Gypsum Induced Compaction Behaviour of Fly Ash Stabilized with Bentonite and Lime including mineralogical and microstructural analysis

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Abstract- This paper mainly deals with the compaction characteristics of fly ash which is further modified with 20, 30, 40 and 50% of bentonite, 5, 10 and 15% of lime, in addition with 0.5, 1.0 and 1.5% of gypsum including physical properties and analytical studies of raw materials and mixes. X-ray diffraction and scanning electron microscope (SEM) including energy dispersive spectroscopy (EDS) analysis carried out. Using standard Proctor test, MDD and OMC found out,where densifications of materials are imperative to reuse for landfilling purposes. In general, test results reveal that the increasing trend of MDD in maximum cases and fluctuation of OMC with the different mixes which may be applied for field applications. This study pretend, the application of waste materials without treatment confers detrimental effect to the whole environment and ground water. Composite is very much effective for field applications as land fill, embankment and road sub-base; mix contains maximum percentage of fly ash which is waste materials and abundantly available with free of cost, and very little percentages of bentonite, lime and gypsum added to it. It's a new concept and very much useful.

Keywords – Fly ash, Bentonite, Lime, Gypsum, Compaction, XRD, SEM

I. INTRODUCTION

Landfilling is one of the useful methods of safe disposal of municipal, industrial, and hazardous wastes. Low-permeable barriers known as liners are an integral part of landfills, which minimize the migration of contaminants to surrounding geo-environment and groundwater [1]. Fly ash is a disposed waste material produced by coal-based thermal power plants where this enormous amount of fly ash faces the problem of vast land requirement, transportation, ash pond construction and maintenance, which may be reduced by recycling of waste material is one of the practical solutions of its disposal problem. Utilizing fly ash as a construction material for civil engineering structures and indirectly may reduce the environmental issues, like dusting, leaching etc. as Class F fly ash contains lower percentages of lime. To improve the engineering properties of Class F fly ash, an attempt has been made to stabilize fly ash with lime and gypsum widely used as barriers because of their cost-effectiveness and high contaminant attenuation in the absence of natural clays, have been widely used as contaminant barriers. Fly ashes containing higher amounts of free lime which causes self-hardening characteristics find bulk and potential applications in civil engineering, and construction field mostly because of their ability to develop considerable strength due to pozzolanic reactions with the available reactive silica only upon addition of certain cementing agents such as cement and lime [2]. According to [3] a good rule of thumb in practice is to allow 1% by weight of lime for each 10% of clay in the soil. Exact prescriptions could be made after tests at and slightly each side of this value. Because it was exceptional for the clay content of soil to exceed 80%, it was usually not necessary to add more than 8% lime. Class C fly ash with high calcium content undergoes high reactivity with water even without the addition of lime [4].To improve the engineering properties of Class F fly ash which contains lower percentages of lime, an attempt had been made to stabilize fly ash with lime or cement [5, 6]. Gypsum had also been used to stabilize fly ash [6, 7].

As liners are used as barrier systems in landfill design to minimize the escape of contaminants from landfills and mitigate their impact on public health and the environment, expansive soil, like, high swelling bentonite with 2:1 expandable layers, high layer charge, high base exchange capacity, very thin flakes, higher surface area, high absorption capacity, high viscosity, thixotropic could be used as barrier clays [8].After modifying the properties of bentonite soils with some additives such as lime which in fact, when expandable clays tend to react readily with lime, losing plasticity immediately as reported by [9] and modified with phosphogypsum [10] to make them suitable for land filling material at site by waste materials in which the functional integrity of landfills depends heavily on the densification of the barrier systems, where bentonite provide the better density while packed together by rearranging of particles and fill up the voids after proper compaction treatment by stabilizers in the presence of optimum water content.The high swelling sodium bentonite was commonly used in earthen structures such as dams, to seal irrigation ditches, to prevent seepage of water from ponds and impounds, and to prevent water from entering basements of homes by the mechanism of swelling for water impedance, and fills the pores and voids in the material into which it was incorporated preventing water or other liquids from moving through the barrier [11].[12] Studied on expansive soil stabilized with phosphogypsum and reported a decrease in the plasticity index, and in the optimum moisture content, where an increase in the dry unit weight and in the unconfined compressive strength with the addition of phosphor gypsum was observed.

When lime is added to a clay soil, it has an immediate effect on the properties of the soil as cation exchange begins to take place between the metallic ions associated with the surfaces of the clay particles and the calcium ions of the lime which alters the density of the electrical charge around the clay particles which leads to them being attracted closer to each other to form flocs, the process being termed flocculation which is primarily responsible for the modification of the engineering properties of clay soils when they were treated with lime [13] as well as a lime fixation point which represent the optimum addition of lime needed for maximum modification of the soil and corresponds with the point, where further addition of lime didnot bring about further changes in the plastic limit when lime was added to a clay soil [14].[15]Studied the use of a lime-pure new gypsum mixture to reduce the potential for swelling of expansive clay soil from an economic point of view and obtained promising results.

[16] Observed that fly ash addition to expansive soil reduced the optimum moisture content (OMC) and increased the maximum dry density (MDD) of the mixes. Conversely, [17] noted that ion-exchange reactions occurring on mixing fly ash with clay soils produced flocculated and agglomerated soil fabric that lowered the MDD and increased the OMC of the mixes. [18]Obtained the increased values of MDD and OMC after compaction test using two different types of fly ashes with various lime percentages upto 10% which revealed the addition of lime imparts plasticity, otherwise non-plastic fly ashes resulting in a marginal increase in dry density and moisture content values. [19]Reported after compaction test of soil-fly ash-lime-gypsum mixes, where the MDD was increased after the addition of fly ash to soil and OMC decreased which mightbe attributed to improvement in gradation of soil with addition of fly ash which is predominated with silt-sized particles and MDD decreased with an increase in OMC after addition of varying lime content to soil-fly ash mixtures due to the enhanced flocculation and cementation of clay particles counteracting the effect of compactive effort.Similar observations found out by several researchers [20-22]. Furthermore decrease in MDD and increase in OMC was observed with the addition of gypsum in soil-fly ash-lime mixtures which might be due to enhanced binding of particles in the presence of gypsum. [23]Reported similar kind of result for sulfate soil stabilized with lime and fly ash.
The rest of the paper is organized as follows. Materials, Sample preparation and Programme are explained in section II. Experimental results and discussions are presented in section III. Analytical results and discussions are given in Section IV. Concluding remarks are given in section V.

II. MATERIALS, SAMPLE PREPARATION AND PROGRAMME

A. *Materials –*

An attempt has been made to develop a cost-effective engineering material from wastes of Indian thermal power plants for landfilling, construction of embankments and liners/covers. Class F fly ash sample has been used in this investigation as a landfilling material. To recycle this waste material different additives viz., bentonite and locally available lime are mixed in various mix proportions and modified by the pure gypsum (Calcium sulfate dihydrate ($CaSO_4$. $2H_2O$)) also in small proportions considering the economic aspect. In this entire paper, all mixes are designated with common coding process as mentioned: FA stands for fly ash; B stands for Bentonite, L stands for lime, G stands for gypsum. The Arabic numerals before FA, B, L and G indicate their respective weight percentages in the mix. The test results on physical properties in Table 1 as per ASTM guidelines and chemical compositions in Table 2 of the fly ash sample, bentonite, lime and gypsum are presented.Properties of water used in this study is shown in Table 3.

Fly ash

The fly ash sample used in this study is the key material of land filling procured from Kolaghat Thermal Power Station West Bengal, India. According to ASTM C618 [24], this fly ash may be categorized as Class F type.Low lime content fly ashes falling in Class F are commonlyfound in India and in many countries around the world.

Bentonite

Bentonite comprised predominantly of the smectite group of minerals, where the property of ion-exchange and the exchange reaction are very important in many of the applications in which the smectite minerals are used.Commercially available bentonite powder is collected from the localmarket. According to [25],the soil is classified as highly plastic clay with a high degree of expansion.

Lime

ASTM C1529 [26]analytical grade quicklime is used throughout the test program for stabilizing the fly ash with bentonite and collected from the local market.

Gypsum

To accelerate the bonding between particles, anhydrous gypsum having specific gravity 2.32 of 0.5, 1.0 and 1.5% by dry weight is also used to reduce the stabilized matrix by producing more ettringite.Pure gypsum ($CaSO_4.2H_2O$) named as Calcium Sulfate di-hydrate have been used in the present study as a modifier of mixes and collected from the local market.

