Microstructure and leaching characteristics of fly ash-mine overburden-lime mixtures

B. Behera and M. K. Mishra

Abstract**—**Fly ash is a major problem for disposal. Its gainful application is being tested at many places. An experimental investigation has been carried out to convert fly ash in combination with another waste material for mining applications. This paper highlights microstructure and leaching characteristics of the developed fly ash, mine overburden and lime mixtures. Fly ash from a local unit and mine overburden from an Opencast coal mine, India were collected, characterized, mixed with lime in different ratios and compacted to different values. The compacted specimens for California bearing ratio tests were cured for 7 and 28 days. The bearing ratio values and microstructural analysis were determined to know the effect of curing. The compacted specimens in the permeability mould were cured for 7 days to carry out the leaching test. The leachate effluents collected from leaching study were analyzed for the metals, Ni, Cr and Pb by a flame atomic absorption spectrophotometer.

Keywords**—**Fly ash, leachate effluent, microstructure, mine overburden

I. Introduction

During the combustion of pulverized coal in power stations more than 80% of the ash residue exits the furnace and dispersed in the combustion product gases and is designated 'fly ash'. This fly ash is removed from the furnace exit gases, either by electrostatic precipitators or bag filters, before the latter are vented to the atmosphere. The current annual production of fly ash worldwide is estimated around 600 million tons, with fly ash constituting about 500 million tons at 75-80% of the total ash produced [1]. In India, over 130 millions of tonnes of fly ash are produced yearly. As per an estimate fly ash generation is expected to increase to about 170 million tonne by 2012 and 225 million tonne by 2017. Fly ash has been considered as a “Polluting Industrial Waste” till about a decade back and was being disposed off in ash ponds occupying large areas of land [2]. But it has been reconsidered as a resource material over a period of time and is being widely used for manufacture of cement, part replacement of cement in mortar and concrete, manufacture of bricks, blocks, tiles, roofing sheets and other building components, construction of roads /embankments, reclamation of low lying areas, back-filling of mines, agriculture and related application, etc. In all these applications, fly ash has found to contribute positively on technical parameters as well as on environmental aspects. Utilization of fly ash in construction helps in saving of land areas. However all these applications do not accommodate all the fly ash that is generated so new avenues are explored.

In India, non-cementitious fly ashes (Class F type) are generated in more quantities than cementitious fly ashes (Class C type) due to burning of bituminous coals in thermal power plants. These types of fly ashes contain higher amount of SiO_2 and Al_2O_3. These react with an activator rich in CaO such as lime, cement, lime kiln dust or cement kiln dust in the presence of moisture to form cementitious compounds for stabilization applications. Various investors studied the enhancement of the strength of fly ash as a result of actions between fly ash and lime [3]–[7]. Arora and Aydilek [8] evaluated the engineering properties of Class F fly ash amended soils as highway base materials. They mixed fly ash (40%) with sandy soils with plastic fines contents and activated the mix with 7% cement and obtained California bearing ratio (CBR) of 140%. The class-F fly ash after stabilizing with 10% lime and 1% gypsum achieved a CBR value of 172% at 28 days curing reported by Ghosh and Subbarao [9]. Ghosh and Subbarao [10] evaluated the microstructural characteristics of Class F fly ash stabilized with lime (6 and 10%) and gypsum (1%). Stabilization of fly ash with proper additives was one of the promising methods to mitigate the problems of leaching and dusting [11]. Investigation of Goswami and Mahanta [12] on the use of fly ash and lime for stabilization of lateritic soil was likely to have no significant impact on the environment, as most of the toxic metals present in the fly ash were within the threshold limits. The high pH induced by lime treatment of the mixes helped in keeping most of the metals within the stabilised soil matrix. The quantity of a metal in the leachate is predominantly influenced by the hydraulic conductivity of the stabilized material and the concentration of a metal in the leachate [13]– [14]. Wang et al. [15] carried out comparative leaching experiments for trace elements in raw coal, fly ash and bottom ash and identified lead (Pb) and Arsenic (As) as the potential toxic elements. The pH of the solution and leaching time were also found to strongly influence the leaching behavior. The leaching intensity of strontium (Sr),

Dr. M. K. Mishra, Assoc Professor, is with the National Institute of Technology, Rourkela - 769008 India (corresponding author phone: 91-661-2462602; fax: 91-661-2462022; e-mail: mkmishra@nitrkl.ac.in). He acknowledges the fund provided by Fly Ash Unit, DST, Govt. of India vide approval No: *FAU/DST/600(17)/2008-09* dated *12.09.2008*

Ms. B. Behera, Research Scholar, is with the National Institute of Technology, Rourkela-769008 India (e-mail: banita@nitrkl.ac.in).

zinc (Zn), lead (Pb), nickel (Ni) and arsenic (As) were found to increase with decreasing pH of the solution. The stronger the acidity of solution, the larger is the leaching intensity of these elements.

