Geotechnical Investigation of Fly Ash Composite Materials for Filling Mine Voids

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ABSTRACT

Of the 750 million tons of fly ash that are produced annually worldwide, only a small portion e.g., 40% to 50% of the same is being used for productive purposes, such as an additive or stabilizer in cement, bricks, embankments, etc. The remaining amount of fly ash produced annually must either be disposed of in controlled landfills/ mine fills or waste containment facilities or stockpiled for future use or disposal. As a result of the cost associated with disposing of these vast quantities of fly ash, a significant economic incentive exists for developing new and innovative, yet environmentally safe applications for the utilization of fly ash. The main aim of the present investigation was designed to develop an engineered backfill material to be placed in mine voids using fly ash as the major component. An experimental setup was designed and fly ash samples from 7 numbers of thermal power plants situated in different parts of the country were collected. Investigation into detailed physical, chemical, morphological, and mineralogical characterizations have been carried out to choose the most favorable fly ash composite material for filling mining voids. Lime was selected to enhance the strength characteristics of fly ash composite materials. A lime pH optimization study was carried out to find the optimum quantity of lime that was added to the fly ash to increase the in-place strength of fly ash composite materials. UCS, Brazilian Tensile strength, and Triaxial tests were conducted at varying curing periods i.e., at 0 days, 7 days, 14 days, 28 days, and 56 days. Ultrasonic pulse velocity was measured, and microstructural analysis was carried out to examine the strength behavior of the developed composite materials. FTIR study was carried out to find the effect of lime addition on strength parameters. The SEM images were also obtained to study the ettringite formation in the fly ash composite materials. From the results of this study, it is inferred that there is substantial strength gain with the curing period. Empirical equations are developed to predict the flow and strength behavior of the selected and optimized fly ash composite materials.

Keywords: fly ash composite material, strength parameters, compressive strength, tensile strength, mine filling

1. Introduction

The engineering properties of a material depend largely on the composition of the material. There exists wide variation in the composition of fly ash depending on coal types, types of furnaces, temperature, collection technique adopted, etc. (Pandian *et al.,* 1995). The geotechnical properties of the developed composite materials were determined as per established methods. All the results of the current investigation and their corresponding analyses have been presented in different sections as mentioned below. The engineering properties of a material such as UCS, Brazilian tensile strength, etc. are dependent on the moisture content and dry density. Typically, the higher the compaction the better is its geotechnical characteristics. Hence it is necessary to achieve the desired degree of compaction to meet the expected properties (Nicholson *et al.,* 1994). Compaction is the process of increasing the density of the material by the application of mechanical energy such as tamping, rolling, and vibration. It is achieved by forcing the particles closer with a reduction in air voids. Optimum moisture content (OMC) is the moisture content at which compacted material reaches the maximum dry density of solid particles.

2. Materials

The following materials were used for this study:

i. Fly ash
ii. Lime

3. Methods

Geotechnical properties of developed fly ash composite materials (FCMs): The developed composite materials were subjected to various engineering tests such as Compaction behaviors, UCS Test, Brazilian Tensile Strength Test, Ultrasonic Pulse Velocity Test, Micro Structural Analyses, and X-Ray Diffraction (XRD) analysis, the results of which are presented in the following sub-sections.

4. Results and Discussions

4.1. Compaction Characteristics

The compaction characteristics of the developed fly ash composite materials were carried out to determine the optimum moisture content and maximum dry density the results of which are presented in Figure 1. As the water content was increased, the dry density of the specimen increased. The optimum MDD of the developed composite materials was found to be 1443 Kg/m^3 (Table 1) and the corresponding value for OMC was 12.51%.

Table 1 Engineering properties of FCM

Moisture Content (%)	Dry Density (Kg/m^3)
7.37	1260
9.97	1346
12.51	1443
16.11	1341
20.85	1254

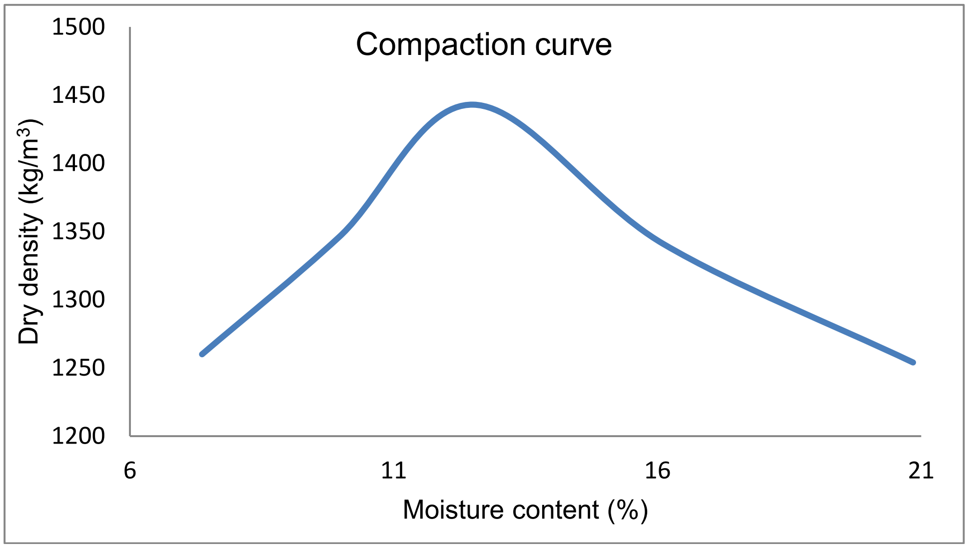

Figure 1: Compaction curve of fly ash composite material

4.2. Unconfined compressive strength

The unconfined compressive strength (UCS) of a material is its resistance to any externally applied load. It reflects inter-granular cohesion as well as the strength of cementing material holding those grains. The samples were tested both with and without lime addition. The sample without lime addition did not exhibit any significant strength value. It was only 297 kPa. There was no appreciable change in the strength value at different curing periods as well and hence those data are not reported here. However, lime addition changed the strength behavior significantly. At 7 days of curing period the uniaxial compressive strength of the composite increased manifold. It failed at 1.215 MPa thus achieving a 305% increase (Table 2). Then the increase rate reduced to 10% exhibiting 1.450 MPa at 14 days. But the rate of increase increased to 54% at 28 days of curing to exhibit 2.85 MPa. The specimen continued exhibiting increased strength value at 56 days though with a much-reduced rate (Figure 2). All the samples exhibited the shear-type of failures thus confirming the development of cohesion between particles (Figure 3).

Table 2: UCS values of FCM at different curing periods

Curing Period (days)	Compressive Strength (MPa)
0	0.297
7	1.215
14	1.450
28	2.850
56	2.950

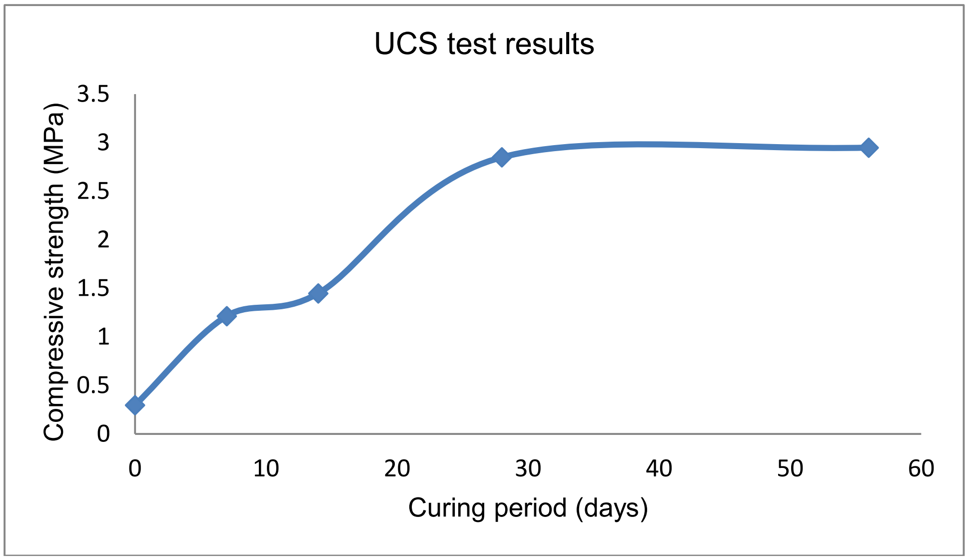

Figure 2.UCS values of fly ash composite material at different curing periods

Figure 3: Post failure profiles of UCS samples

4.3. Brazilian tensile strength characteristics

Tensile strength is an important property to predict the cracking behavior of the filled mass and is a vital parameter to evaluate the suitability of fly ash as a filling material in mining voids. In the present study tensile test was conducted on developed composites to evaluate the tensile strength as well as the cracking behavior of the material. The tensile strength of the fly ash composite material showed significant improvement with curing periods. At 28 days curing the tensile strength values increased at 100% and 200% to that of at 7 and 14 days respectively (Table 3). Marginal increase was also observed at 56 days curing period (Figure 5). All the specimens failed at the middle through an induced force which is tensile in nature (Figure 4). The failure occurred within 70 to 110 seconds thus confirming to that suggested in ASTM D3967.

