of conglomeration, remanent magnetization, spin states of iron, relative abundance of magnetic phases etc. present in the samples. The samples consist of coarse, fine and ultra fine particulate materials of varying dimensions, showing magnetic properties. This work highlights the presence of ultrafine particulates bearing magnetism and puts a special emphasis on magnetic measurements with an objective that the same could be used as probe for investigation of such samples as regards their presence and estimation under same situations. Besides, the authors have undertaken this work to gather an idea about the nature of hazards in the surrounding habitats of the thermal power plants run on coals. In fact, ultrafine particulate matter of the fly ashes is one of the potential air pollutants inhalation of which would cause several physiological disorders and other related health problems. The present paper may invoke attention in its own way to some sections of people concerned for environmental management and related issues.

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