The overburden (O/B) material is a very important raw material which has been traditionally used in a limited way almost restricted to the mine itself. Typically it is dumped back to voids created by surface mining operations. It causes geotechnical and environmental problems on random disposal. The overburden is highly heterogeneous. It usually consists of a mixture of coarse-grained particles to rock fragments and fine-grained particles. Gradation results suggest that fines and coarse grains are approximately equally represented in the soil [16]. The present study reports the results of California bearing ratio (CBR) test, microstructural and leaching analyses of the fly ash, mine overburden and lime mixtures.

II. MATERIALS AND METHODS

A. Materials

Fly ash was collected from a local thermal power plant. The mine overburden was collected from Bharatpur opencast coal mine, Talcher, India. The additives selected were commercially available superior grade quick lime (make: Rajasthan Lime, Goyal Udyog, India). The chemical composition and physical properties of the fly ash and mine overburden are represented in Table I and Table II. The major chemical constituents in the fly ash and mine overburden were silica, alumina and iron. Calcium content was less in the fly ash and according to ASTM 618 specification, this fly ash is classified as “Class F” fly ash. The specific gravity of fly ash was found to be less than that of mine overburden, due to the less iron content.

TABLE I
CHEMICAL COMPOSITIONS OF FLY ASH AND O/B (WEIGHT %)

Constituents	Fly ash	Mine Overburden
SiO_2	50.88	49.8
Al_2O_3	34.78	28.49
Fe_2O_3	6.31	8.32
CaO	0.52	1.09
K_2O	1.42	0.39
MgO	0.51	1.23
TiO_2	2.95	0.69
Na_2O	0.2	--
LOI	2.4	10

Note: O/B = Overburden, LOI = loss on ignition

B. Methods

Weight fractions of fly ash of 15%, 25% and 35% were used to mix with mine overburden. The lime content of 3% and 6% by weight of the total mix (fly ash and overburden) was used in the study. The modified Proctor compaction (heavy compaction) test was performed to determine the maximum dry density and optimum moisture content of the fly ash, overburden material and all the mixes as per IS: 2720 (Part 8). Samples for CBR tests were prepared at their respective optimum moisture content (OMC) and maximum dry density (MDD). The ingredients such as fly ash, mine overburden and lime were blended in the required proportion in dry state. Then required amount of water corresponding to OMC was added to the mixes and mixed thoroughly. The mixture was left in a closed container for uniform mixing and prevents loss of moisture to atmosphere. Then the sample was statically compacted to 95% of modified Proctor maximum dry density in the standard CBR mould of 150mm diameter and 175mm height, such that the height was maintained at 127mm. A circular metal spacer disc of 148 mm diameter and 47.7mm height was used to compact the sample. California bearing ratio (CBR) tests were performed in accordance with IS: 2720 (Part 16). The samples were soaked for four days in water and were allowed to drain for 15 min before test to obtain soaked condition results. The curing periods adopted were immediate, 7 days (3 days moist curing + 4 days soaking) and 28 days (24 days moist curing + 4 days soaking). CBR tests were carried out at the end of respective curing period. Two surcharge disks, each weighing 2.5 kg, were placed over the sample and a plunger, 50 mm in diameter, was used to penetrate the sample at a rate of 1.25 mm/min during CBR test. Scanning electron microscopy (SEM) analyses were conducted on 28 days cured specimens. A JEOL JSM 6480 LV, (Japan) model SEM fitted with EDX micro analyzer was used for the SEM study.