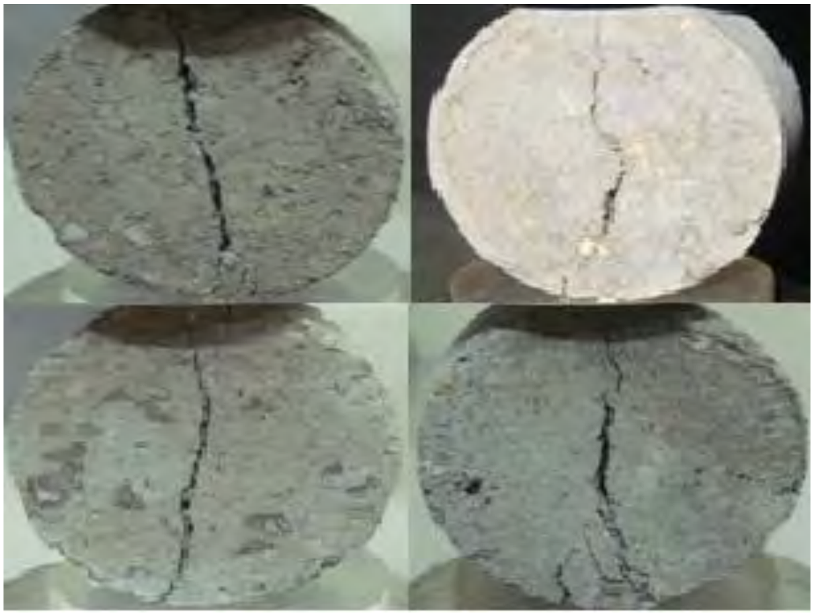

Figure 4. Post failure profiles of Brazilian tensile test samples

Table 3: Relationship between curing period and Brazilian tensile strength

Curing period (days)	Brazilian tensile strength (kPa)
0	057
7	150
14	180
28	300
56	335

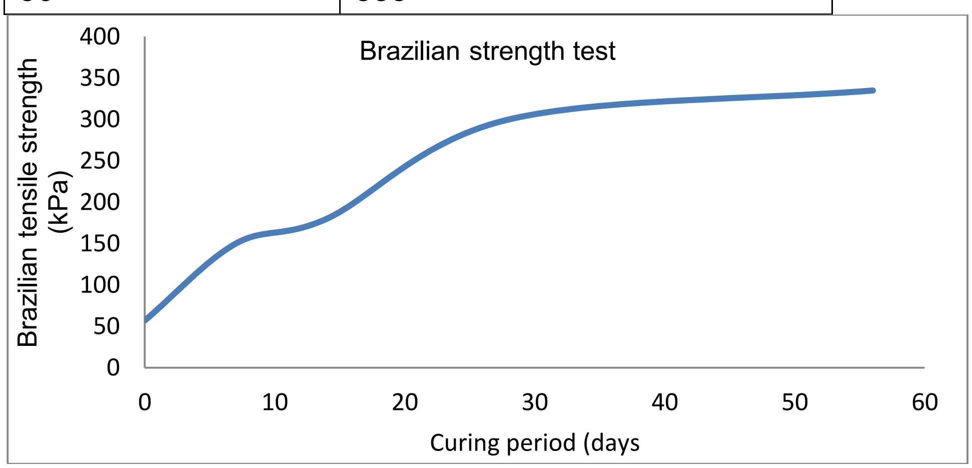

Figure 5: Tensile strength values of developed composites at different curing periods

4.4. Shear strength parameters

The post failure profile of a triaxial test specimen is presented in Figure 6. The shear strength parameters of compacted fly ash composite materials are presented in Table 4. There is little change in cohesion and angle of internal friction values at 7- and 14-days curing (Figure 7 and 8). Both cohesion and angle of internal friction increased with curing period. At 28 days curing the friction angle is about 35^0 which are typical of any medium hard rock (Lama and Vutukuri, 1978). This confirms that the developed composite material would be suitable to support roof load and would also resist putting pressure on barricades.

4.5. Ultrasonic Pulse velocity

The P-wave velocity depends on the quality of transmission, cohesiveness of constituent materials, dampness, presence of weaknesses such as cracks, voids, etc. Its accuracy also depends on the homogeneity of the specimen. The ultrasonic pulse velocities varied between 1410 m/s to 2158 m/s at varying curing periods from 7 days to 56 days (Table 5). Maximum values were obtained at 56 days curing period, thus confirming the increased conductivity in the sample. But it increased by 12% at 28 days thus reflecting improved transmissivity of the wave due to enhanced pozzolanic activity. The rise is marginal between 7 and 14 days of curing.

Table 4: Shear strength parameters of fly ash composite materials

Curing period	Cohesion (kPa)	Angle of internal friction (degrees)
7	53	27.85
14	54	28.35
28	71	34.60
56	78	36.52

Figure 6: Post failure profile of a triaxial test specimen

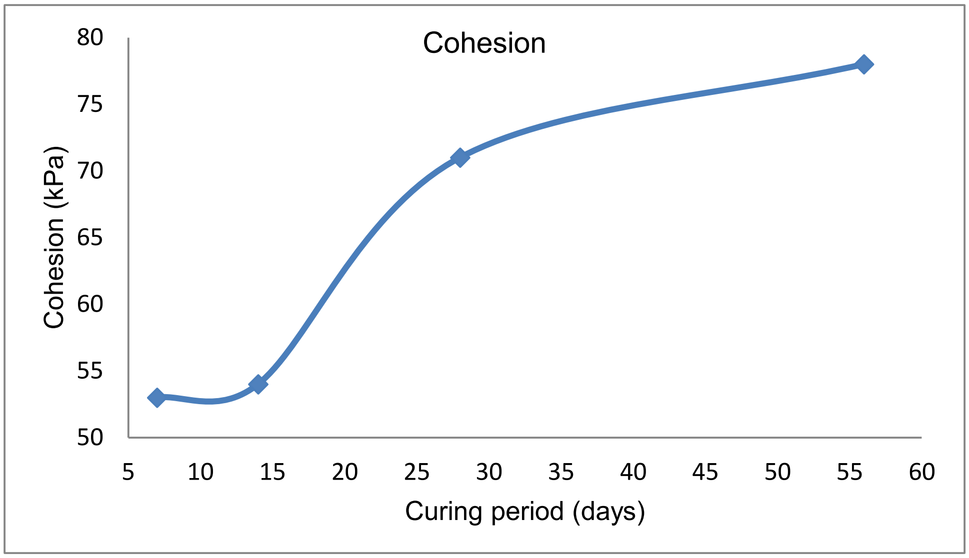

Figure 7: Relationship between curing period and cohesion

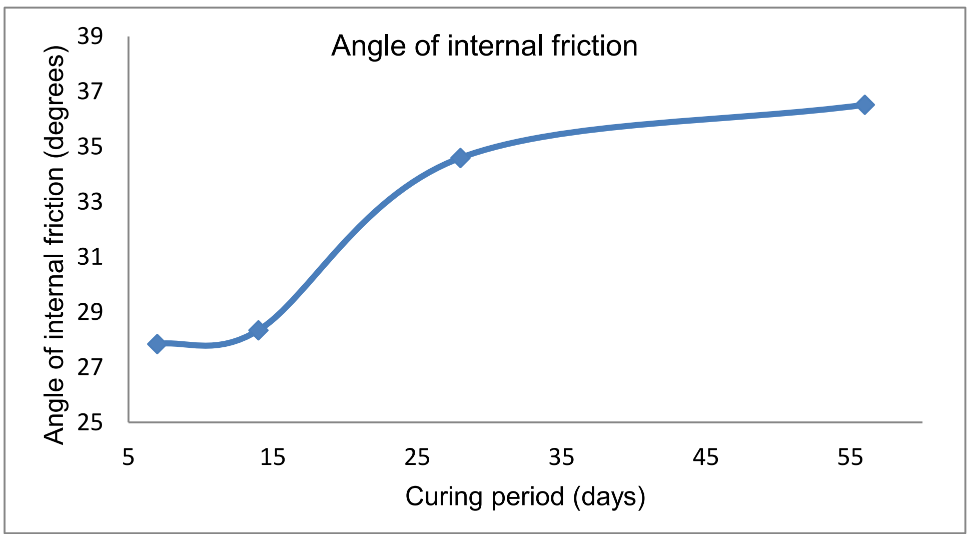

Figure 8: Relationship between curing period and angle of internal friction

Table 5: P-wave velocities of developed composites at different curing periods

Curing period (days)	P-wave velocities (m/s)	Poisson's Ratio
7	1410	0.44
14	1414	0.41
28	1577	0.40
56	2158	0.28

The P-wave velocity at 56 days of curing period was 2158 kPa and least values were obtained for 7 days of curing period which confirms to the results obtained in UCS and BTS tests. The Poisson's ratio is also an important parameter of a material under loading. The Poisson's ratio values were obtained from ultrasonic pulse velocity test as well. The Poisson's ratio values of each composite decreased with increase in curing period. The Poisson's ratio values varied between 0.28 and 0.44 of all developed composites cured at 7, 14, 28 and 56 days (Table 6). The Poisson's ratio values of each composite did not change significantly with longer curing periods which are the typical characteristics of any material. Young's modulus (E) values were also obtained from nondestructive testing (Table 7). The Young's modulus (E) values increased with curing period confirming to enhance pozzolanic activities resulting in higher stiffness of the composites. The velocity of propagation increases with increased stiffness of the material (Yesiller *et al*., 2000). The density of the material also increased with curing period (Table 8) confirming to strength gain.

