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Geotechnical characterization of fly ash composites for backfilling mine voids

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Abstract. Backfilling of mine voids is mandatory to avoid subsequent ground stability problems in the form of subsidence. River sand and mill tailings have been widely used since a long time as backfilling materials. However, with a strict regulation banning river sand mining in India, research for developing alternative engineering materials substituting sand has gained importance. In the present study four fly ash composite materials (FCMs) was developed from the fly ash obtained from a captive thermal unit of Rourkela Steel Plant (RSP). The main constituent of the composite were fly ash, lime and gypsum. Detailed physical, and engineering properties were determined for the FCMs. Significant increases in the compressive strength were obtained after 56 days of curing time. A detailed SEM studies was undertaken to account for the increase in strength with time. The fly ash composite developed from RSP has potential to be used as substitute to sand for backfilling the mine voids.

Key words. backfill, fly ash composites, gypsum, lime, mine voids.

1. Introduction

Typically large underground space left out after mining operations have been creating various types of ground stability problems in many mining area in India. Subsidence is a very common phenomenon in many coal mining areas. Most of the subsidence problems are reported to have occurred suddenly and those often remain as serious threats to the subsequent development of townships. Backfilling or sand stowing has been the method followed for decades to counter the ground subsidence as well as to improve pillar recovery. Some of the common types of materials used for backfilling are waste rock, mill tailings, quarried rock, sand and gravel. It is often observed that sand or mill tailings as backfill material remain loose and merely serve as a temporary working platform rather than offering any lateral stress on the opening walls to improve the stability situation (Srivastava, 1995, unpublished). Also there are reports that where cemented backfills are used, the cost of backfill tend to be 10–20% of the total operating cost of the mine and cement represents upto 75% of that cost (Grice, 1998). Notwithstanding these observations, the unavailability of

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river sand for backfilling mine voids is a major concern and mine operators and planners around the world are emphasizing on developing an alternative material to sand. In India regulatory restrictions on sand mining for backfilling are due to be implemented very soon (Kumar et al., 2003).

Fly ash is produced in large quantities in India from the thermal power plants. There are numerous successful case histories on the utilization of fly ash either alone or mixed with lime, gypsum or both. Typically fly ash has been used for soil stabilization (Chu et al., 1955), as embankment material (Raymond, 1961), structural fill (DiGioia and Nuzzo, 1972), for injection grouting (Joshi et al., 1981), as a replacement to cement (Gopalan and Haque, 1986; Xu and Sarkar, 1994), in coastal land reclamation (Kim and Chun, 1994) and in roads and embankments (Kumar, 2003). Maser et al. (1975) reported successful studies on fly ash–cement mixture for subsidence control. Fawconnier and Korsten (1982) reported that the use of pulverized fly ash filling had effectively stabilized the coal pillars reducing the risk of pillar failure in areas of low safety factor. Galvin and Wagner (1982) observed improved strata control using fly ash fill. Petulanas (1988) also reported the use of high volume fly ash for subsidence control. Palariski (1993) reported the use of fly ash, mill tailings, rock and binding agents to make consolidated backfill material to improve extraction percentage in coal mines. Kumar et al. (2003) have reported the use of pond ash (a mixture of fly ash and bottom ash) of grain size between −75 and +20 mesh with equal percentage of water (by weight) for underground stowing of a coal mine. The pond ash water mixture with additives exhibited 100% settlement of solids within 30 min of placement. The settlement time further reduced with an increase in additive concentration. The additives also improved the percolation rate of the mixture with little adverse affect on mine water. The barricades placed to arrest the fill material showed negligible load due to the placement signifying self-standing behavior of the mixture. Using paste backfilling technology containing 65–70% solids of Coal Combustion Byproducts (CCBs) and coal processing waste, Chugh et al. (2001) reported that 9,200 tons of backfill were injected underground successfully.

In the present investigation fly ash was mixed with additives as lime and gypsum at 15% to 20% and 0% to 5% by weight, respectively to form a fly ash composite material to develop an alternative material for backfilling mine voids. The primary objective has been to achieve setting properties to the backfill material such that it develops compressive strength to offers lateral stress to the surrounding walls and pillars and effect in increasing the stability of the opening. With the objective in background an extensive laboratory investigation have been undertaken to examine the feasibility of developing a strong alternative material with the fly ash composite.

2. Preparation of Fly Ash Composite Material (FCM)

Fly ash has been a major constituent of the composite material used in the present investigation. The fly ash was collected from a thermal power unit of Rourkela Steel

Plant (RSP) in dry state by electrostatic precipitator (ESP). The ash was in loose state with average water content less than 4%. The fly ash is of ASTM class F type, and was chosen for its low lime content as well as its availability in abundance compared to the class C type. Further, it would also facilitate the evaluation of adding admixtures for enhancing the pozzolanic activity. On the basis of the literature reviewing, two different lime proportions 15% and 20% of fly ash (by weight) were selected. Similarly, percentages of gypsum were 0 and 5% of fly ash (by weight). This gave four different composites such as 15% lime (L)–0% gypsum (G), 15%L–5%G, 20%L–0%G and 20%L–5%G with fly ash as base material. The additives selected were commercially available superior grade quick lime and analytical grade, anhydrous gypsum. The addition of lime enhances the pozzolanic reactivity of fly ash containing insufficient free lime required for pozzolanic reaction with its reactive silica. Analytical quality gypsum was chosen to avoid the interference of impurities because impurities may retard the initial hydration process. Those hydrated gypsum commonly available in field would reduce the optimum moisture content without affecting the dry density and hence strength. Depending on the sample dimension, required quantities of fly ash, lime and gypsum were mixed thoroughly in dry state. All the samples were prepared at maximum dry density and Optimum Moisture Content (OMC) of the respective mixes as obtained from modified Standard Proctor tests. After dry mixing water quantity corresponding to OMC was spread over the dry mix and thoroughly mixed by hand. Then it was kept inside a polythene bag for 1 h for moisture homogenization. The process was again repeated after 1 h for better homogenization before being used to prepare the core samples for engineering properties.