Table -1 Physical Properties of Materials

Sl.No.	**Properties**	**Fly Ash**	**Bentonite**
1.	**Specific gravity**	**2.21**	**2.80**
2.	**Sand size (4.75–0.075 mm)**	**6.00**	**1.00**
3.	**Silt size (0.075–0.002 mm)**	**84.00**	**26.80**
4.	**Clay size (< 0.002 mm)**	**10.00**	**72.20**
5.	**Liquid limit (%)**	**NP**	**226.30**
6.	**Plastic limit (%)**	**NP**	**32.00**
7.	**Plasticity index (%)**	—	**193.70**
8.	**Optimum moisture content (%)**	**26.20**	**33.60**
9.	**Maximum dry density (kN/m^3)**	**13.00**	**13.72**

Table –2 Chemical Compositions of Materials

Sl.No.	**Chemical Compositions**	**Fly Ash (%)**	**Bentonite (%)**	**Lime (%)**	**Gypsum (%)**
1.	**Loss on Ignition**	**1.14**	**9.52**	**7.85**	**12.83**
2.	**SiO_2**	**60.36**	**51.43**	**2.77**	**0.266**
3.	**Fe_2O_3**	**6.04**	**11.68**	**0.19**	**0.030**
4.	**Al_2O_3**	**28.21**	**19.16**	**1.66**	**0.039**
5.	**CaO**	**1.22**	**0.89**	**84.94**	**44.76**
6.	**MgO**	**0.87**	**2.70**	**1.75**	**0.078**

7.	**SO_3**	**0.65**	**Nil**	**0.11**	**41.55**
8.	**Na_2O**	**Few traces**	**3.49**	**Nil**	**0.286**
9.	**K_2O**	**Few traces**	**0.19**	**Nil**	**0.019**
10.	**pH**	**6.99**	**7.75**	**Nil**	**7.00**

Table –3 Properties of Water

Descriptions	**pH**	**Conductivity (mS/cm)**	**Total Dissolved Solids (g/L)**	**Dissolved Oxygen (mg/L)**
Water	**6.67**	**0.061**	**0.040**	**5.67**

B. Sample Preparation-

58 (fifty-eight) different types of samples are prepared by various mix proportions where fly ash is first mixed with varying percentages of bentonite (i.e., 20, 30, 40 and 50%) on a dry weight basis; after that fly ash is stabilized with 5, 10 and 15%lime. Restricted amounts of 0.5, 1 and 1.5% pure gypsum with respect to total weight of mixes are added, as the addition of higher percentages would lead to reduced durability with the formation of ettringite. In this way to get the paramount effects in the field, 58 mix designation is provided and total 60 numbers of compaction tests are conducted including fly ash and bentonite.

C. Programme-

Experimental programme

Atterberg's limits of fly ash and bentonite including combinations are carried out according to respective ASTM guidelines. Particle size-analysis of fly ash and bentonite as well as the specific gravity of fly ash and bentonite including all combinations is carried out as per ASTM guidelines. The MDD and OMC value corresponding key materials and each combination are determined by carrying out standard Proctor compaction test procedure as per ASTM (D698) guidelines.

III. EXPERIMENTAL RESULTS AND DISCUSSIONS

A. Experimental Results-

OMC and MDD values of standard Proctor compaction tests are obtained from the mixes of fly ash stabilized with bentonite percentages of 20, 30, 40 and 50 on the dry weight of fly ash. The range of MDD values is13.76 –14.68 kN/m^{3}and OMC shows in between 23.4 – 21.6 %. OMC and MDD values of the mixes of fly ash stabilized with lime percentages of 5, 10 and 15 are in the ranges between 22.3 – 21.7 % and 13.33 –13.27 kN/m^3, mixes of fly ash modified with very small amount of gypsum percentages of 0.5, 1.0 and 1.5 are in the ranges between 24.7 – 25.6 % and 12.58 – 12.68 kN/m^3 respectively. OMC and MDD values of the mixes stabilized with 20, 30, 40 and 50 percentages of bentonite and 5, 10 and 15 % of lime on dry weight basis of fly ash are in the ranges between 23.8 – 24.4 % and 13.53 – 13.86kN/m^3 for 5% lime, 23 – 24.1 % and 13.24 – 13.74 kN/m^3 for 10% lime, and 23.5 – 25.3% and 13.5 – 13.82 kN/m^3 for 15% lime respectively for all the percentages of bentonite. OMC and MDD values of the mixes of fly ash stabilized with 20, 30, 40 and 50% of bentonite, 5, 10 and 15 % of lime and modified with 0.5, 1.0 and 1.5 % gypsum on dry weight basis are in the ranges between 23.2 – 24.8% and 13.46 –14.10 kN/m^{3}for 5% lime and 0.5% gypsum, 24.2 – 24.2% and 13.59 –14.12 kN/m^3 for 5% lime and 1.0% gypsum, 23.5 – 23.0% and 13.6 – 14.06 kN/m^3 for 5% lime and 1.5% gypsum, 23.40 – 24.40% and 13.69 –14.22 kN/m^3 for 10% lime and 0.5% gypsum, 24.5 – 24% and 13.88-14.4 kN/m^3 for 10% lime and 1.0% gypsum, 23.2 – 22.4% and 13.54 – 14.18 kN/m^3 for 10% lime and 1.5% gypsum, 24.3– 22.3 % and 13.51 – 14.34 kN/m^{3}for 15% lime and 0.5% gypsum, 23.3 – 20.4% and 13.6–14.78 kN/m^3 for 15% lime and 1.0% gypsum, and 25.2 –19.9% and 13.38 – 14.9 kN/m^3 for 15% lime and 1.5% gypsum respectively for all the percentages of bentonite. All the values are shown in the plots.

B. Discussion on Experimental Results-

Effects of bentonite and lime on MDD and OMC of fly ash-bentonite mixes, and fly ash-lime mixes respectively have been discussed elaborately. Influence of gypsum on MDD and OMC of fly ash-gypsum mixes, the influence of gypsum on MDD and OMC of fly ash-gypsum mixes, the influence of lime on MDD and OMC of fly ash-bentonite-lime mixes and influence of gypsum on MDD and OMC of fly ash-bentonite-lime-gypsum mixes have also been discussed in this section.

Effect of bentonite on MDD and OMC of fly ash-bentonite mixes

In Figure 1, 20 to 50% bentonite clay is mixed with fly ash on a dry weight basis to stabilize fly ash, where bentonite clay plays an important role in controlling the compaction results which showsthe variation in OMC and MDD. Results show OMC lies between 23.4 – 21.6% and MDD 13.76 –14.68 kN/m^3 compacted at standard Proctor compaction energy. It implies that after addition of bentonite from 20 up to 50%with fly ash, the value of MDD increases and the value of OMC decreases with the increase of bentonite in the mixes. A similar trend was observed by [16]. It reveals that ion-exchange reactions occurring on mixing fly ash with clay soils produced flocculated and agglomerated soil fabric that lowered the MDD and increased the OMC of the mixes which were also found by [17].This may be attributed to improvement in a gradation of fly ash with the addition of bentonite which is predominated with silt-sized particles of fly ash. It also may be noticed that initial pH of bentonite soil with the addition of fly ash increases marginally. This may be due to the presence of small quantity of free lime present in fly ash. Further the increase in MDD may be due to the filling of voids by the finer particles of bentonite and the decrease in OMC with the increase in moisture content, the friction between the particles of fly ash-bentonite decreases; thereby particles come closer and also at the same time with increase in bentonite clay content, the air voids between the particles of fly ash of mixes are reduced and as a result of which air content decreases, and the degree of saturation increases which was also reported by [29].OMC decreases, and the irregular shape of fly ash particles and the presence of holes and crevices in the clinker like particles resulted in air being entrapped which helps the particles to come closer.

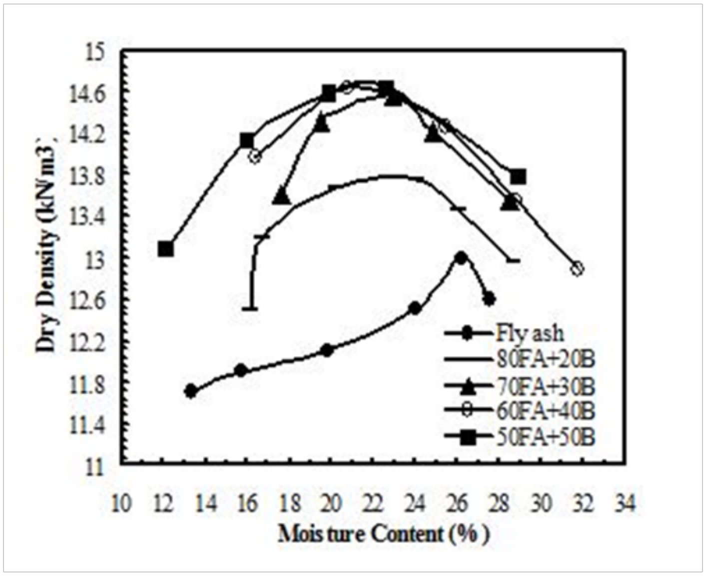


Figure 1. Dry density versus moisture content curves for fly ash and fly ash-bentonite mixes.