Table 6: Relationship between curing period and Poisson's ratio

No. of days cured	Poisson's ratio
07	0.44
14	0.41
28	0.40
56	0.22

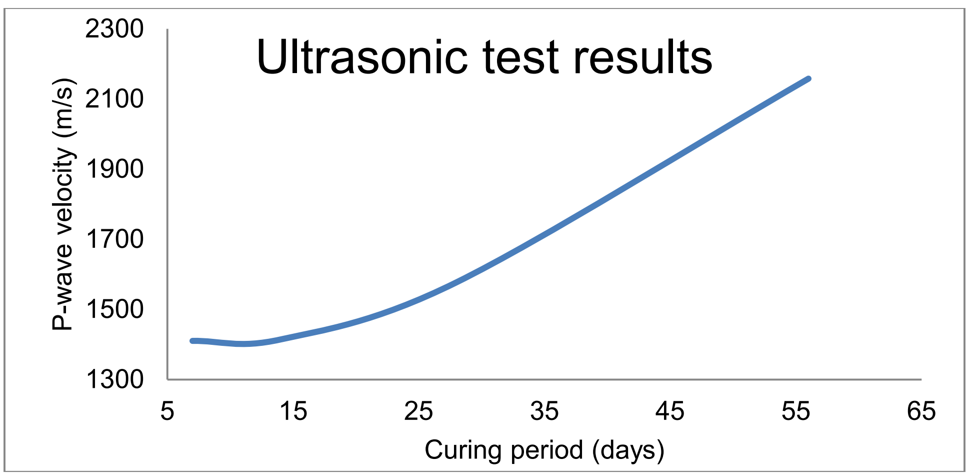

Figure 9: P-wave velocities of developed composites at different curing periods

Table 7: Ultrasonic test parameters

Ultrasonic test parameters	Curing Period (days)			
	7	14	28	56
Young's modulus (kPa)	950337	1196933	1764446	5750525
Bulk modulus (kPa)	1038263	2146938	3377293	4795166
Shear (Rigidity) modulus (kPa)	133268	425325	661389	2211523
S-wave velocity (m/s)	235	560	580	697

Table 8: Relationship between curing period and density

Curing period (days)	Density (Kg/m^3)
7	1460
14	1470
28	1520
56	1700

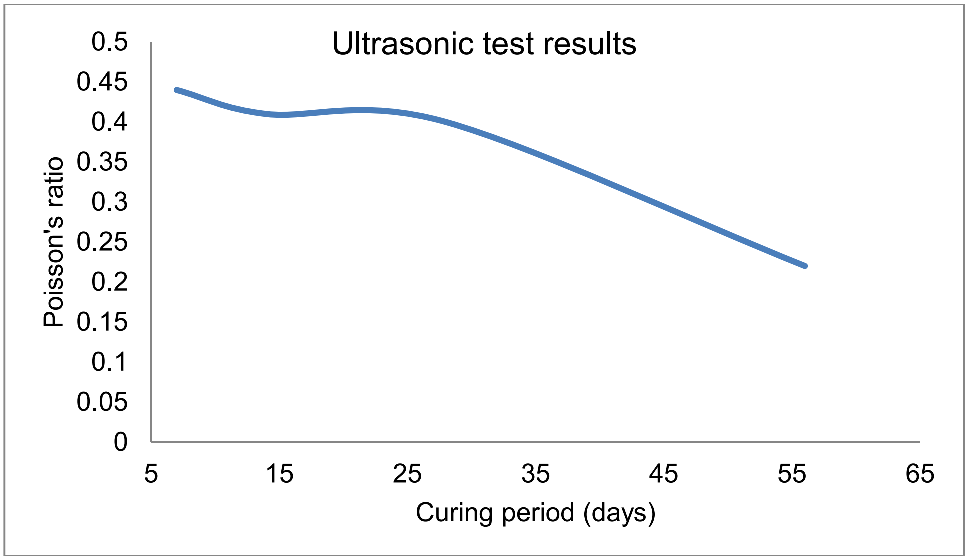

Figure 10: Relationship between curing period and Poisson’s ratio

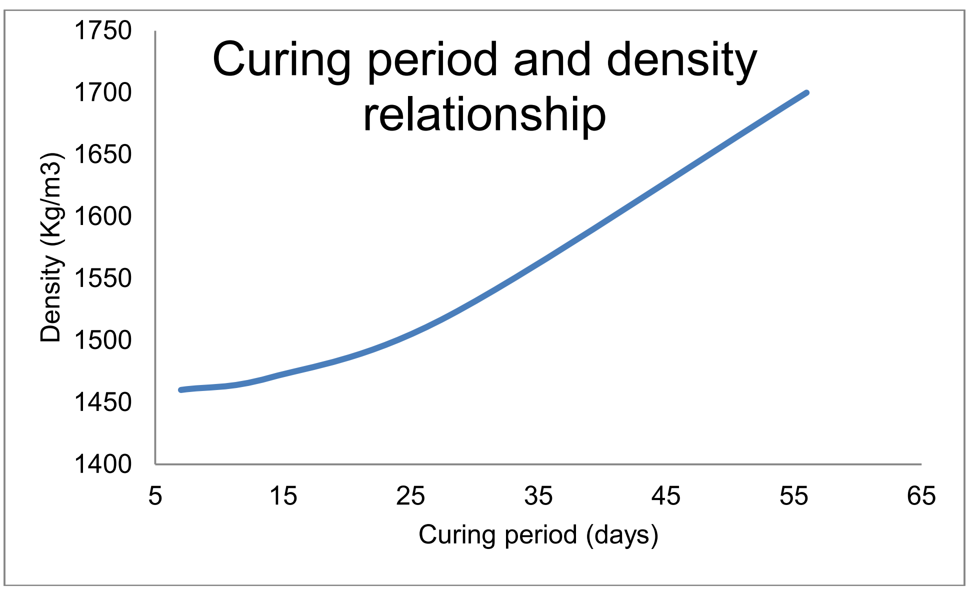

Figure 11: Relationship between curing period and density

4.6. Micro-structural analysis

The SEM images show development of gel at different stages of pozzolanic reaction. It confirms to the observation that during early stages, the reactive particles in the fly ash composite served as nucleation sites for hydration and pozzolanic reaction products as (C-S-H, C-A-H, C-A-S-H) [Lav *et al*., 2000]. Cementitious compounds are formed around fly ash particles (Figures 12-16). The composite at 56 days of curing period exhibited dense-gel-like mass covering all reactive particles completely and filling up the inter-particle space with blurred grain boundaries (Figures 16). It appears like a massive unit compared to the other composites. The dense gel acted as a binding substance and appeared to be evenly distributed to form compact structure, thus creating more contact and higher cohesion that in turn reflects in greater strength values. It was observed from static laboratory tests that all samples exhibited maximum strength values at 56 days. So, its SEM analysis was carried out to understand the micro-structural aspects. The strength values of the developed fly ash composites increased with increasing curing period due to the formation of calcium silicate hydrate (CSH) and calcium aluminate silicate hydrate gels (CASH) around fly ash particles (Cetin *et al*., 2010).