3. Experimental Investigation

The physical properties such as chemical composition, grain size analysis, particle size distribution, specific gravity and specific surface area were determined following the procedures of Indian standards as per IS Codes (1987). The grain size analysis of the untreated fly ash was carried out as per IS:2720 Part-4 conforming to ASTM D 422 by dry sieve analysis and followed by hydrometer analysis method for the fractions passing 75 μ sieve. The engineering properties such as compressive strength, elastic modulus, cohesion, angles of internal friction of the fly ash composite were determined for each composition of FCM. The samples were cast to NX size core i.e., 54 mm diameter and 108 mm length for compressive strength tests. Samples were prepared with uniform tamping and weights of the prepared samples were checked against the maximum dry density required as already mentioned. Those samples that did not have the weight within ±1% of the required weight were rejected. These samples were taken out of mould after 24 h and kept in moist proof containers that were in turn placed inside humidity control chambers where the temperature was maintained at about 30 °C ±1%. The reported values were the average of about 10 representative samples tested for each parameter. In all total about 800 tests were conducted.

4. Results and Discussion

4.1. PHYSICAL PROPERTIES

The chemical composition of the fly ash is given in Table 1 and Table 2 gives the physical characteristics of the fly ash. The grain size distribution of the fly ash is given in Figure 1. It is observed that more than 90% of fly ash particles are between 0 and 50 μm that indicates a favorable trait for pozzolanic reaction (Mehta, 1985), particles range from fine sand to silt sizes. The size distribution of the fill material has a fundamental bearing on the fill density that can be achieved. The effective size of the particle corresponding to 10% finer is 1.6 μm. The size of the particle corresponding to 60% finer is 8.4 μm. The coefficient of uniformity ($C_u = D_{60}/D_{10}$) and the coefficient of gradation ($C_c = D_{60}/D_{10}$) are 5.4 and 1.7, respectively. It implies that the chosen fly ash possess good grading quality and amenable to be a suitable fill material.

The specific gravity of the fly ash was determined according to IS:2727 guidelines by Le-Chartelier method with kerosene oil. The average specific gravity value obtained is 2.54. The specific surface area of the fly ash was determined by Blaine air permeability method, as per IS:1727–1967 guidelines and the corresponding value is 1.1049 m^2/cc. It is known that silica–lime reaction is pH dependent. The higher the pH, the better is the solubility of silica as well as the lime–silica reaction in producing pozzolanic cementitious compounds.

The pH of the system increases if addition of lime is in excess of lime required for the silica to react. Addition of lime increases the pH of the composites. But the addition of lime in excess of lime required for the reactions with reactive silica makes

Table 1. Chemical characteristics of fly ash

Constituents	Percentage (%)	Constituents	Percentage (%)
Carbon	2.10	P_2O_5	0.17
Volatile matter	0.147	SO_3	0.24
Fe_2O_3	8.83	K_2O	0.79
MgO	0.84	CaO	1.11
Al_2O_3	27.73	Na_2	0.14
SiO_2	58.9	TiO_2	2.09

Table 2. Physical characteristics of fly ash

Parameters	Value	Parameters	Value
Color	Light gray	Liquid limit (%)	40.89
Dry density (kg/m^3)	1380	Plastic limit (%)	Non-plastic
Optimum moisture content (%)	38.7	Specific gravity	2.54
Permeability (m/s)	$(3.5–3.7)\times10^{-6}$		

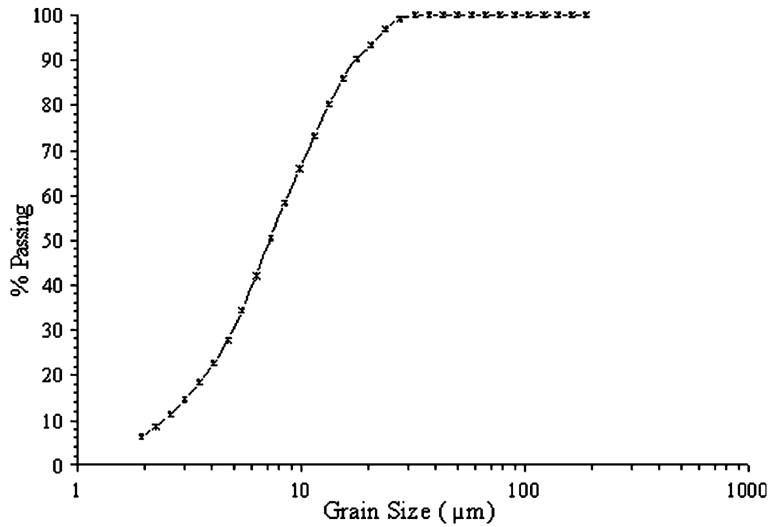

Figure 1. Grain size analysis of the untreated fly ash.