Effect of lime on MDD and OMC of fly ash-lime mixes

In Figure 2, fly ash stabilized with quicklime (CaO = 84.94%) of 5–15% to accelerate the fixation process of compaction matrix from the locally available market. Fly ash is a pozzolanic material, which is defined as siliceous or siliceous and aluminous, and therefore, its engineering behaviour can be improved by the addition of cement or lime. Here the plot depicted that after the compaction of fly ash stabilized with lime, the MDD of mixes range between 13.33 –13.09 kN/m^3 and OMC 22.30–23.40% which attributed that

MDD decreases with increase in OMC up to some extent i.e., from 5 to 10% lime addition, an opposite trend of results were observed by [18].It may be revealed that the fall in density is more significant at 10% lime addition than at 15% of lime. In lower ranges i.e., 10% lime reacts quickly with the fly ash and brings changes in base-exchange aggregation and flocculation, resulting in an increased void ratio of the mix leading to a decrease in the density of the mix. Addition of lime beyond this value (lime fixation point) is mainly utilized for pozzolanic reactions. The increase in optimum moisture content is probably on account of additional water held within the flocs resulting from flocculation due to the lime reaction. Similar observations were noticed by [20, 21, and 22].for different percentages of admixtures. After the addition of 15% lime, MDD obtained 13.09–13.27 kN/m^3 as well as OMC 23.4–21.7% found. It is noticed that with the further addition of lime to the fly ash, there is further increase in maximum dry density and decrease in optimum moisture content. The presence of extra lime content having 15%, may be the cause for this increased dry density due to more reduction in void spaces occupied by lime contents after compaction, reacting with previously untreated lime content chemically but without any additional water absorbance. After the chemical reaction, like base-exchange and aggregation, the dry unit weight of the mixes may be increased which result the increment in MDD. The decrease in optimum moisture content can be attributed to the increasing amount of fines which require more water content because of their larger surface area but the fines already inhabited the water into the pore spaces during the addition of 10% lime does not require further any additional water; somehow the reverse trend was obtained by [20,21, and 22].

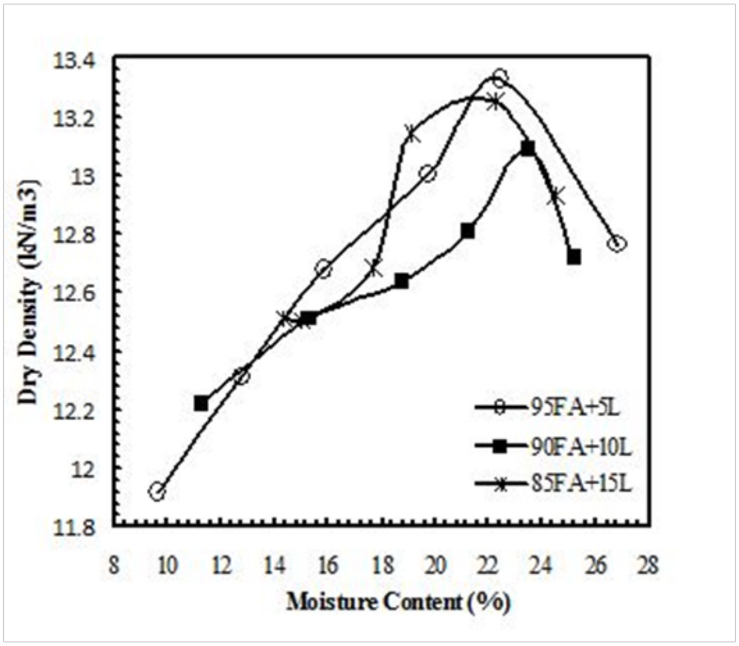


Figure 2. Dry density versus moisture content curves for fly ash and fly ash-lime mixes.

Influence of gypsum on MDD and OMC of fly ash-gypsum mixes

Figure 3 depicted the compaction effects of fly ash stabilized with gypsum of 0.5 to 1.5%. The MDD values are 12.58, 12.70 and 12.68 kN/m^3 for 0.5, 1.0 and 1.5% of gypsum mixed with fly ash, and OMC values in the range of 24.70–25.60%. Addition of only small amounts of gypsum such as up to 1.5% is considered, as higher amounts are known to have deleterious effects due to the formation of ettringite, a calcium tri sulfo aluminate attributed the increase in OMC and fluctuation in MDD. This can be attributed to the participation of even the relatively crystalline silica present in the coarser particle of fly ash in pozzolanic reaction. Gypsum is known to accelerate strength of process by altering the course of hydration of calcium silicate which is predominantly formed in the early hydration stages. Similar logic provided by [18].

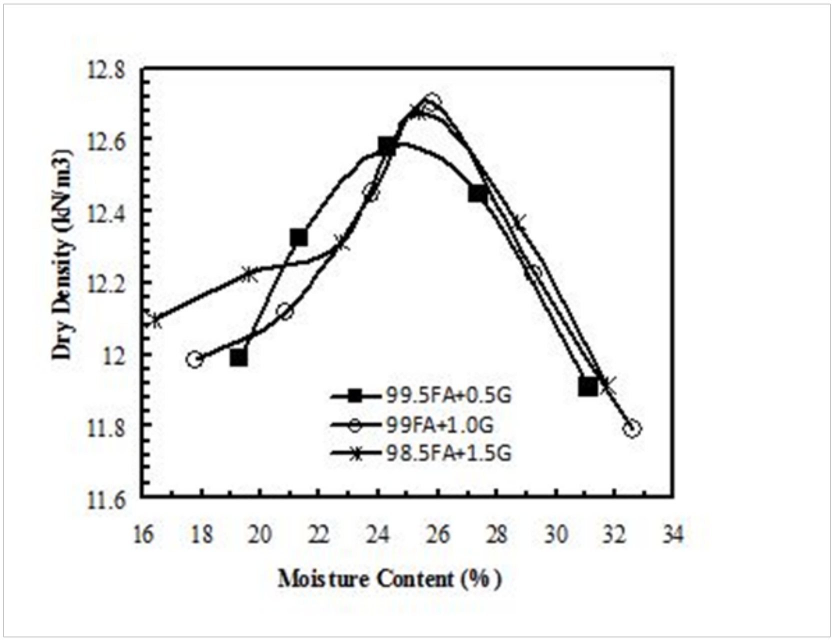
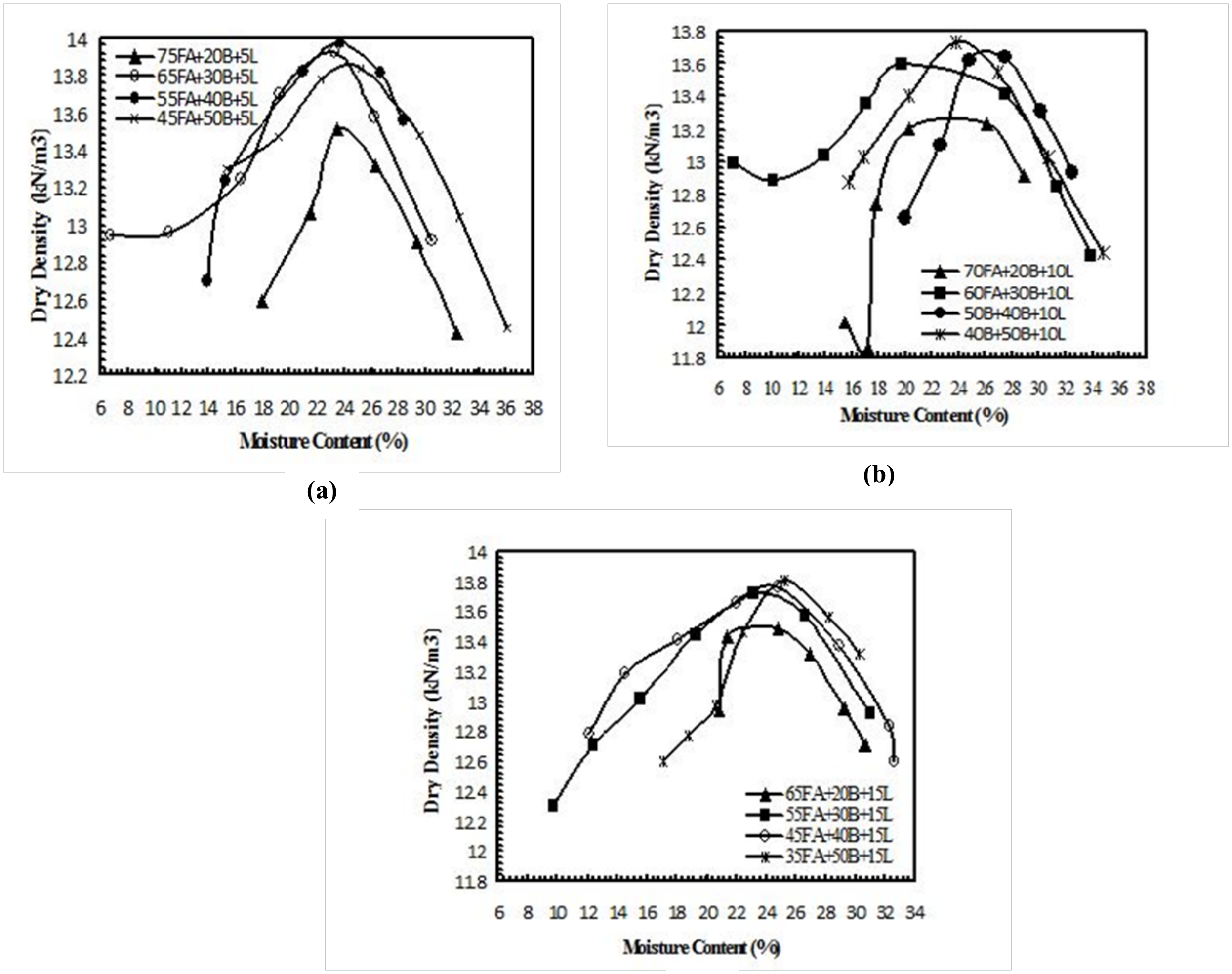


