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Preparation and Characterization of Ceramics from Coal Fly Ash

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Fly ash is produced in large quantities from thermal power plants utilizing coal as fuel. While many applications of fly ash such as cement, construction, bricks *etc*. are being explored, a large share of fly ash is still unutilized. One of the applications being examined for fly ash utilization is the production of glass and glass ceramics. This study reports the investigation on sintering/melting of fly ash with additives with the objective of obtaining ceramics using coal fly ash from a thermal power plant near Delhi. Attempts were made to melt mixtures of fly ash with different additives by subjecting them to temperatures of 850, 900 and 950 ºC. The samples showed different extent of sintering or melting based on the additives. X-ray diffraction analysis of the samples showed them to be polycrystalline in nature with crystalline phases dependent on the initial composition of the sample.

Key Words: Coal fly ash, Glass ceramics, Sintering, Fly ash utilization.

INTRODUCTION

Thermal power plants in India are known to produce *ca.* 110 million tons of coal fly ash annually. Its production is estimated to rise to 180 million tons by the year 2015[1]. India still depends on coal for its major energy needs, thus the management and utilization of coal fly ash will remain a significant challenge for Indian thermal power plants. Fly ash is being examined as a resource for many applications and a large amount is utilized in the cement, brick and other construction industries[2-4]. However, a significant amount is still unutilized and its disposal causes considerable environmental concern[5]. Fly ash is known to contain heavy and hazardous metals like Zn, Ni, Pb, Hg and Cd that can harm the environment[6]. Improper disposal of fly ash in land fill sites can cause leaching of the toxic metals into the soil and groundwater causing damage to the ecosystem[5]. This highlights the need to look into technologies that not only use the waste fly ash as a resource but also immobilize the toxic metals thereby preventing them from harming the outer environment[4,7,8]. Utilization of coal fly ash for preparation of glass or ceramics fulfills this need. This technique is advantageous since it employs a waste as a raw material to produce a commercial product which would otherwise have proved hazardous for the environment. This study analyses the possibility of using coal fly ash from a power plant near Delhi for the production of ceramics. In this power plant, *ca.* 23.7 million tons of ash was put to use in the year 2007-08 in dry ash evacuation systems (DAES), as fly ash bricks, in agricultural applications, as embankments on the road[9].

EXPERIMENTAL

The fly ash was obtained from a thermal power plant near Delhi. Table-1 shows the composition of a typical coal fly ash sample taken from this power plant[10]. To prepare the samples, a 5 g mixture of fly ash with appropriate additives was taken. The additives were chosen to assist in formation of low melting phases which will enable sintering at lower temperatures or in formation of glassy melt. Additives like Na_2O, K_2O were used with the aim of lowering the melting temperature of the mixture; other oxides like CaO, MgO and Fe_2O_3 were added to serve as nuclei of crystallization for low melting phases. Details of the 19 chemical compositions that were tested are shown in Table-2. Samples 1, 2, 3 and 8 were prepared with a high percentage (92-97 % by weight) of fly ash with considerably low quantities of additives. Samples 13-16 also had high percentage of fly ash in the range 85-90 % by weight. On the other extreme, sample 9 had only 30 % fly ash with Na_2O and CaO additives. Na_2O was used as an additive in samples 5 and 12. Samples no. 4 and 11 were prepared with CaO alone and CaO with Na_2O as an additive respectively. Samples 6 and 7 were prepared with additions of 20 and 30 % of alumina, respectively. It is believed that waste glass when added to a glass preparation mixture helps it to attain various new

TABLE-1
TYPICAL CHEMICAL COMPOSITION OF COAL FLY ASH USED IN THIS STUDY[10]

Component	Wt %
SiO_2	65.0
CaO	0.5
Al_2O_3	22.1
Fe_2O_3	3.3
MgO	0.4
Total alkali content (Na_2O and K_2O)	1.2

TABLE-2
COMPOSITIONS OF FLY ASH WITH ADDITIVES

Sample No.	Composition details
1	97 % fly ash, 3 % MgO
2	97 % fly ash, 3 % Al_2O_3
3	92 % fly ash, 2 % MgO, 3 % CaO, 3 % Fe_2O_3
4	70 % fly ash, 30 % CaO
5	66 % fly ash, 34 % Na_2O
6	50 % fly ash, 2 % MgO, 8.5 % Na_2O, 3 % Fe_2O_3, 30 % Al_2O_3, 6.5 % CaO
7	55 % fly ash, 20 % alumina, 12.5 % Na_2O, 12.5 % CaO
8	96 % fly ash, 1 % MgO, 3 % Fe_2O_3
9	30 % fly ash, 35 % Na_2O, 35 % CaO
10	1 g of fly ash having (5 % MgO, 18 % CaO, 77 % fly ash) with 4 g of waste glass
11	34 % fly ash, 33 % CaO, 33 % Na_2O
12	34 % fly ash, 66 % Na_2O
13	87 % fly ash, 13 % Na_2O
14	90 % fly ash, 2 % MgO, 8 % Na_2O
15	85 % fly ash, 2 % MgO, 13 % Na_2O
16	85 % fly ash, 4 % MgO, 11 %Na_2O
17	80 % fly ash, 10 % Na_2O, 10 % borax
18	90 % fly ash, 10 % borax
19	80 % fly ash, 10 % CaO, 10 % borax