TABLE II
PHYSICAL PROPERTIES OF FLY ASH AND MINE OVERBURDEN

Property	Fly ash	Overburden
Specific gravity	2.16	2.6
Particle size analysis (%)		
Gravel (>4.75 mm)	--	9.71
Sand (4.75 mm – 0.075 mm)	22.17	32.91
Silt (0.075 mm – 0.002 mm)	75.04	43.73
Clay (<0.002 mm)	2.79	13.65
Consistency limits		
Liquid limit (%)	30.75	25.70
Plastic limit (%)	Non-plastic	15.04
Shrinkage limit (%)	--	13.44
Plasticity index (%)	--	10.66

Leaching study was conducted by permeability method as per IS: 2720 (Part 17) for the collection of leaching effluents. Samples for permeability test were prepared following the same process as explained for CBR test. In this case, the wet mixtures of the samples were compacted in the permeameter mould of 100mm internal diameter and 127mm height. After compaction the samples were cured for 7 days. Permeability tests were carried out at the end of 7 days curing period. The leaching effluents coming out from specimens through the outlet of the permeability moulds were collected. After collection of leachate samples, 1.5ml of concentrated Nitric acid per litre of sample was added to the leachate samples immediately to acidify the samples. The leachate samples were analyzed for the metals Ni, Cr and Pb by an Atomic

Absorption Spectrophotometer (make: AA 200, Perkin Elmer).

III. Results and Discussion

The optimum moisture content and maximum dry density values for fly ash, mine overburden and all the mixes are reported in Table III. The maximum dry density of flyash is lower than that of mine overburden as flyash is non-cohesive in nature.

TABLE III
Maximum dry density and Optimum moisture content values

Mix	MDD (kg/m^3)	OMC (%)
Fly ash	1396	20.06
Mine overburden	2040	8.15
15%FA+85%O/B	1965	8.77
25%FA+75%O/B	1872	10.8
35%FA+65%O/B	1832	11.56
(15%FA+85%O/B)+3%L	1841	13.2
(15%FA+85%O/B)+6%L	1833	12.81
(25%FA+75%O/B)+3%L	1775	12.2
(25%FA+75%O/B)+6%L	1766	12.21
(35%FA+65%O/B)+3%L	1736	13.8
(35%FA+65%O/B)+6%L	1715	14.2

Note: MDD = Maximum dry density, OMC = Optimum moisture content, FA = Fly ash, O/B = Overburden, L = Lime.

A. *California bearing ratio behaviour*

The CBR values of fly ash, overburden and lime mixtures increased with increasing curing period (Fig. 1).

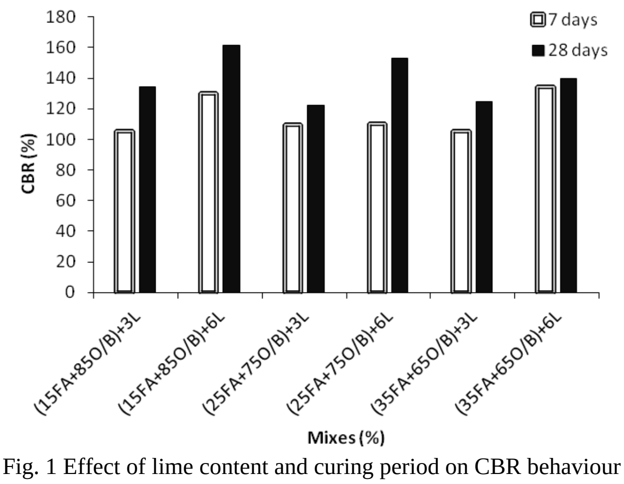

Fig. 1 Effect of lime content and curing period on CBR behaviour of the mixtures

The CBR values continued the increasing trend at 7 days cured samples that vary from 105% to 134% and at 28 days cured samples that vary from 122% to 161%. It is observed that the mixture containing 15% fly ash and 85% overburden with 6% lime exhibited maximum CBR value as compared to that of other mixtures at 28 days curing. The percentage gain in CBR values vary from 4% to 39% at 7 to 28 days curing. Similar increases in strength with increasing curing period were reported elsewhere [17]–[18].

B. *Microstructural behaviour*

SEM is a powerful analytical technique for the evaluation of particulate matter. Scanning electron microscope uses a beam of energetic electrons to examine objects on a very fine scale. It is capable of performing analyses of selected point locations on the sample. Scanning electron micrographs for the samples of all the mixtures cured for 28 days are shown in Fig. 2.