Figure 3. Dry density versus moisture content curves for fly ash and fly ash-gypsum mixes.

Influence of lime on MDD and OMC of fly ash-bentonite-lime mixes

Figure 4(a and b) illustrate fly ash mixed with 5 and 10 % of lime for different percentages of bentonite. From these figures, results obtained of MDD and OMC are 13.53–13.86 kN/m^3 and 23.80–24.40%, and 13.24–13.74 kN/m^3 and 23.00–24.10% for 5%, and 10% of lime respectively. These figures revealed that with the addition of lime content upto 10% for different percentages of bentonite with fly ash, MDD of mixes gradually decreases more or less for all the bentonite percentages i.e., 20–50% bentonite, and OMC generally fluctuated for the same compactive effort. This may be attributed that an immediate formation of cementitious products results in the reduction in dry density which also reducescompactibility as well as the density of the treated mix. A similar observation was found by [30] for different percentages of the mix. But in plot 8(c), with the further addition of 15% lime, values of MDD and OMC achieved are 13.50–13.82 kN/m^3 and 23.50–25.30%. It reveals that MDD and OMC of mixes gradually increase than that of 5% and 10% lime content. This may be recognized that at a higher percentage of lime with high silica content, when mixed with fly ashes of siliceous material and bentonite with higher percentages of lime, during the addition of 10% lime, increases the flocculation and agglomeration considerably which affects the OMC and MDD of the mixes. Which partially supports the concepts of earlier researchers, i.e., with an increase in lime content, the maximum dry density of soil-lime mixes decreased and optimum moisture content increased. The fall in density is more significant at lower percentages of lime than at higher percentages of lime with bentonite mix. In lower ranges (i.e., <4%) lime reacts quickly with the soil and brings changes in base exchange aggregation and flocculation, resulting in an increased void ratio of the mix leading to a decrease in the density of the mix. Addition of lime beyond this value (lime fixation point) given by [14] is mainly utilized for pozzolanic reactions. The increase in optimum moisture content is probably on account of additional water held within the flocs resulting from flocculation due to the lime reaction.

However, the increasing tendency of MDD is notified marginal after 10 % lime. This is due to the flocculation of bentonite clay minerals counteracting the effect of compaction effort. The gradual increment in MDD with the increase in lime content reflects enhanced the short time of reactions and the rearrangement of lime contents with siliceous materials of fly ash. Decreasing and increasing the tendency of OMC upto 15% lime may be the cause of interacting minerals of bentonite predominated the agglomeration and enhance to decrease in OMC and enhancement in water-holding capacity within flocs, which can accommodate water molecules resulting in an increase in OMC. [31]Reported that the addition of Ca $(OH)_2$ triggers the onset of pozzolanic reaction early by increasing the solubility of silica as it breaks the Si-O bonds in the silica-rich glassy phases of fly ash. The hydration of fly ash begins immediately with the depolymerization of glassy phases releasing alumina and silica thus increases the MDD of the mix.



(a)

(b)

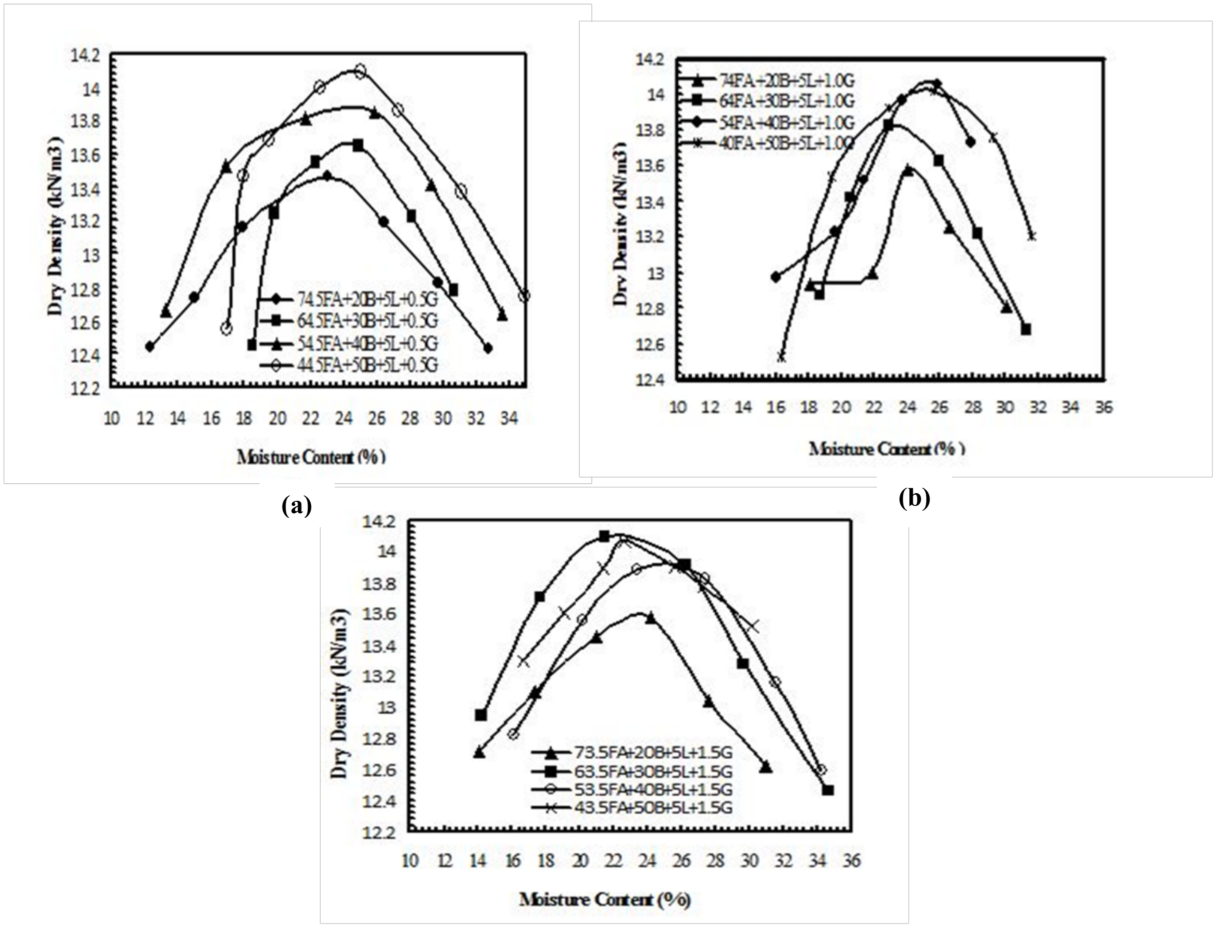
(c)