the pH constant as the solution becomes saturated with the lime content (Sivapullaiah et al., 1995). The constant pH of the solution is an indicator of saturation of the silica–lime reaction or pozzolanic activity that is accompanied by mechanical strength gain. The pH of the raw fly ash was determined using Systronics scale pH meter, accurate up to ± 0.02 units. The measurement was carried out as per the procedure outline by Jackson (1958) at room temperature. The pH of the raw fly ash was 7.22. It was observed that the pH increased by more than 55% for all the fly ash composites (Figure 2). It showed that samples were at the threshold of initiation of

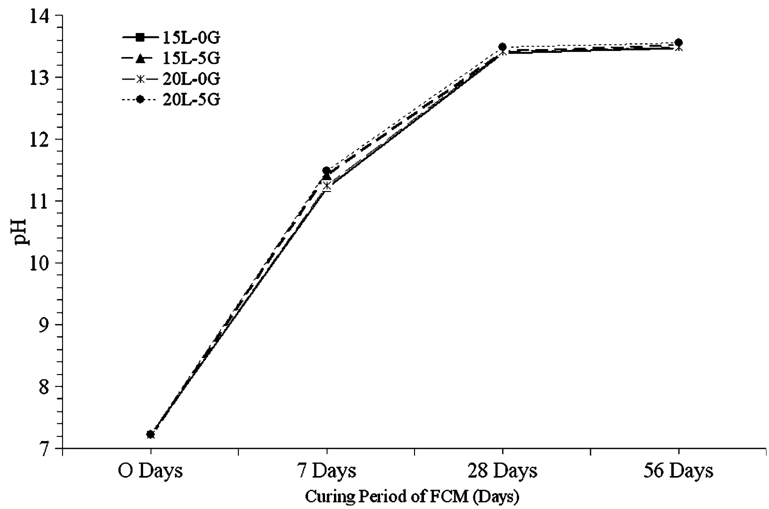

Figure 2. Change of pH over curing time for FCMs.

pozzolanic activity. At 28 days curing period the pH values were more than 13.2 for all the samples conforming to the occurrence of pozzolanic reactions. The percentage gain in pH values from the values obtained at 7 days time was between 17% and 19%. However, it was also observed that pH at 56 days did not gain appreciably compared to the values at 28 days for all the fly ash composites.

Liquid limit is a measure of the water content at the transition of fly ash from plastic state to liquid state. Fly ash in contact with water forms cementitious gel that is voluminous in nature that has a good water holding capacity. Depending on the reactive silica the water holding capacity of the fly ash decreases after the optimum liquid limit it reached. The liquid limit test for the raw or untreated fly ash samples was conducted by Cone Penetration method as per IS: 2720 (Part-V) 1965 guidelines. The average value of the liquid limit found to be 40.9%.

Pure hydrated lime was added to increase the pozzolanic activity. Lime stabilization of low lime fly ash would also mitigate the dusting problem. The chemical composition of the lime by dry weight is shown in Table 3.

4.2. ENGINEERING PROPERTIES

4.2.1. *Moisture content–dry density relationship*

Compaction of particles is greatly affected by its dry density and moisture content. Up to a certain value the dry unit weight of the compacted material increased with that of the moisture content and then it decreased. Due to addition of admixtures like lime and gypsum the optimum moisture content (OMC) and maximum dry density relationship will change. So the OMC and dry density of the composites of 15% L, 15% L with 5% G, 20% L and 20% L with 5% G mixes was carried out as per IS: 2720 (Part-4) – 1980 by modified Standard Proctor Hammer Compaction Method. The compaction energy imparted through each of the test was about 2700 KN-m/m^3. The maximum dry density and optimum moisture content (OMC) for each composite type obtained is shown in Figure 3. It is observed that the moisture content and dry density relationships vary between 28% to 31% and 1.32 to 1.39, respectively. The addition of gypsum reduces the density of the composite as compared to that of without gypsum. The normalized dry density and normalized

Table 3. Chemical composition of lime

Minimum array (Acidimetric)	95.0%
Maximum limits of impurities	
Chloride (Cl)	0.1%
Sulphate (SO_4)	0.5%
Iron (Fe)	0.1%
Lead (Pb)	0.02%
Loss on ignition	10%

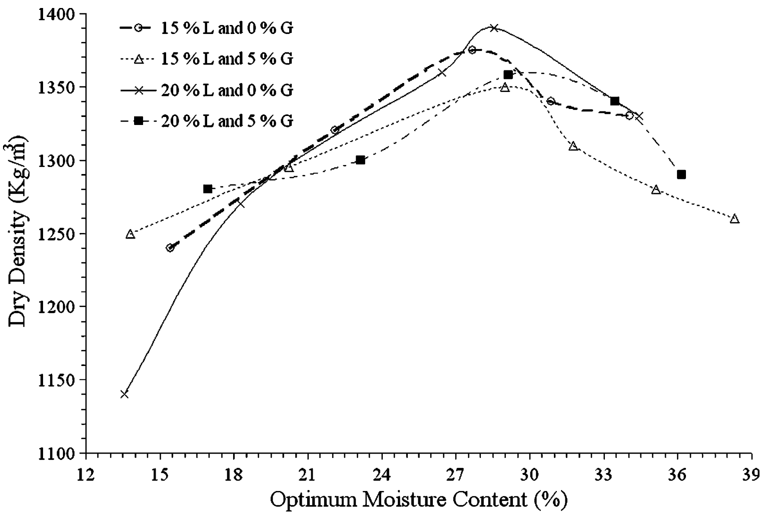

Figure 3. Optimum moisture quantity and maximum dry density relations for FCMs.