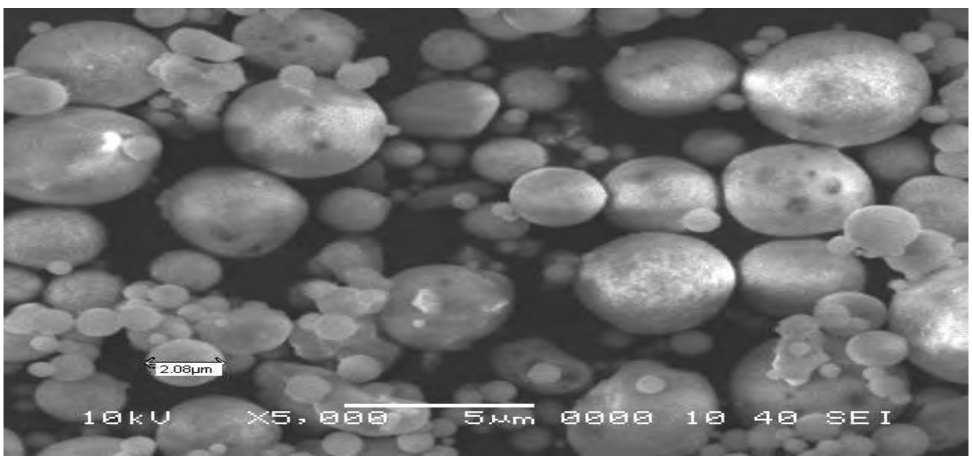

Figure 12: SEM image of untreated fly ash at 5000x

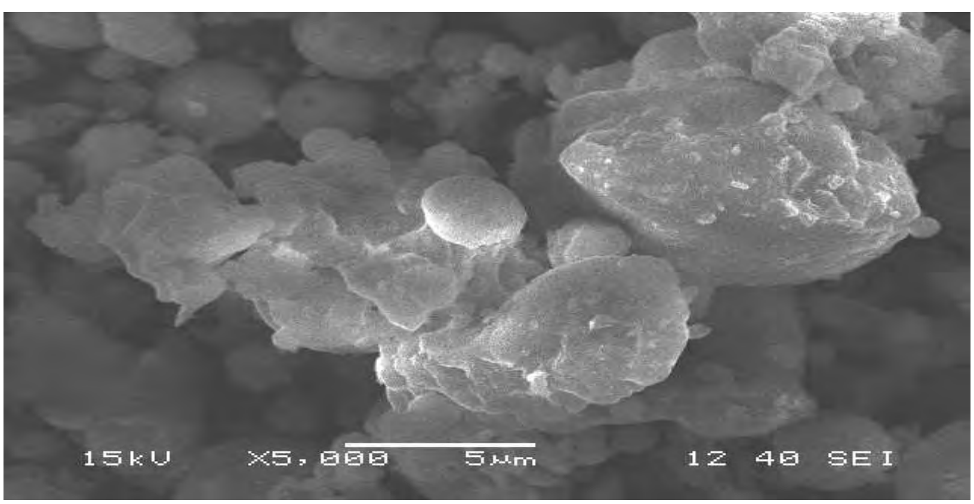

Figure 13.SEM image of 7 days curing at 5000 x

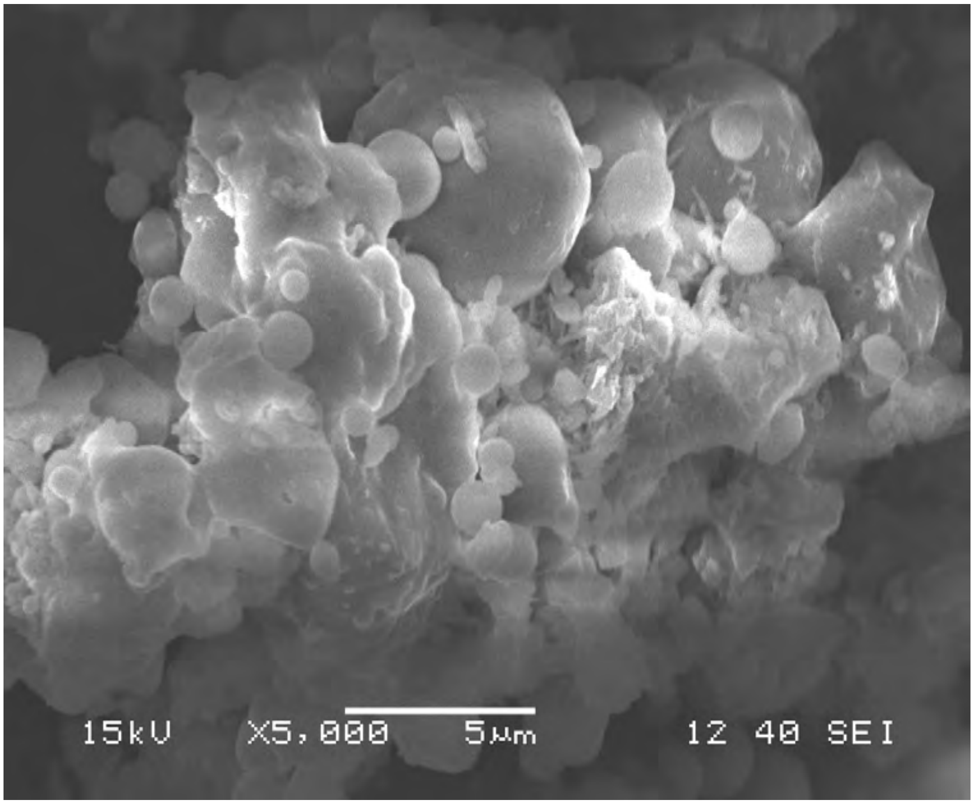

Figure 14.SEM image of 14 days curing at 5000 x

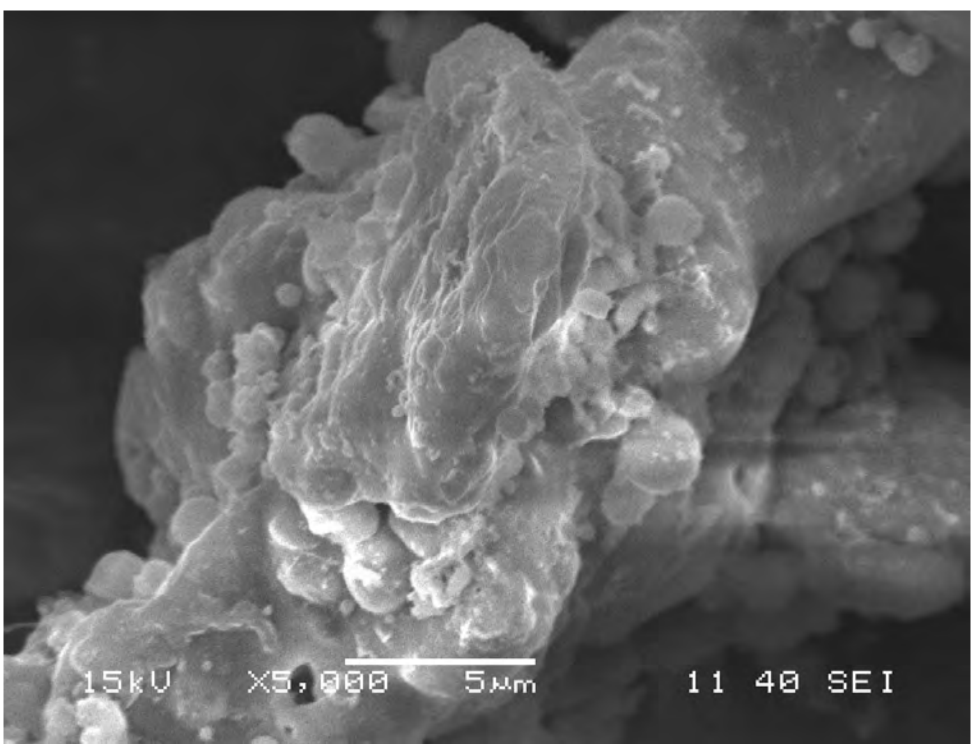

Figure 15.SEM image of 28 days curing at 5000 x

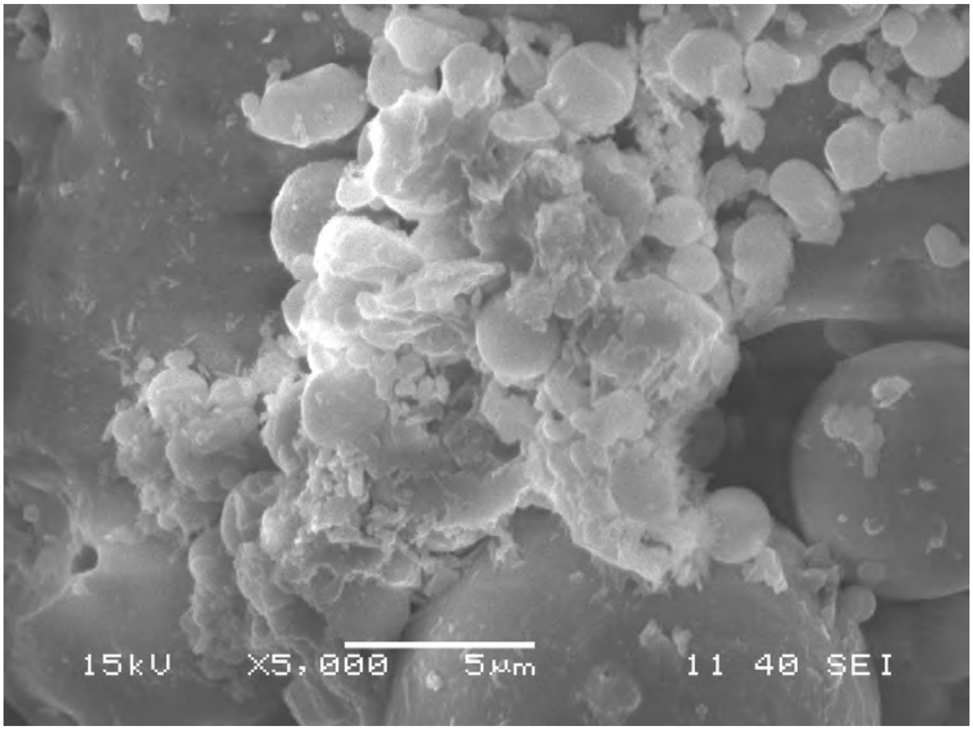

Figure 16.SEM image of 56 days curing at 5000 x

4.7. X-ray diffraction analysis of FCM

The mineralogical analyses of the composites are very important to determine the changes in the mineralogical phases due to pozzolanic reactions. Cementing compounds such as CSH, CAH and CASH were identified in 3% cement stabilized fly ash only and fly ash – black cotton soil mixes at 28 days curing by XRD analysis (Krishna, 2001). The strength development is also dependent on the amount of hydration products as well as their interlocking mechanisms (Lav and Lav, 2000). The formation of reaction products such as calcium silicate hydrates CSH; Calcium aluminates hydrates (CAH) and Calcium aluminates silicate hydrates (CASH) were confirmed from x-ray diffraction analysis (Figures 17-20). These new cementitious compounds induce aggregation effect in fly ash and bind the particles together to form fly ash clusters and resulted in overall enhanced strength behaviors of composites. Quartz the primary mineral present in fly ash indicated by sharp peaks at 27^0 (approximately).