Figure 4. Dry density versus moisture content curves forfly ash stabilized with (a) 5 % of lime along with different percentages of bentonite; (b) 10 % of lime along with different percentages of bentonite; and (c) 15 % of lime along with different percentages of bentonite

Influence of gypsum on MDD and OMC of fly ash-bentonite-lime-gypsum mixes

Figures 5(a, b and c) and Figures 6 (a, b and c)reveal the changes of OMC and MDD of fly ash stabilized with 20−50% bentonite with 5% lime alone and after that modified with 0.5, 1.0 and 1.5% of gypsum ($CaSO_4.2H_2O$). To get the best affects on compaction results, gypsum percentages are slightly increases upto 1.5% from 1.0% of gypsum. From the plots, in general it is observed that MDD of the mix, which is the most important factors to enhance the engineering properties of filled materials, gradually increases from 13.46 to14.10 kN/m^3 for 0.5% gypsum,13.59 to 14.12 kN/m^3 for 1.0% gypsum, and 13.60–14.06kN/m^3 for 1.5% of gypsum. Whereas, OMC has no significant changes for these mixes. This may be recognized that majority of the earlier investigations on fly ash–lime mixes were focused on understanding basic chemistry of lime–pozzolana reactions and devising methods of accelerating rate of strength gain using an additive, like gypsum where strength is correlated with the dry density of mixes that also given by

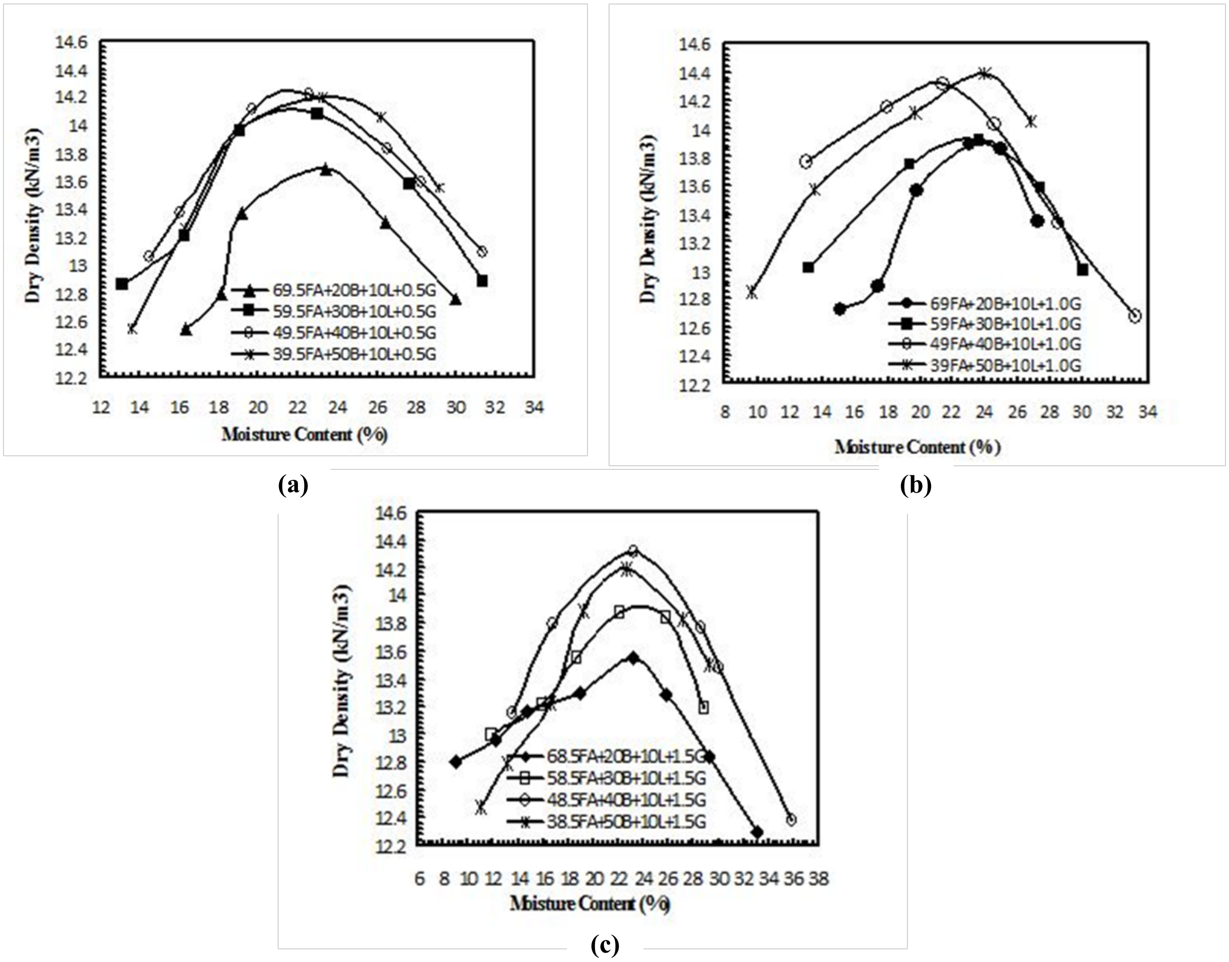
[32]. The maximum dry unit weight for the fly ash-bentonite mix decreased with the addition of lime. The decrease in the dry unit weight is attributed to the fact that lime reacts quickly with the bentonite which leads to an increase in the void ratio of the mix and leads toa decrease in the dry unit weight of the bentonite-lime mix. These observations are in agreement with [22].When fly ash is mixed with benonite, lime and gypsum mixes, the finer particles of fly ashes get placed in between the air gaps and get more densified, results in increment in MDD. The increase in the dry unit weight is accredited to the fact that the gypsum fills up the void spaces left out after the quick reaction of the bentonite with the lime. The similar trend of OMC, i.e., 23.40–24.40%, 24.50–24.00% and 23.20–22.40%, and MDD, i.e., 13.69–14.22 kN/m^3, 13.88–14.4kN/m^3 and 13.54–14.18 kN/$m^3$is also tracked for 10% of lime for the additive 0.5%, 1.0% and 1.5% of gypsum respectively. The effect of altered OMC on compaction results of these mixes can be described considering the chemical reactions of fly ash contents, lime, bentonite and gypsum in the mixes and also from the micro-structural study, i.e., from SEM images and EDAX analysis.



(a) **(b)**

(c)

Figure 5. Dry density versus moisture content curves for fly ash stabilized with (a) 5% of lime and 0.5% gypsumalong with different percentages of bentonite; (b) 5% of lime and 1.0% of gypsum along with different percentages of bentonite; and (c) 5% of lime and 1.5% of gypsum along with different percentages of bentonite