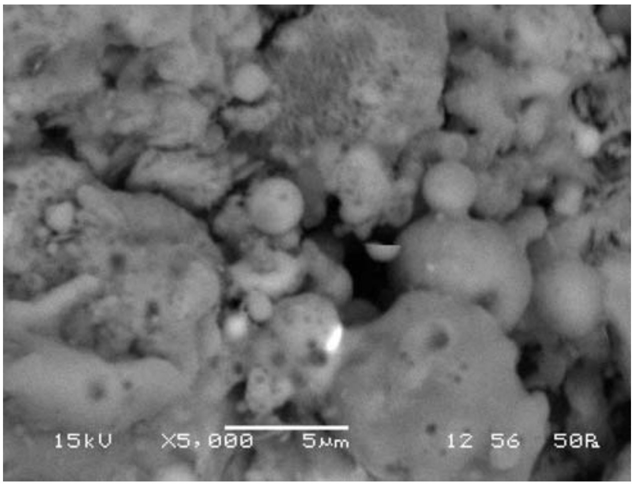

Fig. 2(a) SEM photograph of (15FA+85O/B)+3L

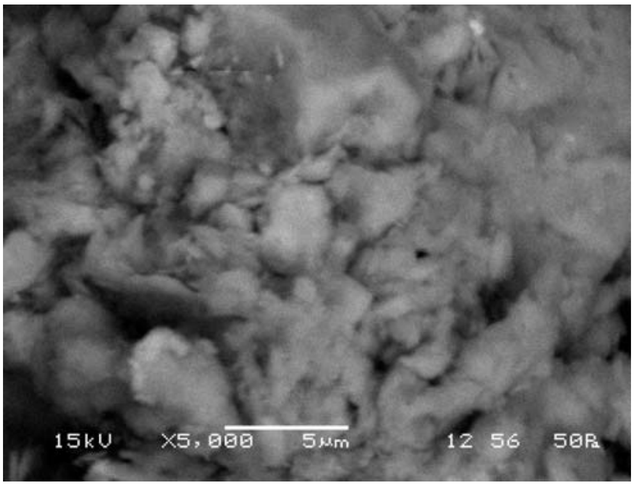

Fig. 2(b) SEM photograph of (15FA+85O/B)+6L

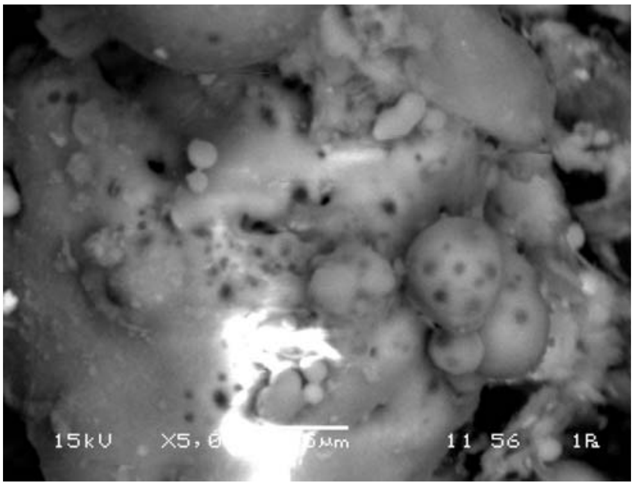

Fig. 2(c) SEM photograph of (25FA+75O/B)+3L

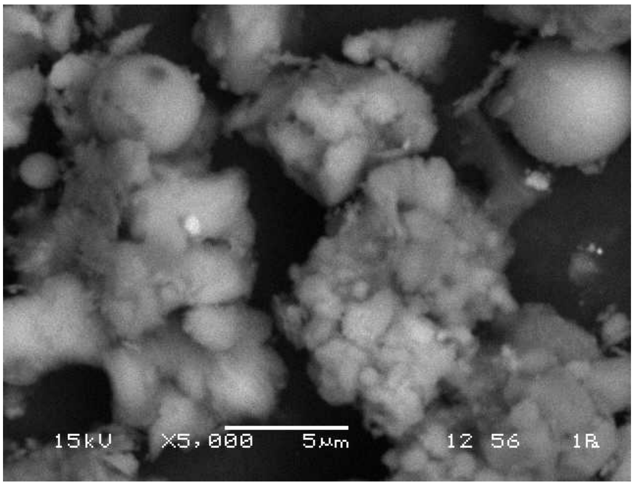

Fig. 2(d) SEM photograph of (25FA+75O/B)+6L

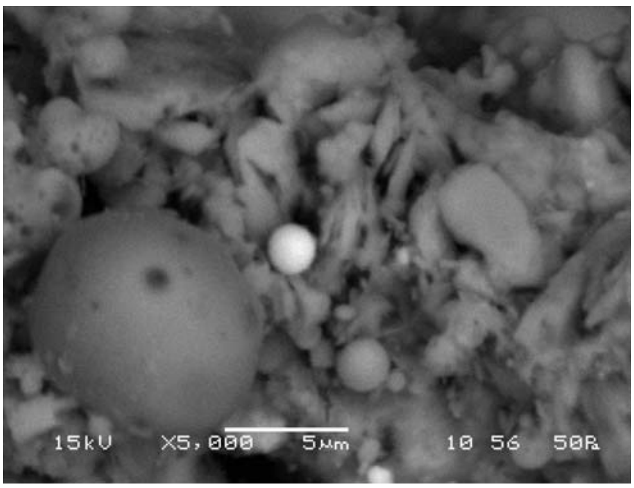

Fig. 2(e) SEM photograph of (35FA+65O/B)+3L

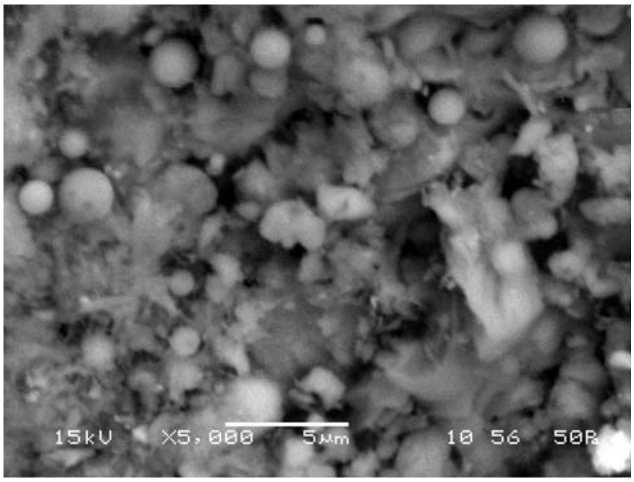

Fig. 2(f) SEM photograph of (35FA+65O/B)+6L

It is observed from micro-analyses that the new cementitious compounds such as calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH) were formed around fly ash and overburden particles as a result of the pozzolanic reaction at 28 days curing. These hydration products filled the pore spaces and maintained a bond between fly ash spheres and overburden particles. It confirms that increase in lime content produces a densified interlocking network and the strength development is also dependent on the amount of hydration products as well as their interlocking mechanisms [19]. The strength development is influenced by the cementitious gel produced and consequently by the amount of lime consumed [20].

C. Leaching behaviour

The concentration of metals in leachate on the 7th day of flow analyzed for Ni, Cr and Pb are presented in Table IV.

TABLE IV
LEACHATE CONCENTRATIONS (PPM) ON 7TH DAY OF FLOW FOR 7 DAYS CURING PERIOD

Mix	Metal		
	Ni	Cr	Pb
Threshold limits	2.0	5.0	5.0
(15%FA+85%O/B)+3%L	0.028	0.415	0.208
(15%FA+85%O/B)+6%L	0.204	0.64	1.681
(25%FA+75%O/B)+3%L	0.231	0.656	ND