water content of the composites are shown in Table 4. In order to take care of the variation in the specific gravity of coal ashes, dry density was expressed in terms of normalized dry density with the following formula:

$$\text{Normalized Dry Density} = [(\text{Dry Density} * 2.65)/\text{Specific Gravity}].$$

4.2.2. *Unconfined compressive strength (UCS)*

Compressive strength test is a measure of the resistance of the composites to external loading. UCS tests conforming to ISRM-1972 guidelines were conducted on untreated fly ash for 7, 28 and 56 days curing period at room temperature that was

Table 4. Engineering properties of fly ash composite material (FCM)

	15%L–0%G		15%L–5%G		20%L–0%G		20%L–5%G	
	Number of days of curing							
Parameters	28	56	28	56	28	56	28	56
Compressive strength (MPa)	5.46	8.51	8.08	11.79	6.88	10.04	8.51	12.22
Young's modulus (MPa)	293.2	320.3	316.9	349.2	304.5	331.2	321.2	348.7
Poisson's ratio (μ)	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35
Cohesion (c) (MPa)	1.09	1.16	1.17	1.26	1.40	1.54	1.43	1.59
Angle of internal friction (θ)	26°	27.7°	29.6°	30.2°	30.0°	31.2°	30.2°	31.6°
Normalized dry density (kg/m^3)	1812.8		1779.8		1832.5		1790.3	
Normalized water content (%)	20.99		21.98		21.64		22.09	

about 30 ± 3 °C. Similar tests were also conducted at the end of 28 and 56 days of curing time for the fly ash composites. As expected the 7 days cured strength of raw fly ash was very low with only 0.076 MPa and a marginal increase in corresponding values were observed for both 28 and 56 days of curing periods. Those results are not reported here. The strength of fly ash composite changed with addition of lime, gypsum and temperature and curing period. The 7 days strength of fly ash composites substantially improved with addition of additives.

The strength value for composite with 15% lime was 1.9 MPa and the same for 20% lime was 2.24 MPa. Addition of 5% lime increased the strength by about 20% for both cases. But as the curing period increased to 28 days the strength showed dramatic improvement. The addition of 15% lime improved the strength of the composite to 5.45 MPa for a 28 days curing period that is about 185% more than that for 7 days period. But the strength gain showed reduced trend after 28 days. It was only 56% for composite with 15% lime in another 28 days. Further addition of 5% lime, the gain percentage was 205% over 7 days period that reduced to 46% for 56 days. Addition of 5% gypsum though increased the strength value it showed reduced gain percentages for both cases (Figure 4). These observations confirm that addition of lime in excess to fly ash composites may not be beneficial or may even have deleterious effects (Gray and Lin, 1972; Yudhvir and Homjo, 1991; Sivapullaiah et al., 1995).

4.2.3. *Unconfined tensile strength*

Tensile strength is a measure of resistance of the composite to external tensile forces. Brazilian indirect tensile strength tests, as suggested in IS: 100082-1981, were carried

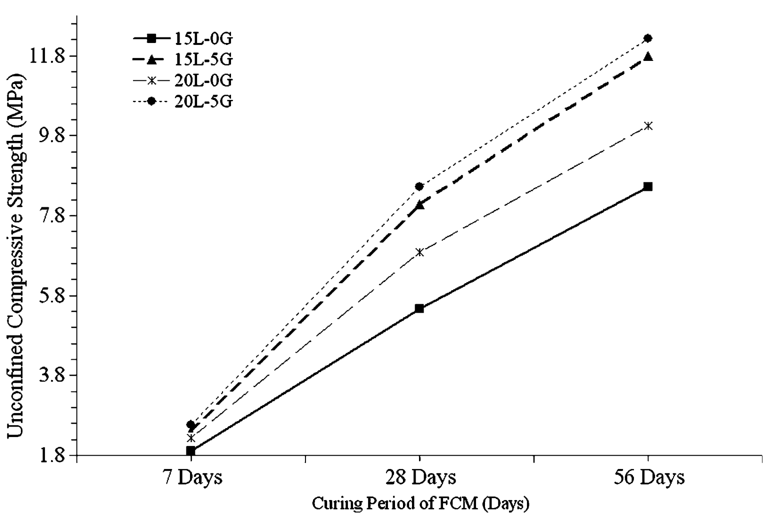

Figure 4. Change of compressive strengths of FCM over curing period.

out to determine the tensile strength of the fly ash composites in the same universal testing machine used to find the compressive strength. The samples for the test measured 54 mm in diameter and 27 mm in thickness were cut from the specimen prepared for compressive strength tests. The samples were loaded along the diametrical axis as followed in the method. It was observed that the tensile strength of samples with 15% and 20% lime were 0.058 and 0.072 MPa, respectively (Figure 5), about 3% of the respective compressive strengths at 7 days curing time. Similar results were also observed for samples without gypsum at 28 and 56 days curing periods. The tensile strengths were between 10% and 11.75% of the compressive strength at respective curing periods. But with the addition of 5% gypsum the tensile strength improved for all the curing period though at 7 days it was less than 7% of its compressive strength. The Brazilian tensile strengths almost doubled between 28 and 56 days curing periods that were more than 15% and 16% with 15% and 20% lime composites, respectively. The test results conform those observation made by Ghosh (1996) that a minimum period of 45 days should be allowed to attain considerable tensile strength.