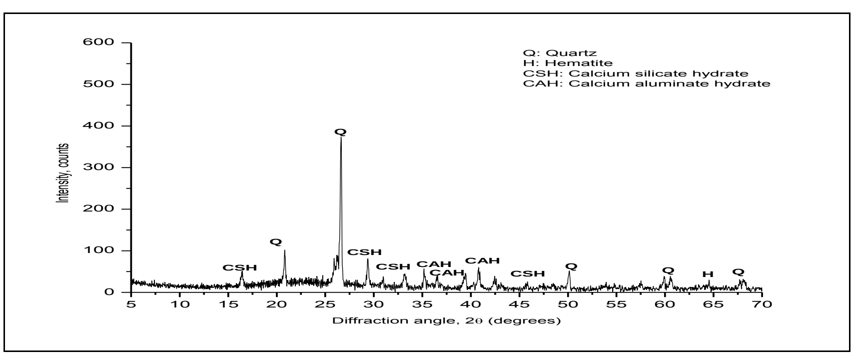

Figure 17.XRD peak of fly ash composite material at 7 days curing

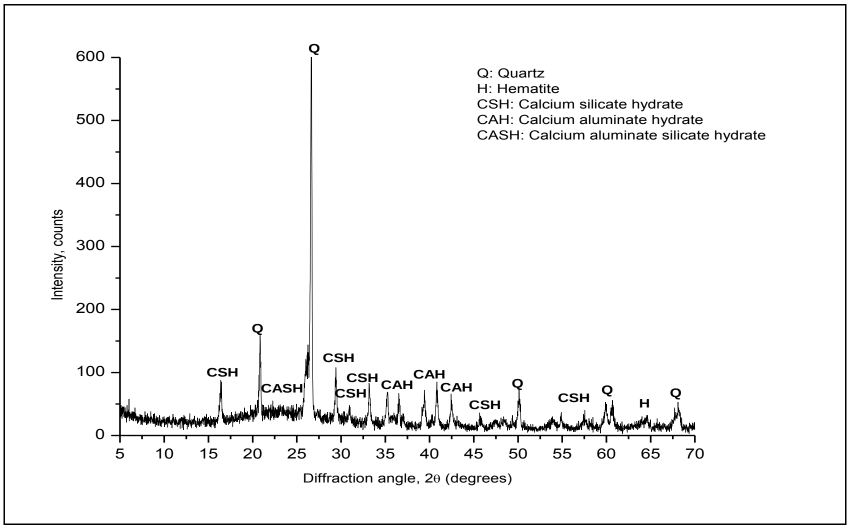

Figure 18.XRD peak of fly ash composite material at 14 days curing

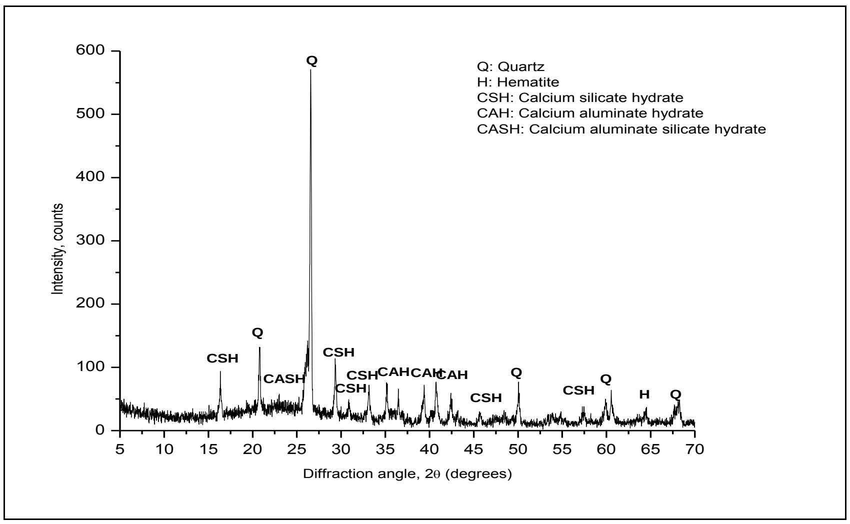

Figure 19.XRD peak of fly ash composite material at 28 days curing

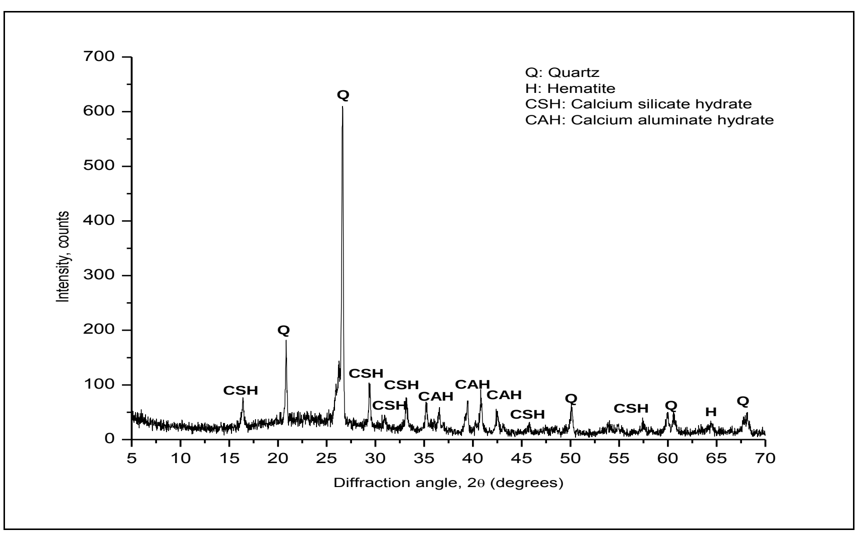

Figure 20.XRD peak of fly ash composite material at 56 days curing

4.8. FTIR Analysis

FTIR (Fourier Transform Infrared Spectrometry) investigation has been carried out to obtain information regarding functional groups of materials/compounds. The quality or consistencies of the fly ash-additive mixture after treatment have been significantly depicted. The FTIR monograph in Figure 21 shows that the untreated fly ash has peaks at 1089.78 cm^{-1}, 796.60 cm^{-1}, and 462.92 cm^{-1}. These peaks attribute to T-O-Si (internal linkage; T = Si or Al); Si-O-Si (external linkage); Si-O-Si or O-Si-O groups of fly ash that are mainly responsible for strength behavior of the material. When the fly ash was mixed with lime, surfactant and NaSal, a doublet (two peaks) was observed at ~ 790 cm^{-1} and a peak at ~ 1400 cm^{-1} region that reflects the presence of O-C-O group (Figures 22 - 23) i.e., no geolitesation even in presence of surfactant and counter-ion (Ojha *et al*., 2004**)**. As the curing period progressed to 7 and 14 days the peaks were observed at ~ 3400 cm^{-1}, 1400 cm^{-1}, 1600-1650 cm^{-1}. The peaks depict OH stretching to OH bonding (Figures 25 – 27). These peaks depict the presence of O-C-O, H-O-H groups. At 28 days curing period 3300 – 3600, 1350 – 1450, 1600-1650 and ~ 950 cm^{-1} peaks with OH, O-C-O, H-O-H and O-C-O stretching, and H-O-H and H-O-H internal Ti-O-Si group bonding were observed (Figure 28). This reflects higher bonding i.e., effective goelitesation (Park & Kang, 2008). The peaks were broader after 56 days of curing (Fig 29).