crystalline phases and properties[11,12]; sample 10 was prepared with this objective. Borax is a popular additive in glass making that helps in lowering the vitrification temperature; samples 17, 18 and 19 were made with borax (10 % by weight) as an additive, with secondary additions of Na_2O and CaO. The 19 compositions were mixed thoroughly in silica crucibles, to ensure homogeneity and were then subjected to heat treatment for 6 to 7 h in a muffle furnace. A heat treatment at 850 ºC was used to study the effect of additives. Selected samples were then heated at 900 and 950 ºC.

Selected samples were analyzed for phases present using X-ray diffraction (XRD). A Philips Xpert MPD diffractometer with monochromatic Cu ka radiation was used.

RESULTS AND DISCUSSION

All the 19 samples were heated at 850 °C were visually examined to check if any fusion or sintering had taken place. Table-3 shows the state of fly ash samples that underwent changes at 850 °C. The other samples remained in powder form. In general it is observed that the compositions that did not undergo changes at 850 °C are the ones with high fly ash content (samples 1, 2, 3 and 8) or with high alumina content (7). Presence of Na_2O was beneficial even for samples with high fly ash content (samples 13, 14, 15, 16). Na_2O is an effective fluxing additive and appears to have caused formation

TABLE-3
CONDITION OF SAMPLES HEATED TO 850 ºC

Sample No.	Composition of sample	Observed change at 850 ºC
4	70 % fly ash, 30 % CaO	Sintered
5	66 % fly ash, 34 % Na_2O	Sintered
6	50 % fly ash, 2 % MgO, 8.5 % Na_2O, 3 % Fe_2O_3, 30 % Al_2O_3, 6.5 % CaO	Sintered
8	96 % fly ash, 1 % MgO, 3 % Fe_2O_3	Sintered
9	30 % fly ash, 35 % Na_2O, 35 % CaO	Sintered
10	1 g of fly ash having (5 % MgO, 18 % CaO, 77 % fly ash) with 4 g of waste glass	Sintered
11	34 % fly ash, 33 % CaO, 33 % Na_2O	Sintered
12	34 % fly ash, 66 % Na_2O	Sintered
13	87 % fly ash, 13 % Na_2O	Sintered
14	90 % fly ash, 2 % MgO, 8 % Na_2O	Sintered

of low melting compounds which contributed to sintering at 850 ºC. Even though sample 6 has high alumina, the presence of many additives has promoted sintering compared to 7 which has only Na_2O and CaO. Samples 9 and 11 which have similar compositions with almost equal amounts of fly ash with Na_2O and CaO underwent sintering.

The effect of temperature showed that at 900 °C, no major increase in extent of sintering of the sample was noticed, at 950 °C some samples showed melting or greater extent of sintering (Table-4). In case of sample 5, at 900 and 950 °C, the samples underwent complete melting and expansion forming porous fragile pieces as shown in Fig. 1. In case of samples 9, 11 and 13, the sample heated to 950 °C showed extensive sintering and appearance of melting at the surface. The photos of 9 and 13 are also shown in Fig. 1. Samples 6 and 14 showed extensive sintering as shown in Fig. 1.


Sample No. 5 Sample No. 6 Sample No. 9
Sample No. 13 Sample No. 14

Fig. 1. Photographs of samples that showed good sintering or melting at 950 ºC

Samples 5, 6, 9, 13 and 14 were analyzed for phases present. Samples 5 and 13 have different fly ash and Na_2O ratios. In case of sample 5 with higher Na_2O content, the phases present are silica and sodium aluminosilicate (Fig. 2). In sample

TABLE-4
CONDITION OF SAMPLES HEATED AT 850, 900 AND 950 ºC

S. No.	Compositions	850 ºC	900 ºC	950 ºC
5	66 % fly ash, 34 % Na_2O	Sintered	Melted and porous	Melted and porous
6	50 % fly ash, 2 % MgO, 8.5 % Na_2O, 3 % Fe_2O_3, 30 % Al_2O_3, 6.5 % CaO	Sintered	Sintered	Sintered
8	96 % fly ash, 1 % MgO, 3 % Fe_2O_3	Sintered	Sintered	Sintered
9	30 % fly ash, 35 % Na_2O, 35 % CaO	Sintered	Sintered with surface melting	Sintered with surface melting
10	1 g of fly ash having (5 % MgO, 18 % CaO, 77 % fly ash) with 4 g of waste glass	Sintered	Sintered	Sintered
13	87 % fly ash, 13 % Na_2O	Sintered	Sintered	Sintered with surface melting
14	90 % fly ash, 2 % MgO, 8 % Na_2O	Sintered	Sintered	Sintered

13, mullite is also present (Fig. 3). It appears that Na_2O reacts with mullite and silica to form sodium aluminosilicates which melts and causes liquid phase sintering or melting of the sample. Sample 14 which was similar to sample 13 except for MgO addition showed similar phases and the MgO appeared unreacted (Fig. 4). Sample 6 which had several additives showed the presence of magnesium and sodium aluminosilicates along with added Al_2O_3 and CaO (Fig. 5). Sample 9 had mixed aluminosilicates of Ca and Na (Fig. 6).