4.2.4. *Slake durability index*

The slake durability test is a measure of effect of weathering on the material and its durability. The test (IS: 10050, 1981) is conducted to assess the resistance offered by a rock or rock-like (in this study fly ash composite) samples to weaken and disintegrate when subjected to two standard cycles of drying and cooling. In the present investigations slake durability tests were conducted on each type of composite samples cured for 28 and 56 days. The results of such tests are reported in Table 5. It is

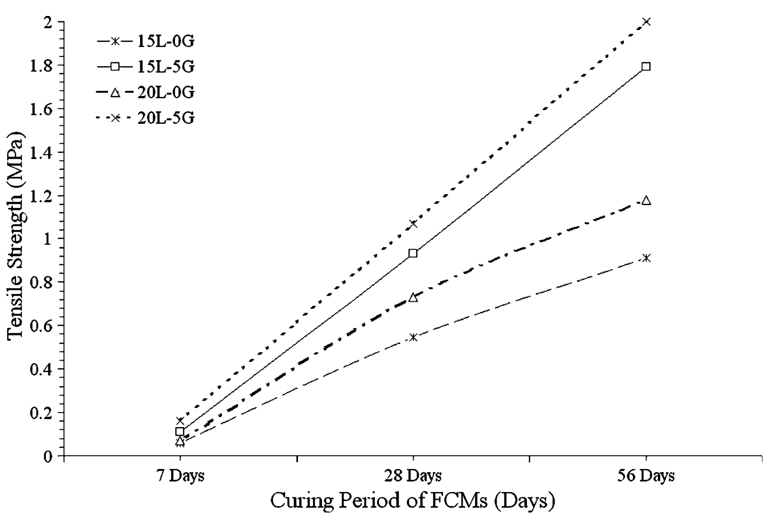

Figure 5. Brazilian tensile strength of FCMs.

Table 5. Slake durability index (%) for the fly ash composites

	28 Days curing		56 Days curing	
Fly ash composite type	1st Cycle	2nd Cycle	1st Cycle	2nd Cycle
15L–0G	96.15	82.3	96.9	84.2
15L–5G	97.1	86.2	97.8	87.1
20L–0G	96.5	85.3	97.4	86.5
20L–5G	98.16	92.1	99	93

observed from the tests that the slake durability index for fly ash composite without gypsum vary between 96% to 97% during 1st cycle for 28 days curing time and increase marginally for 56 days curing period. The results show considerable increase for both curing periods when 5% gypsum was added. It indicates that each type of fly ash composite posses medium to medium high durability as per classifications (Gamble, 1971).

4.3. MICRO STRUCTURAL ANALYSIS OF FLY ASH COMPOSITES

The raw fly ash and each type of composites were examined by scanning electron microscope to investigate the micro structural development of the fly ash composites. The JEOL JSM-5800 scanning electron microscope coupled with an OXFORD ISIS 300 energy dispersive X-ray micro-analyzer was used for the purpose. The samples were initially coated with carbon to maintain conductivity. The examination of the fly ash before and after lime–gypsum treatment reveals that the glassy portions of the spheres are preferentially attacked by lime or lime and gypsum mixtures. The SEM image of untreated fly ash and that of each composite at the end of curing periods are shown in Figure 6a–h, respectively. The scanning electron monographs taken after 28 days curing show that the hardened paste microstructure has already started to develop with the ash particle in different stages of pozzolanic activity. It may be explained by the fact that during early period of curing thin layers of hydration products are formed on the surface of the spherical ash particles due to pozzolanic reactions. The inner part of the thin layer consists of denser mass and the outer layer of the particle is of fine fibrous matter. The particles show evidence of erosion and etching on its surface. Some formation of ettringite rods was also observed. It may be inferred that at early stages the fly ash particles served as nucleation sites for hydration and pozzolanic reaction products (C–S–H, CH and ettringite). The above observation compares favorably with those of Lav and Lav (2000). The images taken after 56 days curing show the microstructure comprises of ash particles in a state of more advanced hydration. The calcium–silicate–hydrate (C–S–H) or calcium–aluminates–hydrate (C–A–H) gel like mass fills the space between particles implying that the ash particles have reacted considerably. It is also observed that the ash particles have reacted considerably leaving some multiple spheres. These observations are in conformation with similar observations made by Xu and Sarkar (1994).