(25%FA+75%O/B)+6%L	0.116	0.735	1.385
(35%FA+65%O/B)+3%L	0.071	0.125	0.095
(35%FA+65%O/B)+6%L	0.014	0.416	0.3

Note: ND: Not detected

Threshold value for maximum contaminant level is considered as 100 times the allowable limit reported elsewhere [14], [21]. It is observed that the leachate effluents contain Ni, Cr and Pb between 0.01 to 1.6 ppm. These concentrations were below the threshold limits. There were suggestions that the concentration of heavy metals in the leachate effluent emanating from the hydraulic conductivity specimens of stabilized fly ash with higher proportions of lime or lime with gypsum were below threshold limits acceptable for contaminants flowing into ground water [14].

IV. CONCLUSION

The results indicated that California bearing ratio of all the mixes increased with addition of lime and curing period. Mine overburden mixed with 15% fly ash and 6% lime exhibited maximum strength and CBR values than other mixtures at 28 days curing. The morphology of all the mixtures showed the formation of hydrated gel at 28 days curing. The voids between the particles were filled by growing hydrates with curing time. Microanalysis confirmed the formation of new cementitious compounds such as calcium silicate hydrate (CSH) gel and calcium aluminate hydrate (CAH) gel which leads to increase in bearing ratio of the material over time. It is observed from the leachate analysis that the concentration of Ni, Cr and Pb in the leachate effluents were below threshold limits and acceptable for contaminants flowing into ground water.

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