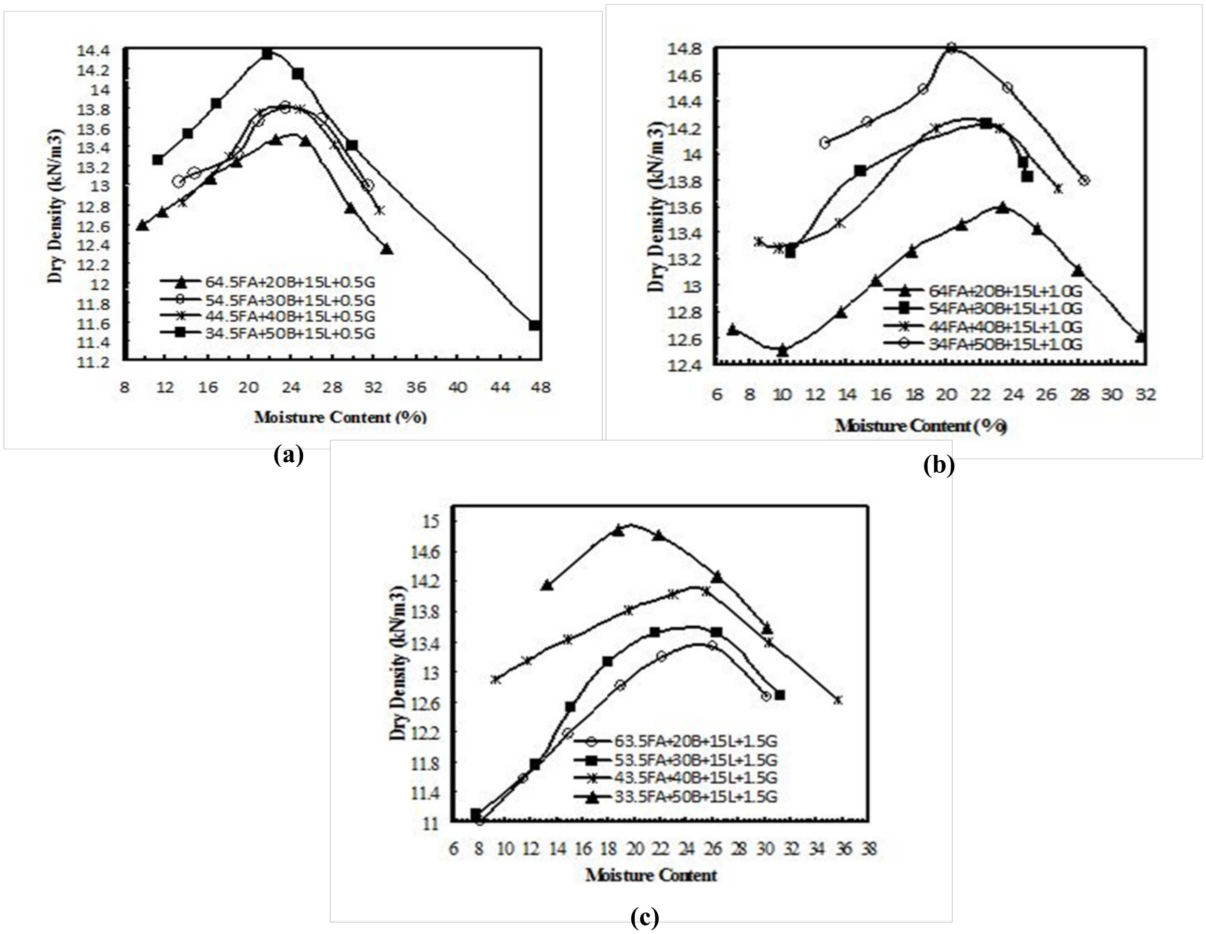


(a) **(b)**

(c)

Figure 6. Dry density versus moisture content curves for fly ash stabilized with (a) 10% of lime and 0.5% of gypsum along with different percentages of bentonite; (b) 10% of lime and 1.0% of gypsum along with different percentages of bentonite; and (c) 10% of lime and 1.5% of gypsum along with different percentages

Figures 7(a, b and c)illustrated the effect of OMC and MDD of fly ash stabilized with different percentages of bentonite such as 20 to 50% with 15% lime, and modified with 0.5%, 1.0% and 1.5% of gypsum,where in every case MDD gradually increases , i.e., 13.51–14.34 kN/m^3, 13.60–14.78 kN/m^3 and 13.38–14.9 kN/m^3, and reached upto maximum values of MDD with the increase in bentonite percentages, and OMC gradually decreases which achieved maximum efficient results of MDD and OMC for the entire study and may be attributed that the larger amount of lime percentages (greater than 10% lime may give more proficient results of MDD and OMC) enhance the compaction process and the influence of gypsum accelerate the whole process with the higher amount of finer particles may react quickly with the bentonite particles and siliceous materials of fly ashes after immediate contact of water results flocculation and agglomeration process and the water content of mixes reduces.

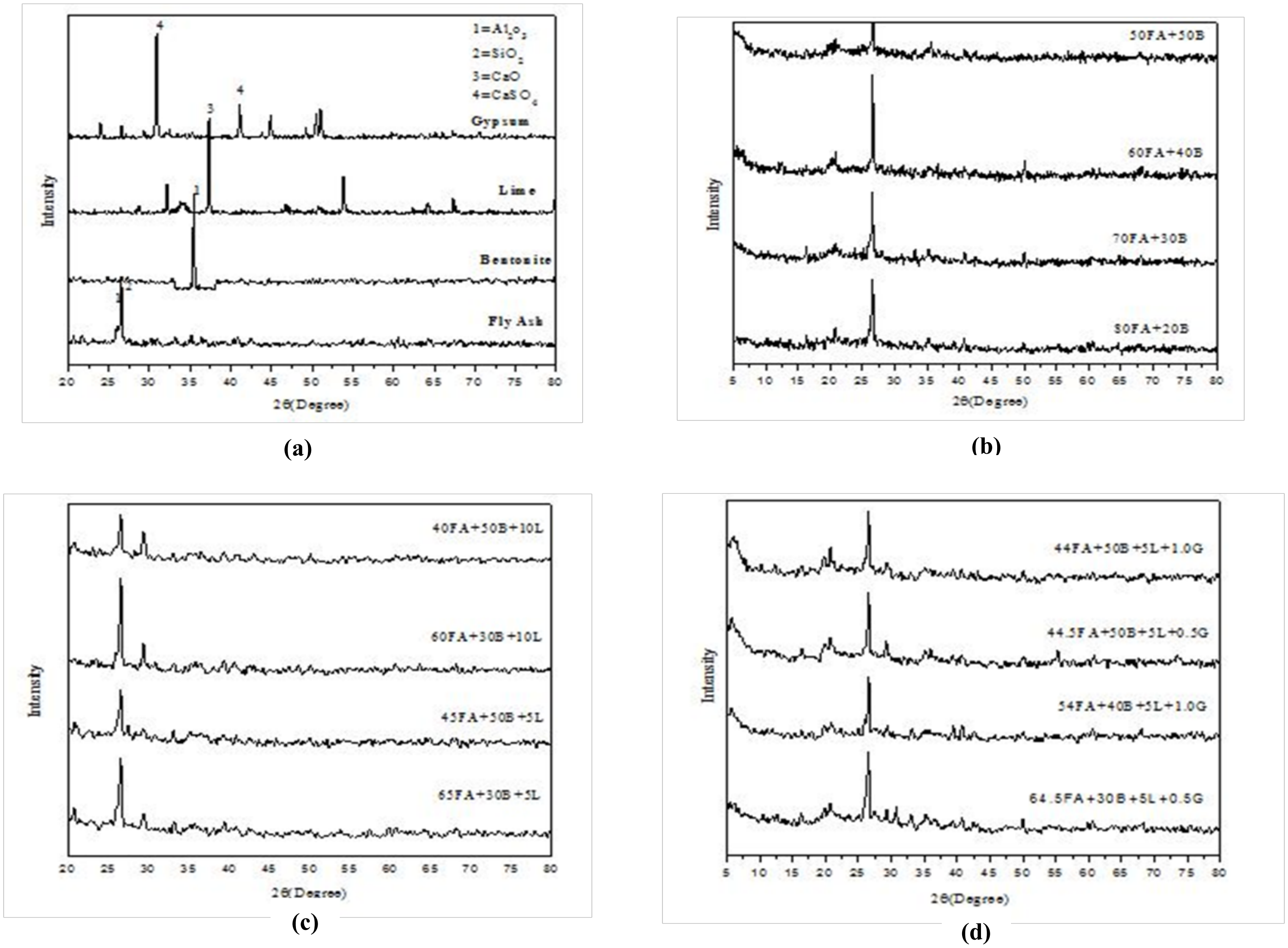


(a) **(b)** **(c)**

Figure 7. Dry density versus moisture content curves for fly ash stabilized with (a) 15% of lime and 0.5% of gypsum along with different percentages of bentonite; (b) 15% of lime and 1.0% of gypsum along with different percentages of bentonite; and (c) 15% of lime and 1.5% of gypsum along with different percentages of bentonite.