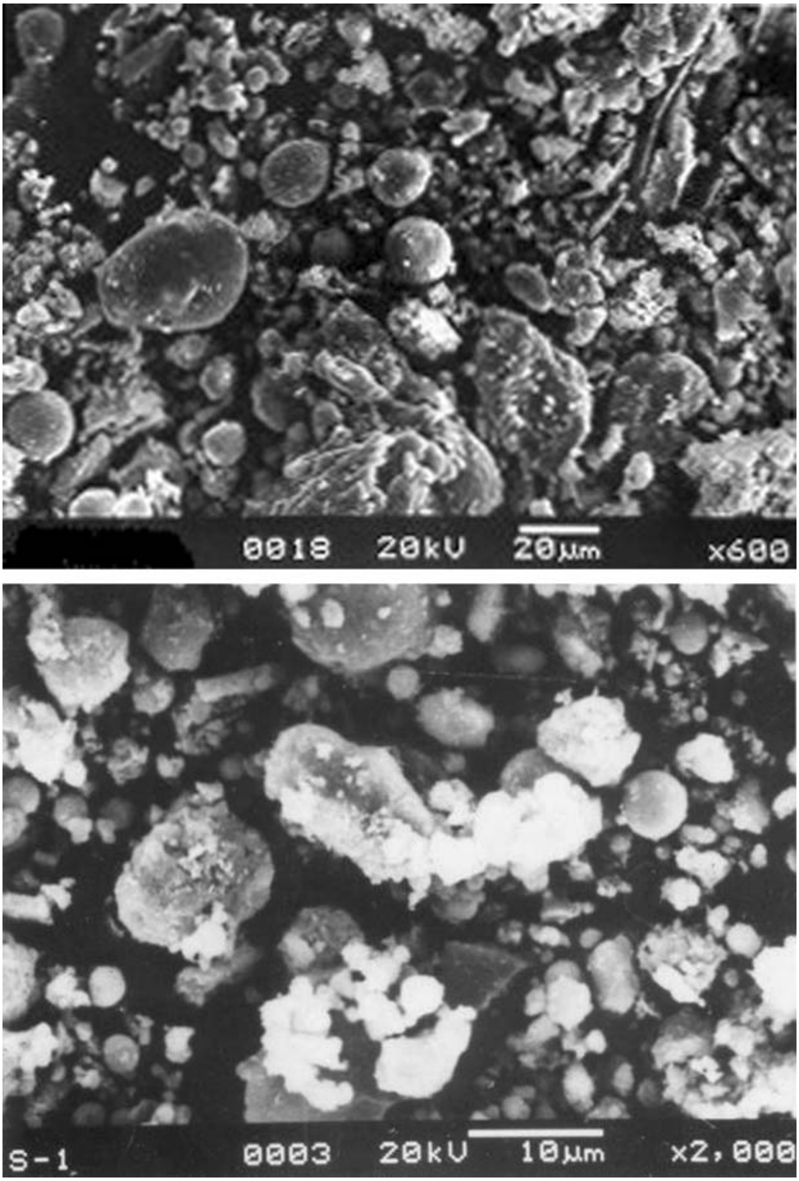

Figure 6. SEM image of untreated fly ash. (a) SEM image of FCM (15% L) at 28 days curing period. (b) SEM image of FCM (15% L) at 56 days curing period. (c) SEM image of FCM (15%L–5%G) at 28 days curing period. (d) SEM image of FCM (15%L–5%G) at 56 days curing period. (e) SEM image of FCM (20% L) at 28 days curing period. (f) SEM image of FCM (20% L) at 56 days curing period. (g) SEM image of FCM (20%L–5%G) at 28 days curing period. (h) SEM image of FCM (20%L–5%G) at 56 days curing period.

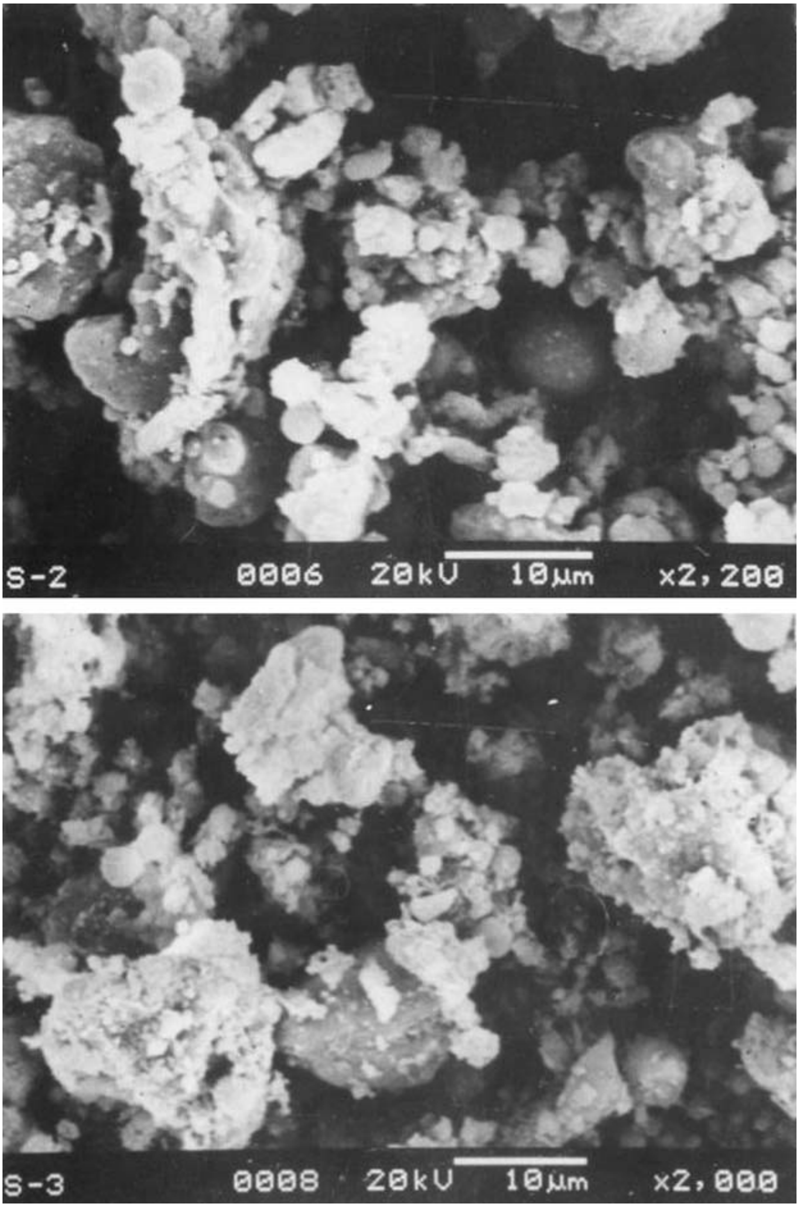

Figure 6. Continued.