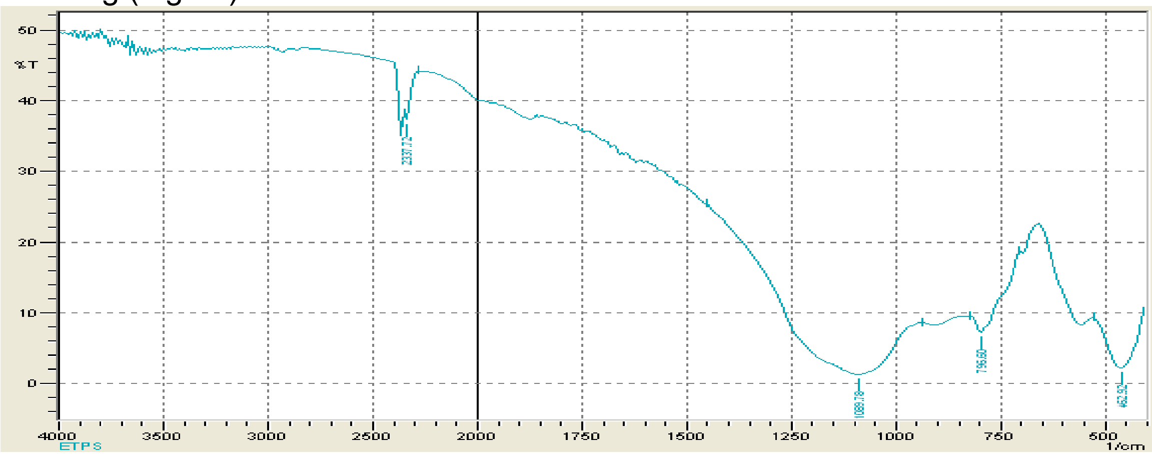

Figure 21: FTIR results of untreated fly ash

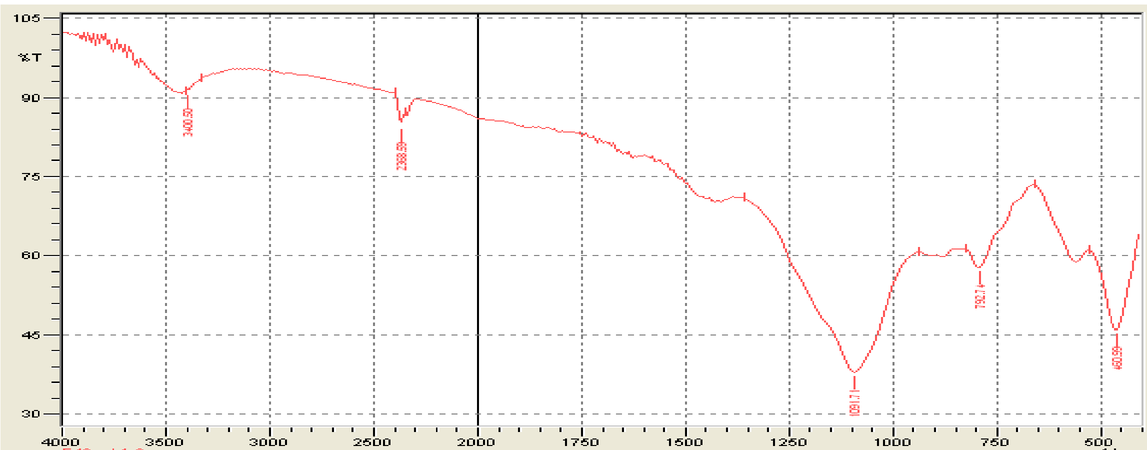

Figure 22. FTIR spectra of treated fly ash (FA 48w50S.1N.1L1.8)

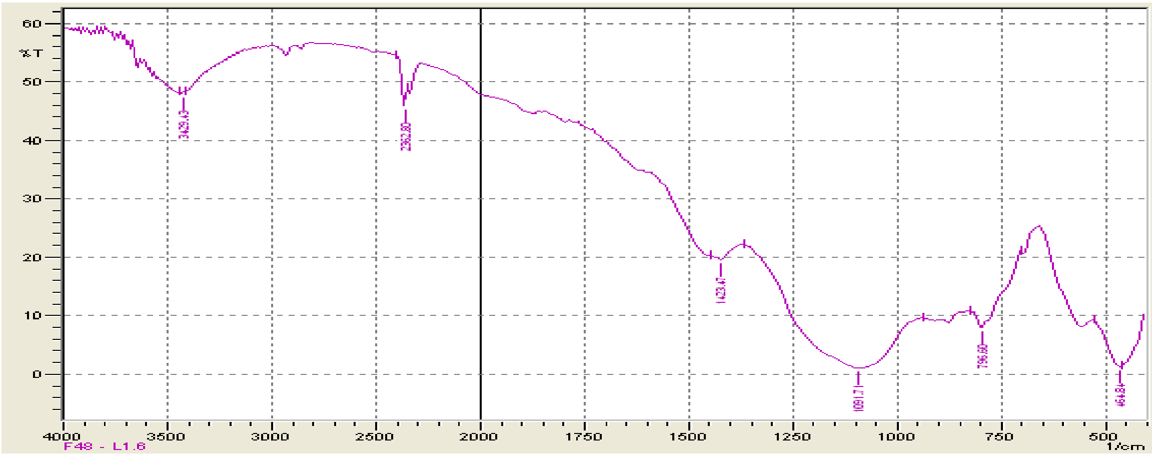

Figure 23: FTIR spectra of treated fly ash (FA 48w50S.2N.2L1.6)

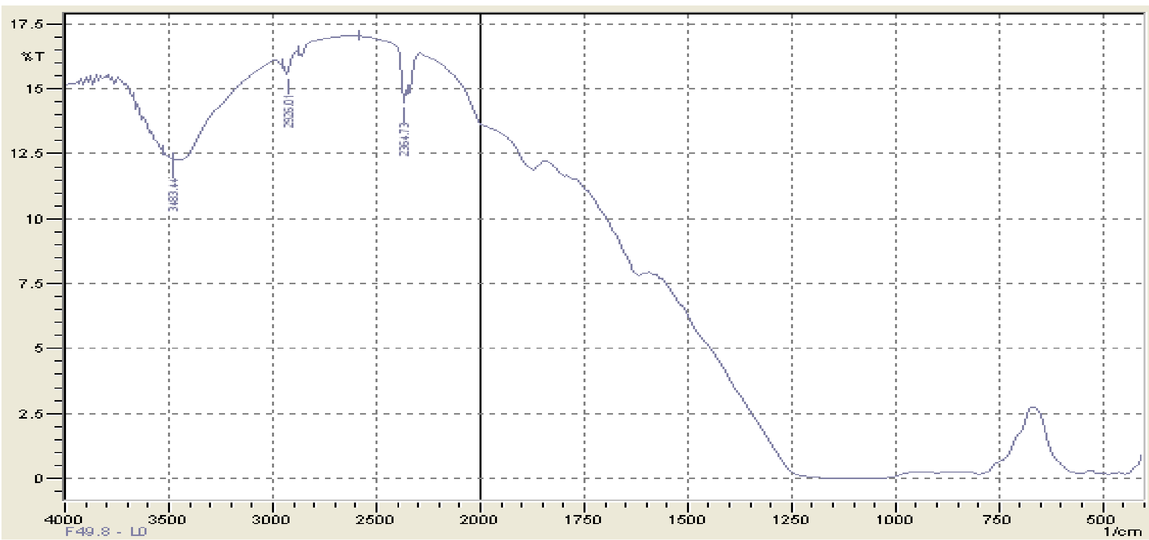

Figure 24: FTIR spectra of treated fly ash (FA 49.8w50S.1N.1L0)

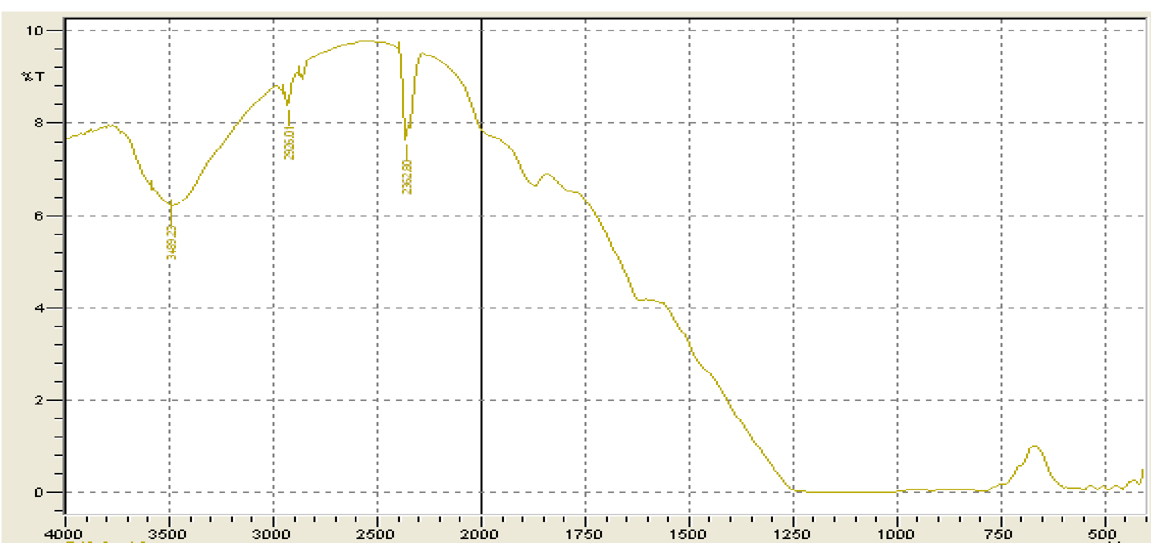

Figure 25: FTIR spectra of treated fly ash (FA 49.6w50S.2N.2L0)