IV. ANALYTICAL RESULTS AND DISCUSSIONS

The analytical analysis (i.e., XRD, SEM and EDAX) are carried out on selective samples of mix with raw materials collected from fractured pieces of oven dried sample taken after getting the MDD and OMC of raw materials like fly ash, bentonite, lime and gypsum and with few mix proportions of materials such as (80FA+20B),(70FA+30B), (60FA+40B), (50FA+50B), (65FA+30B+5L),(45FA+50B+5L),(60FA+30B+10L),(40FA+50B+10L),(64.5FA+30B+5L+0.5G),(44.5FA+50B+5L+0.5G), (54FA+40B+5L+1.0G), (44FA+50B+5L+1.0G) mixes are used for XRD analysis along with (70FA+30B), (50FA+50B), (95FA+5L), (85FA+15L), (99.5FA+0.5G), (98.5FA+1.5G), (45FA+50B+5L), (40FA+50B+10L), (35FA+50B+15L), (64.5FA+30B+5L+0.5G), (54FA+40B+5L+1.0G) and (53.5FA+40B+5L+1.5G), for SEM and EDAX analysis including with individual raw materials such as fly ash and bentonite using standard Proctor compaction tests. SEM and EDAX techniques are employed to study the change in surface morphology and the chemical composition of the samples. XRD is accomplished with Bruker D8 Advance model diffractometer to identify changes in mineralogy and crystalline phases. XRD is carried out with Cu-K_α radiation (λ=1.5406 Å) at 1.2°/minute speed for Bragg's angle2θ ranges 5° to 80°. The diffractometer is operated at an applied voltage of 40 kV and the applied current of 40 mA, where JCPDS (Joint Committee of diffraction society) software is used to analyse data obtained from diffractometer. The procedure mode of XRD is browsing incidence with a step size of 0.02° per second within $2\theta = 5^{\circ}–80^{\circ}$ scan range. Microstructure and images of samples are performed with SEM and EDAX techniques respectively. The constitutive elemental analysis is carried out by attaching an energy dispersive spectrometer to the scanning electron microscope. This focuses an electron beam on a point on the sample. X-ray photographs generated by the sample are converted into electrical impulses and the chemical elements present are determined. The JEOL JSM-7600F FEG-SEM with resolution 1.0–1.5 nM at 15–1.0 kV and voltage of 0.1 to 30 kV is used for the SEM and EDAX studies. Before the microstructural examination, the fractured surfaces of the samples for SEM study are gold-coated, the sample is coated with 10 mA thin layer gold-palladium for 300 s using a sputter coater and the software is used for image analysis is PC-SEM . INCA Energy software is used to analyse the chemical composition of samples with EDAX techniques.



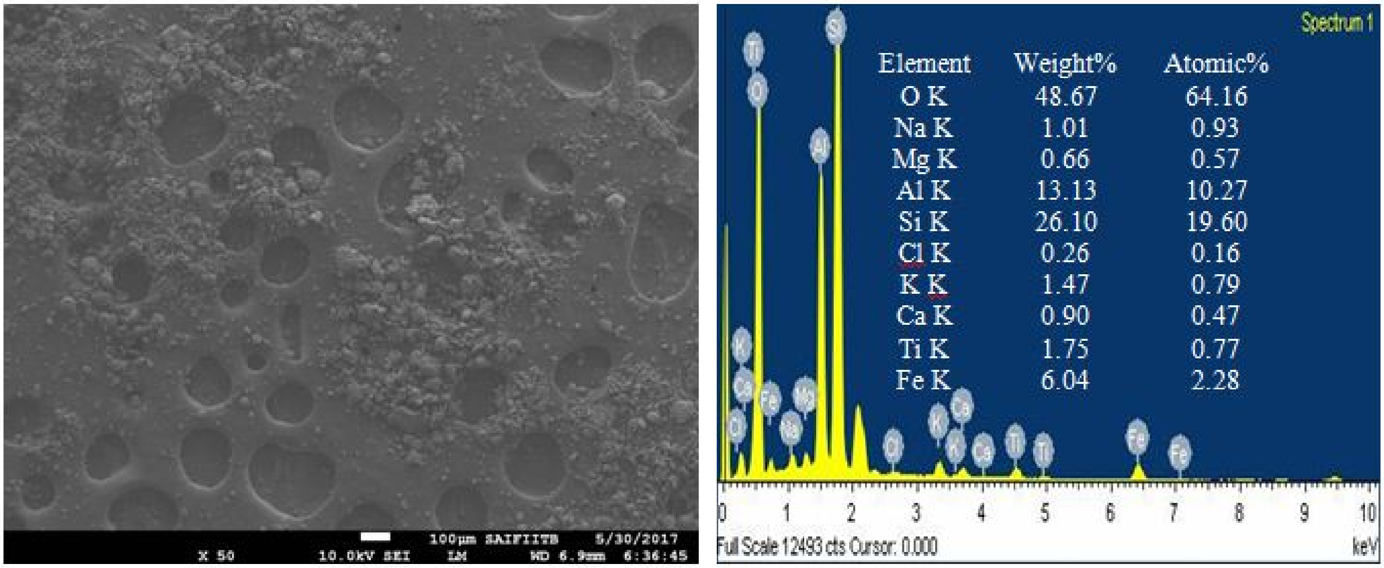
(a) (b)

(c) (d)

Figure 8. XRD analysis of (a) fly ash, bentonite, lime and gypsum; (b), fly ash mixed with bentonite; (c) fly ash mixed with bentonite and lime; and (d) fly ash mixed with bentonite, lime and gypsum

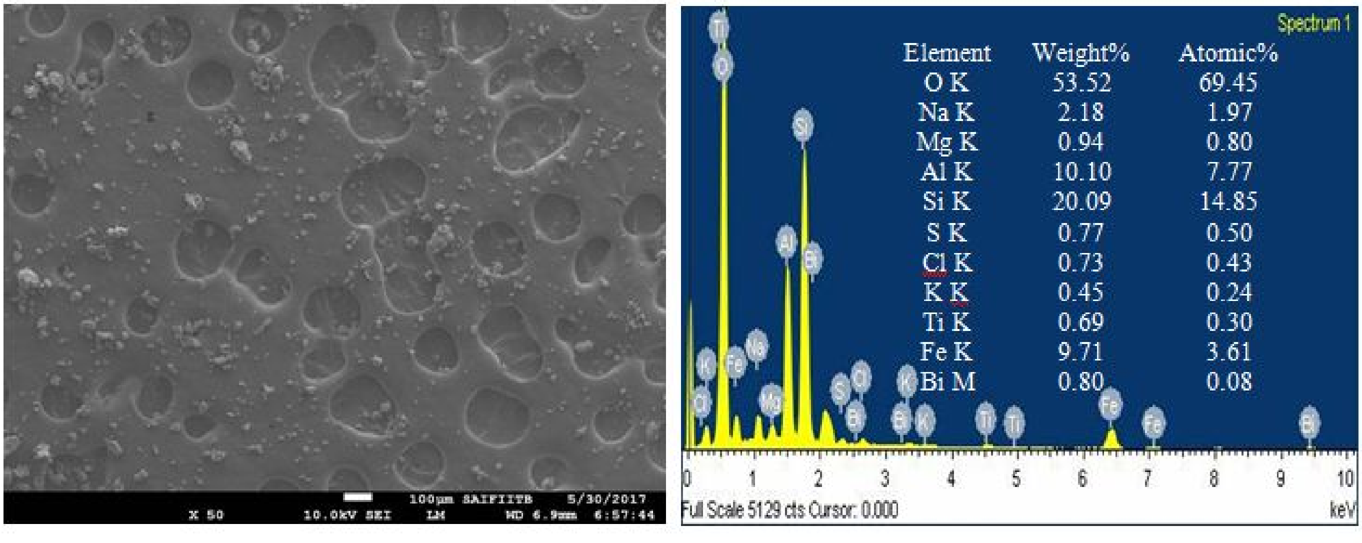
A. Discussion on Analytical Results –

The fractured surfaces of compacted oven-dried samples are examined for the analytical investigation and it is found from the Figures. 8 (a), (b), (c) and (d) with Figures 9 to 20 x-ray diffraction and scanning electron microscope, the mineralogical and micro-structural development of fly ash particles ensures mainly aluminium oxide (Al_2O_3), i.e., mullite, silicon di-oxide (SiO_2), i.e., quartz, and aluminium silicate (Al_2SiO_5) compound from all the major peaks as fly ash is a by-product of powdered coal combustion and also exhibits pozzolanic activity from the large presence of alumina-silica glass; the particles are maximum round and oval shaped which occupies the coarser parts of fly ash which is found from SEM analysis. Bentonite confirms the only mullite with spherical shaped structures. The X-ray diffraction (XRD) analysis of dry powder lime confirmed that all major peaks indicate the presence of hydrated calcium hydroxide [Ca $(OH)_2$] with the formation of calcium oxide (CaO) and calcium sulfate ($CaSO_4$) presents in lime and anhydrous gypsum with a different flower, like, structure. From the mixes, i.e., fly ash stabilized with bentonite, fly ash stabilized with bentonite and lime, fly ash stabilized with bentonite, lime and gypsum together describes that clay minerals are natural pozzolanas that react with lime to produce cementitious calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H),and similar observation highlighted by [33]. The reaction of fly ash with lime produces calcium silicate hydrate similar to that encountered in soil-lime reactions [34].The compounds of XRD analysis of fly ash, bentonite, lime and gypsum are mainly of mullite, quartz, $Ca(OH)_2$, $CaSO_4$**.**