At longer curing period, that is at 56 days, the micrographs show dense gel-like mass covering all the ash particles completely and filling up the inter-particle spaces. The grain boundaries appear blurred and the dense gel acting as a binding substance is seen evenly distributed to form compact structure. It creates more contacts and greater cohesion in the fly ash composite mass that in turn contributes to higher

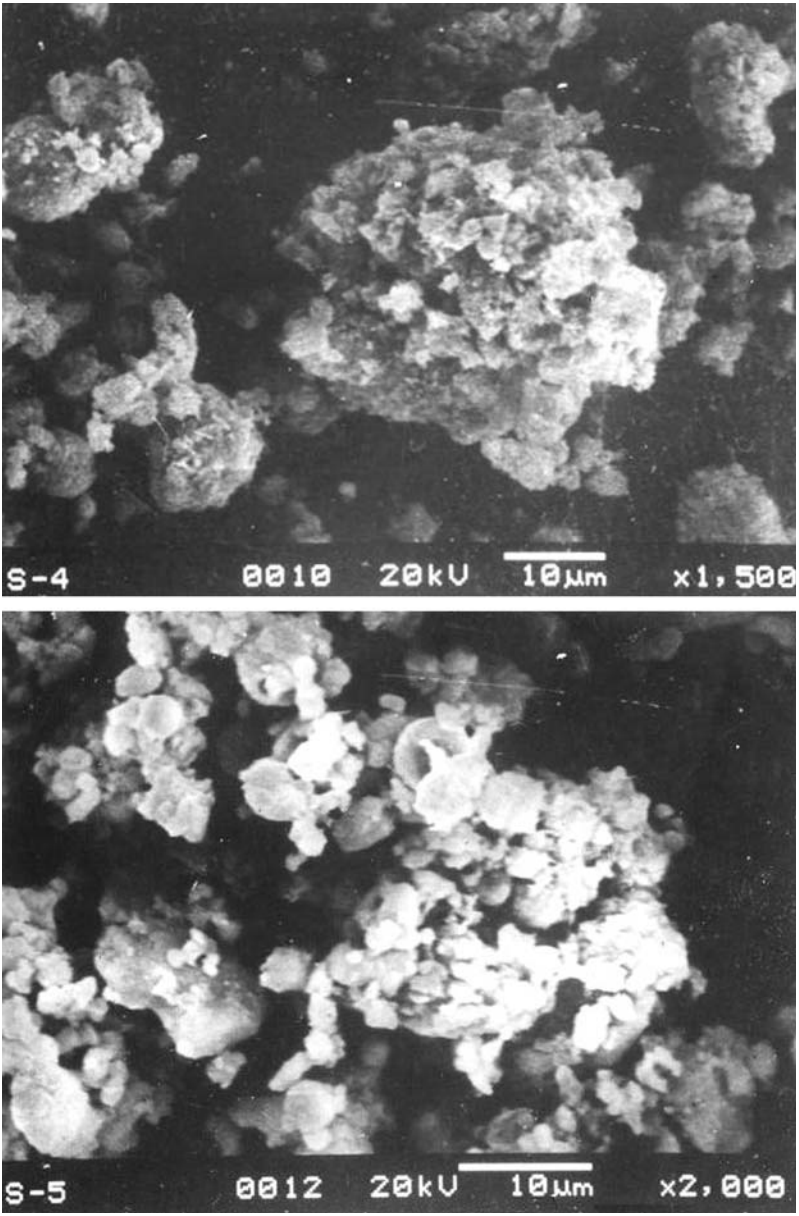

Figure 6. Continued.

densities and greater strength. The fly ash composites have been analyzed for qualitative and semi-quantitative information about the microchemistry of the material. The quantitative analysis indicates little affect due to presence of contaminating elements in the FCM as arsenic, barium, etc. The normalized results of energy dispersive X-ray microanalysis are shown in the Table 6.