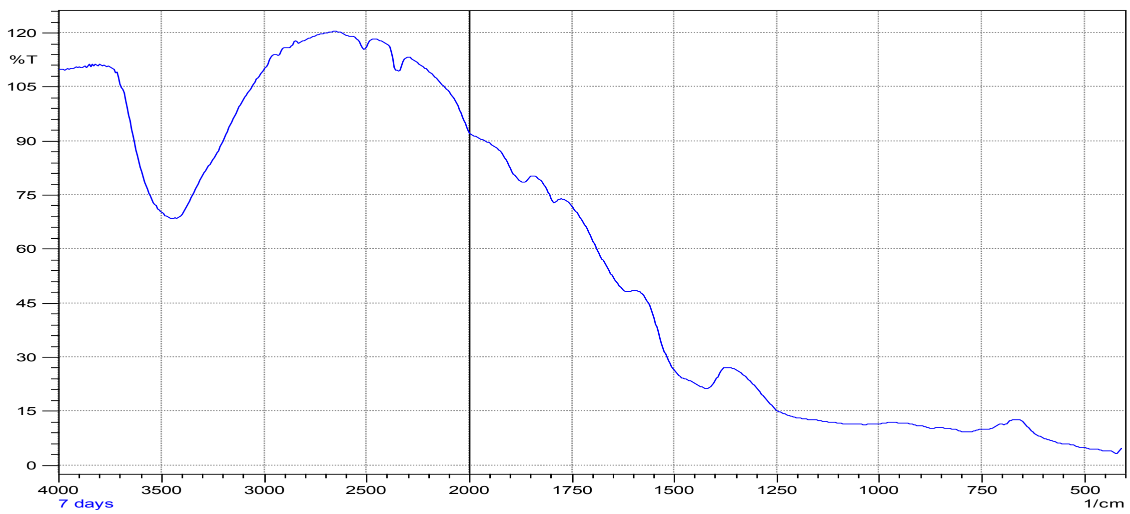

Figure 26. FTIR results of treated fly ash composites at 7 days curing period

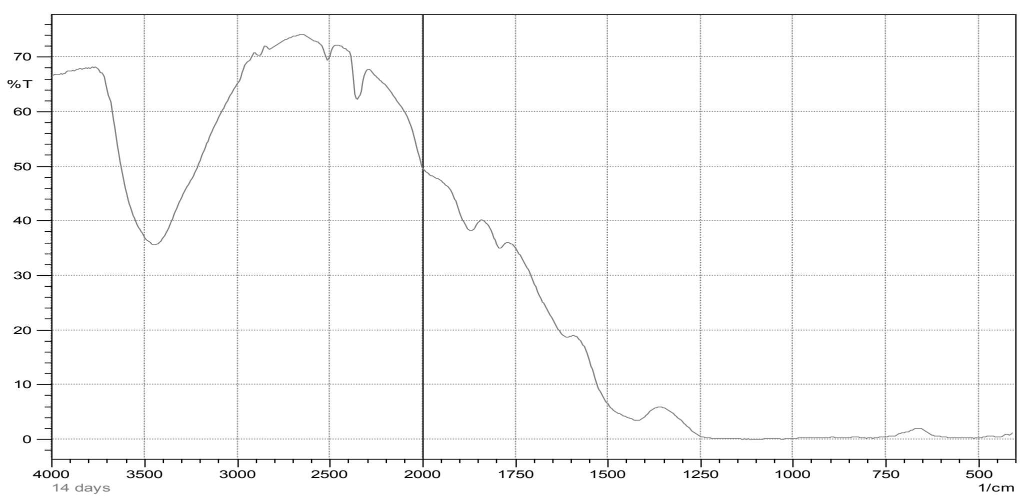

Figure 27: FTIR results of treated fly ash composites at 14 days curing period

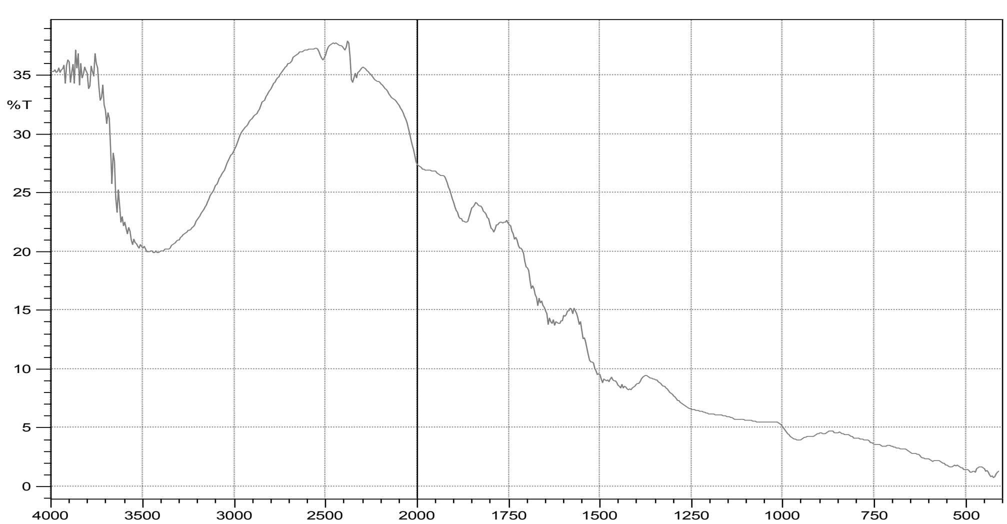

Figure 28: FTIR results of treated fly ash composites at 28 days curing period

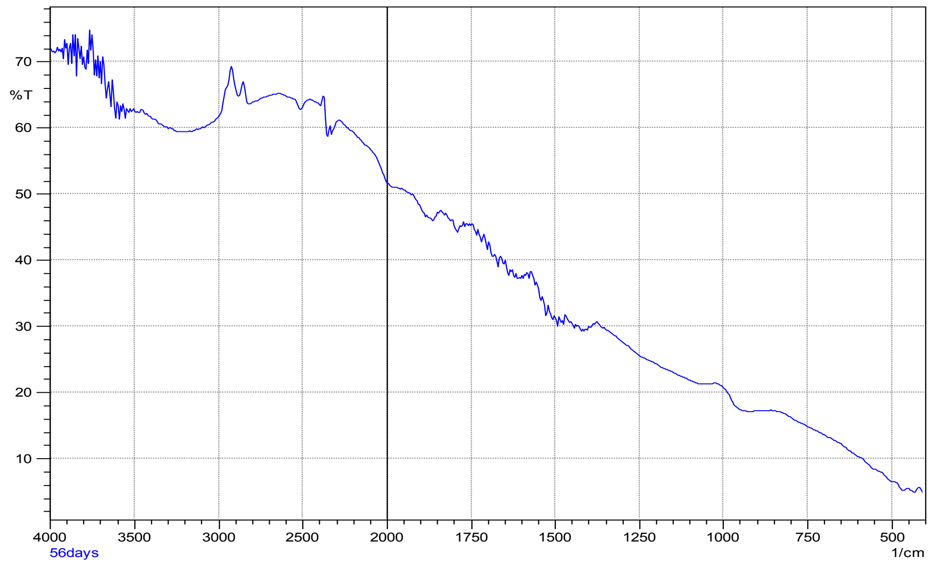

Figure 29: FTIR results of treated fly ash composites at 56 days curing period

5. Development of Empirical models

A part of the research objectives was to develop model equations for the investigation with the parameters like UCS, BTS, and P-wave velocity. Those are reported here for the best fit correlations.

Relationship between UCS, BTS and P-wave velocity

The investigation involved samples for various parametric determinations. Each parameter has been discussed separately earlier. A few empirical models have been developed to establish mutual correlation between UCS and P-wave velocity, BTS and P-wave velocity etc. The data are analyzed using multiple regression models by the method of least squares (Figures 30-34). There exists relation between compressive strength of fly ash-lime and fly ash-lime-gypsum mixes with chemical composition, loss on ignition, CBR and tensile strength using power model (Ghosh and Dey, 2009). It confirms that the relationship between compressive strength and P-wave velocity become stronger with increasing curing period. The results of regression model between unconfined compressive strength, Brazilian tensile strength and P-wave velocity at different curing period are reported (Table 9).