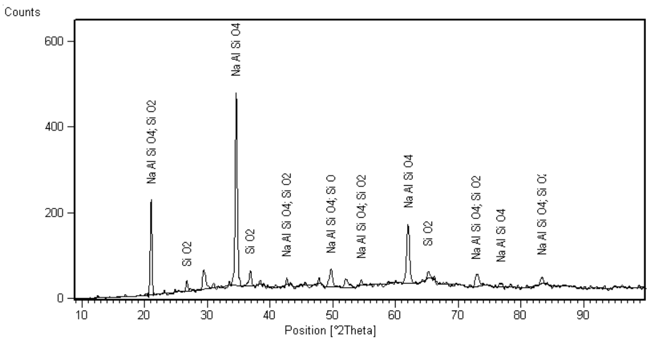

Fig. 2. Phase analysis of sample number 5

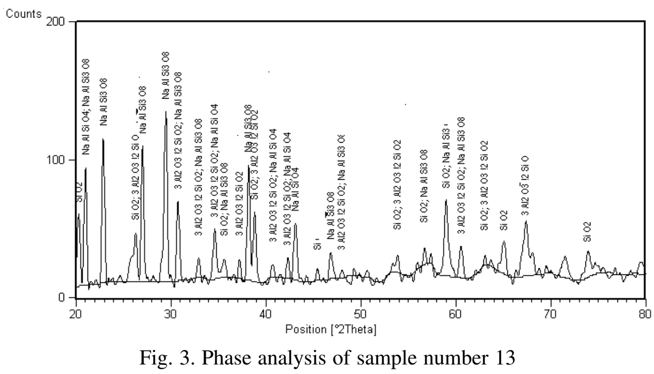

Fig. 3. Phase analysis of sample number 13

Conclusion

In this work attempts were made to produce ceramics from coal fly ash by suitable heat treatment procedure with proper

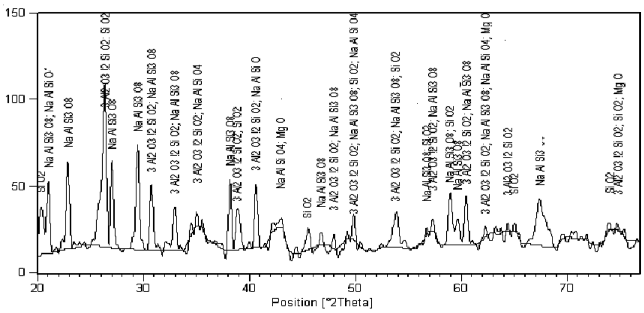

Fig. 4. Phase analysis of sample number 14

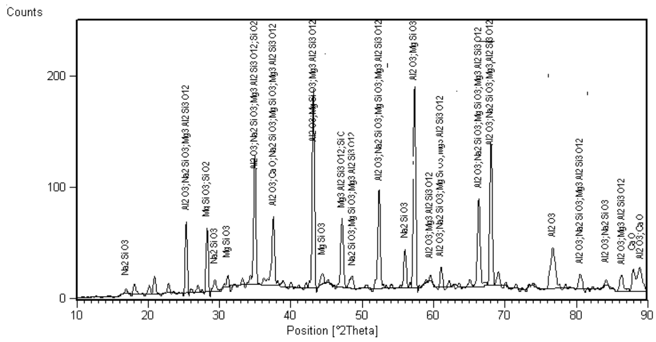

Fig. 5. Phase analysis of sample number 6

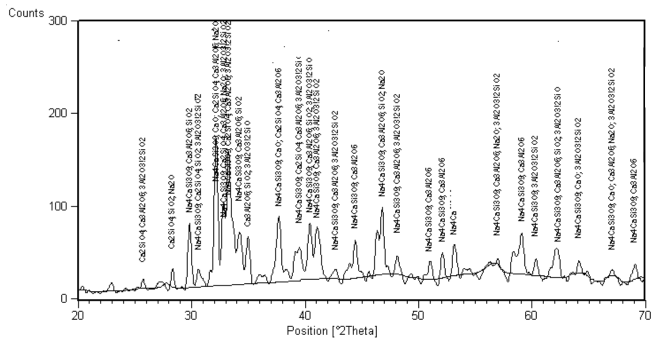

Fig. 6. Phase analysis of sample number 9

fluxing additives. It is observed that when fly ash was used in a high percentage (85-95 %) samples did not show sintering or melting. Na_2O is found to be an effective fluxing additive as observed by the changes in samples containing this additive. XRD analysis of the samples with different additives showed it to be polycrystalline in nature. Samples with Na_2O additive show the presence of sodium aluminosilicate that could have caused the lowering in sintering temperature. Results of the study confirmed the possibility of converting coal fly ash into ceramics at low sintering temperatures with the aid of suitable additives.

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