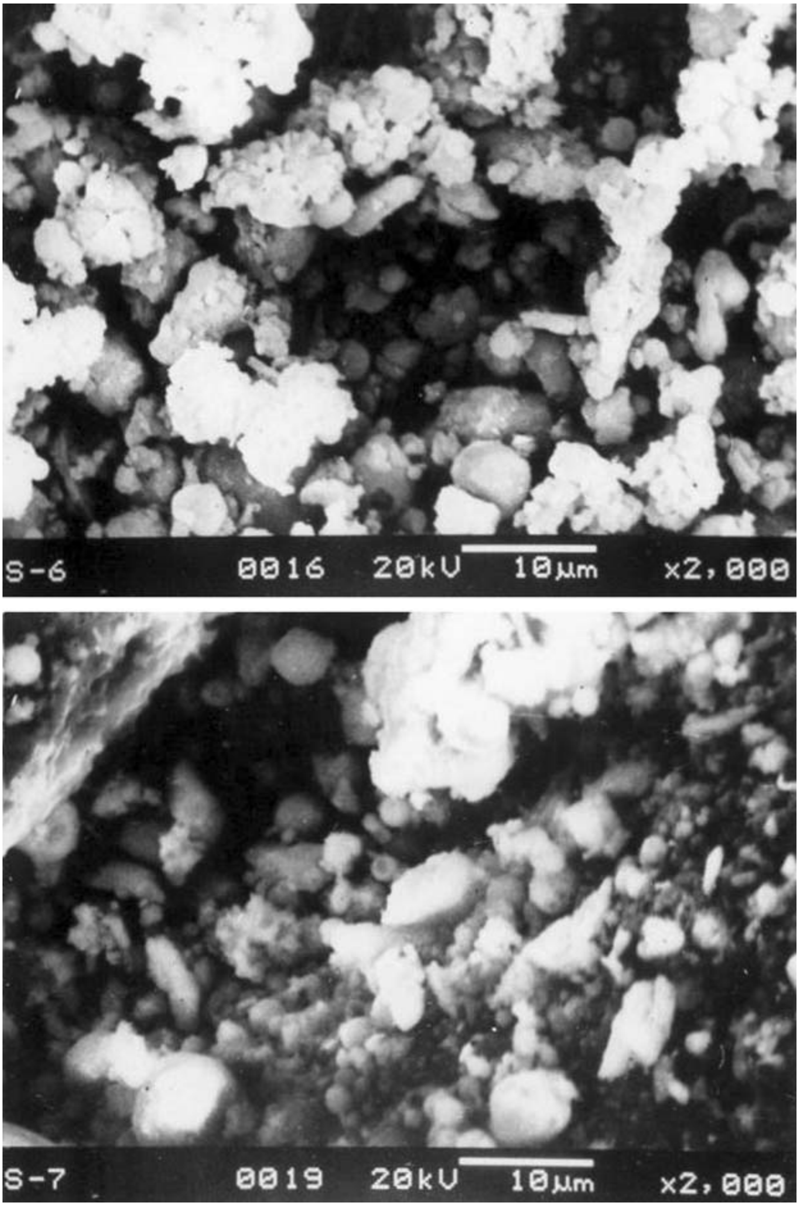

Figure 6. Continued.

5. Conclusion

The loose sand as a backfill material merely occupied the underground space created by mining operation and the studies in the past also have indicted that no lateral stresses are developed due to sand filling to assist the stability of the opening

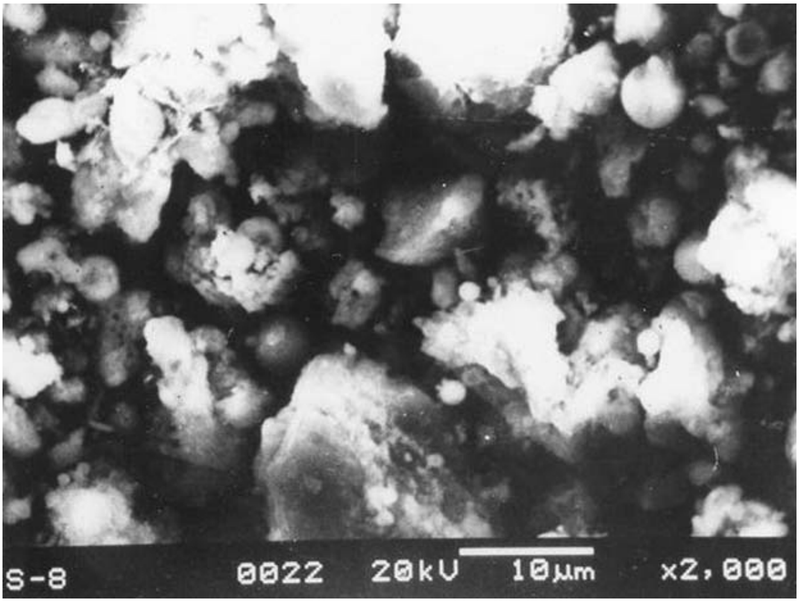

Figure 6. Continued.

Table 6. SEM quantitative results of fly ash composites

	Fly ash composite types (by lime and gypsum percentages, respectively)							
	15–0	15–0	15–5	15–5	20–0	20–0	20–5	20–5
	Number of days of curing							
Elements	28	56	28	56	28	56	28	56
Na	2.33	1.57	0.24	1.10		0.68	5.53	2.55
Mg	1.55	1.49		1.13		1.02	3.43	3.03
Al	24.65	27.07	25.72	23.37	26.24	24.89	16.92	23.35
Si	47.97	57.17	48.33	44.90	52.99	50.69	32.23	37.21
K	0.39	1.11	1.77	0.81	1.68	1.70	2.44	1.59
Ca	18.96	7.79	17.48	16.65	16.24	18.05	24.20	11.26
Ti	0.92	0.84	1.17	2.47	0.06	2.31	0.82	1.29
Fe	3.23	1.16		2.68	2.78		3.97	14.34
S			5.29	5.92		0.67		0.27
Mo				0.96			7.65	1.35
Cu							0.16	0.84
Zn							2.65	
P								1.73
Ba								1.19
Total	100	100	100	100	100	100	100	100

(Srivastava, 1995, unpublished). The other major concern is the non-availability of sand for backfilling the underground mine voids. In view of these observations one of the objectives of the present investigation was to study both physical attributes as well as the engineering properties of the developed fly ash composite material (FCM) as an alternative to sand as a backfilling material. With the promise of the paste backfill technology, the transportation of fly ash is not a serious economic handicap. The fly ash composites developed with addition of lime and gypsum have significantly contributed to the strength characteristics of the composite. The curing time also indicated an increase in the strength of the composite. The SEM studies indicate that at longer curing period that is at 56 days, dense gel-like mass covered all the ash particles filling up the inter-particle spaces completely which is the reason for increased strength without any adverse effect on ground water quality. The major conclusion for the study are that class F type fly ash from thermal power unit of Rourkela Steel Plant has greater potential to be developed into a strong engineering material with the addition of lime and gypsum. The fly ash composite exhibited favorable characteristics to substitute sand as backfilling material.

Acknowledgements

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