Table 9: The developed correlation among various parameters of fly ash composite materials

Parameters to be co-related	Best fit equation	R^2 value
UCS - BTS Co-relationship	y = 94.599x + 43.892	0.9790
Cohesion and angle of internal friction	y = 0.3511x + 9.3572	0.9972
Density and curing period	y = 5.0559x + 1404.8	0.9661
Cohesion and curing period	y = 0.5416x + 49.783	0.8868
Angle of internal friction and curing period	y = 0.1871x + 26.918	0.8562

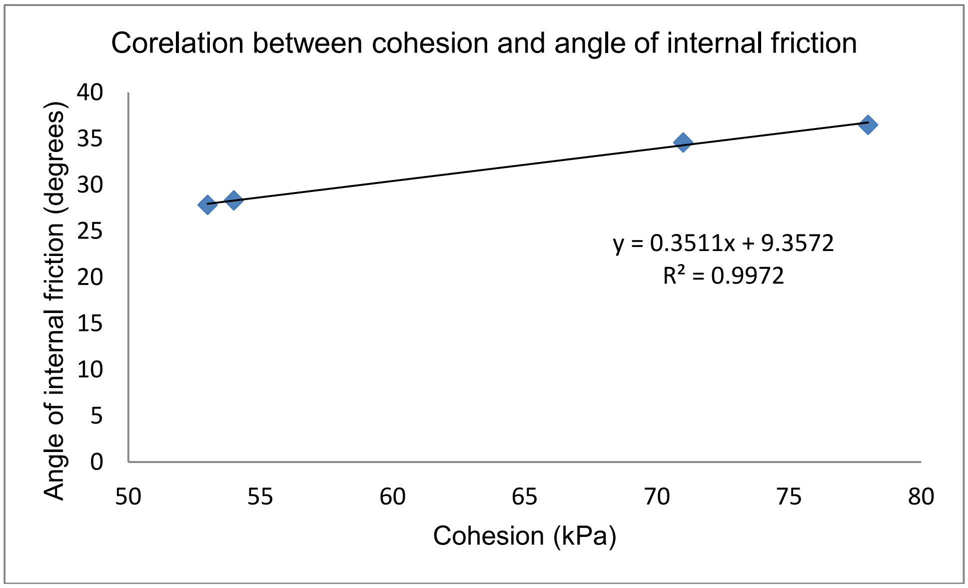

Figure 30: Correlation between cohesion and angle of internal friction

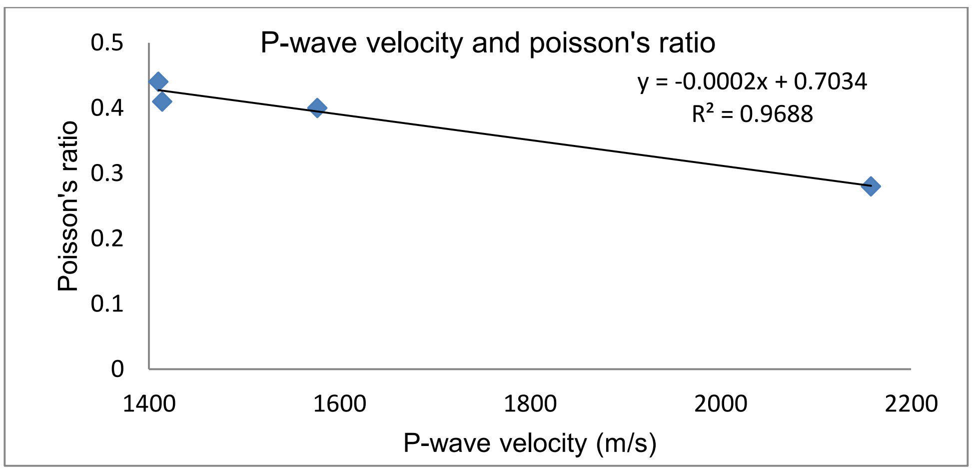

Figure 31: Correlation between P-wave velocity and Poisson's ratio

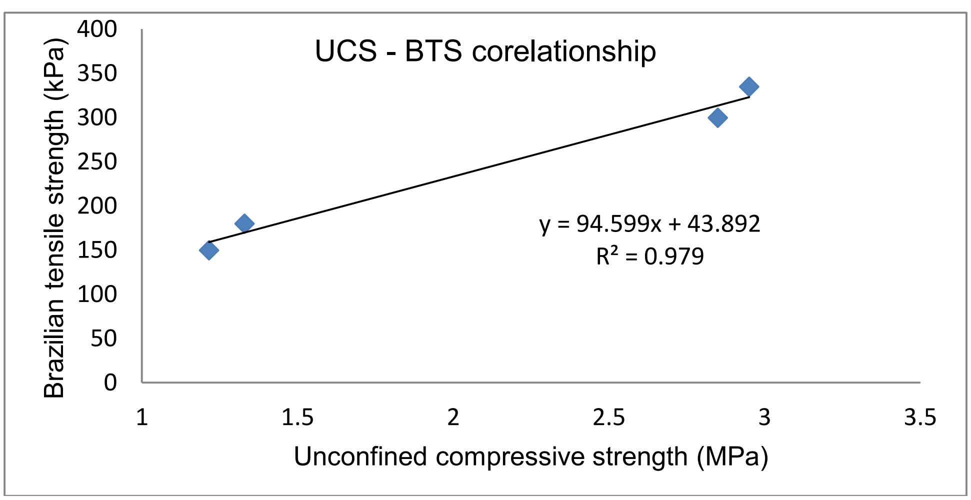

Figure 32: Relationship between BTS and UCS

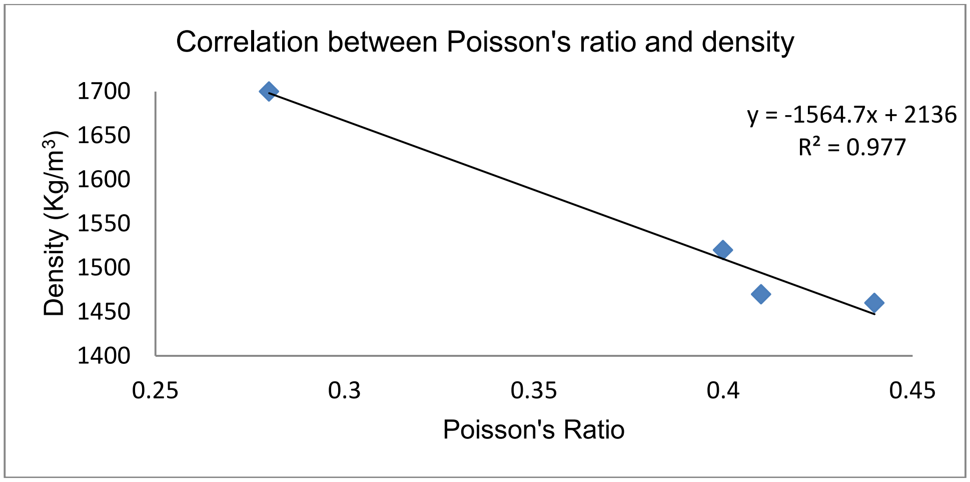

Figure 33: Relationship between Poisson's ratio and density

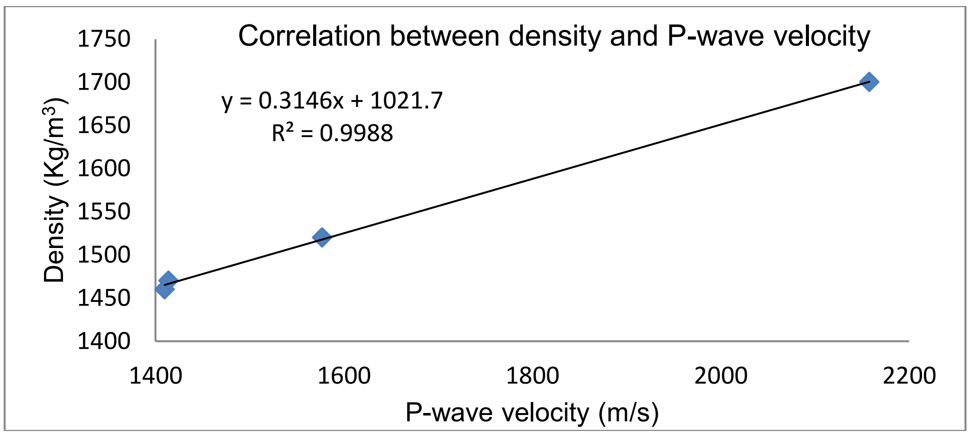

Figure 34. Correlation between density and P-wave velocity

6. Conclusion

In this study, the prime objective was to select an alternate backfill material to fill mining voids. To achieve the objectives seven number of fly ash samples were collected from seven different sources. The seven number of fly ash samples were tested in the laboratory to study their suitability for mine filling purposes. Out of the seven fly ashes studied the best suitable one was selected for further study with respect to its flow and in-place strength characteristics. A surface-active agent (surfactant) was added to the selected fly ash to modify the flow behavior of the slurry in pipelines during its transportation to fill mine voids. Flow study was conducted at different additive concentration to optimize the same. The secondary objective was to measure the in-place strength characteristics of developed fly ash composite materials. The in-place strength characteristics of developed composite materials were measured by using another reagent called lime. The results of all the investigation were reported in this paper which confirmed that the selected fly ash can be filled in the mine voids to serve its purpose of giving support to the roof above it.

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