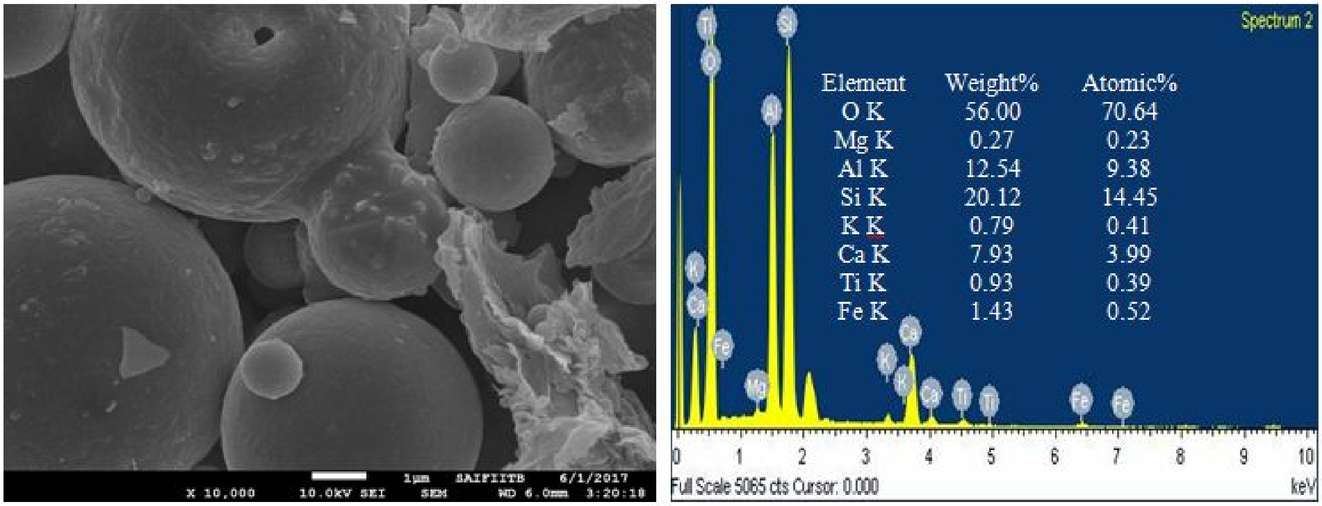
Element	Weight%	Atomic%
O K	48.67	64.16
Na K	1.01	0.93
Mg K	0.66	0.57
Al K	13.13	10.27
Si K	26.10	19.60
Cl K	0.26	0.16
K K	1.47	0.79
Ca K	0.90	0.47
Ti K	1.75	0.77
Fe K	6.04	2.28

Figure 9. SEM image and EDS analysis of (70FA+30B) using spectrum



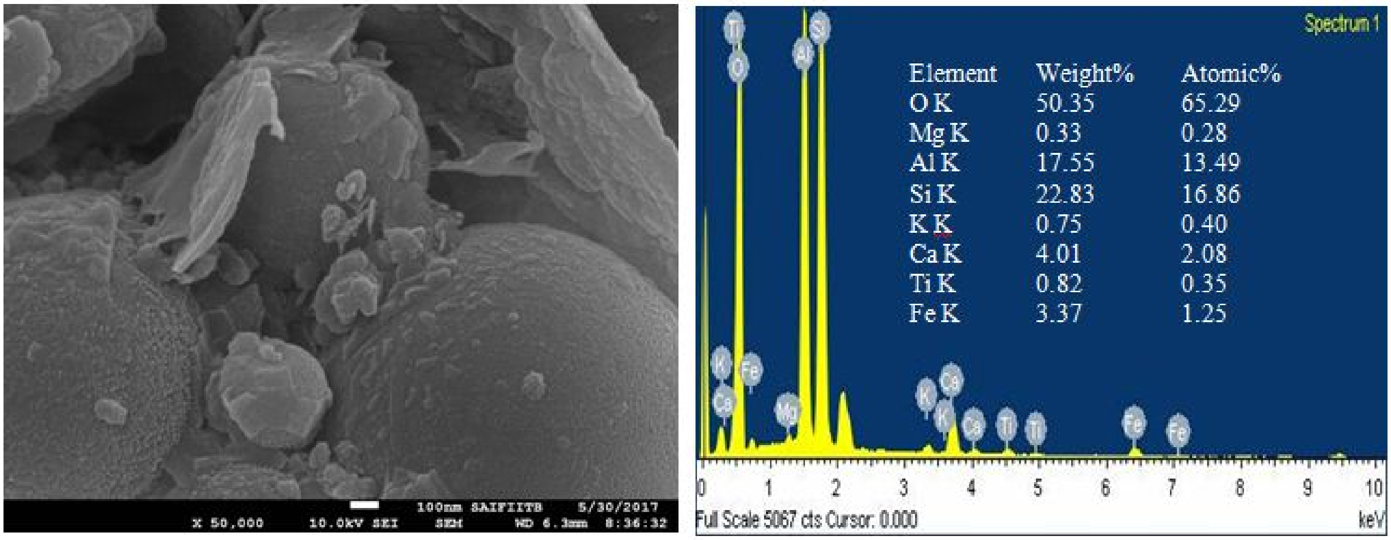
Element	Weight%	Atomic%
O K	53.52	69.45
Na K	2.18	1.97
Mg K	0.94	0.80
Al K	10.10	7.77
Si K	20.09	14.85
S K	0.77	0.50
Cl K	0.73	0.43
K K	0.45	0.24
Ti K	0.69	0.30
Fe K	9.71	3.61
Bi M	0.80	0.08

Figure 10. SEM image and EDS analysis of (50FA+50B) using spectrum



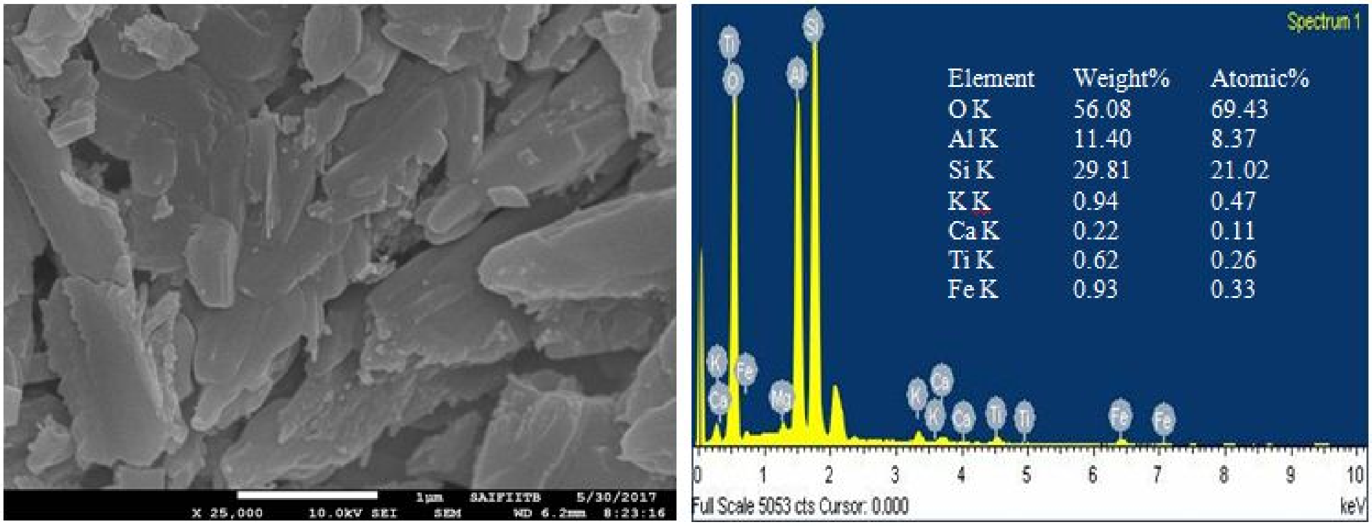
Element	Weight%	Atomic%
O K	56.00	70.64
Mg K	0.27	0.23
Al K	12.54	9.38
Si K	20.12	14.45
K K	0.79	0.41
Ca K	7.93	3.99
Ti K	0.93	0.39
Fe K	1.43	0.52

Figure 11. SEM image and EDS analysis of (95FA+5L) using spectrum



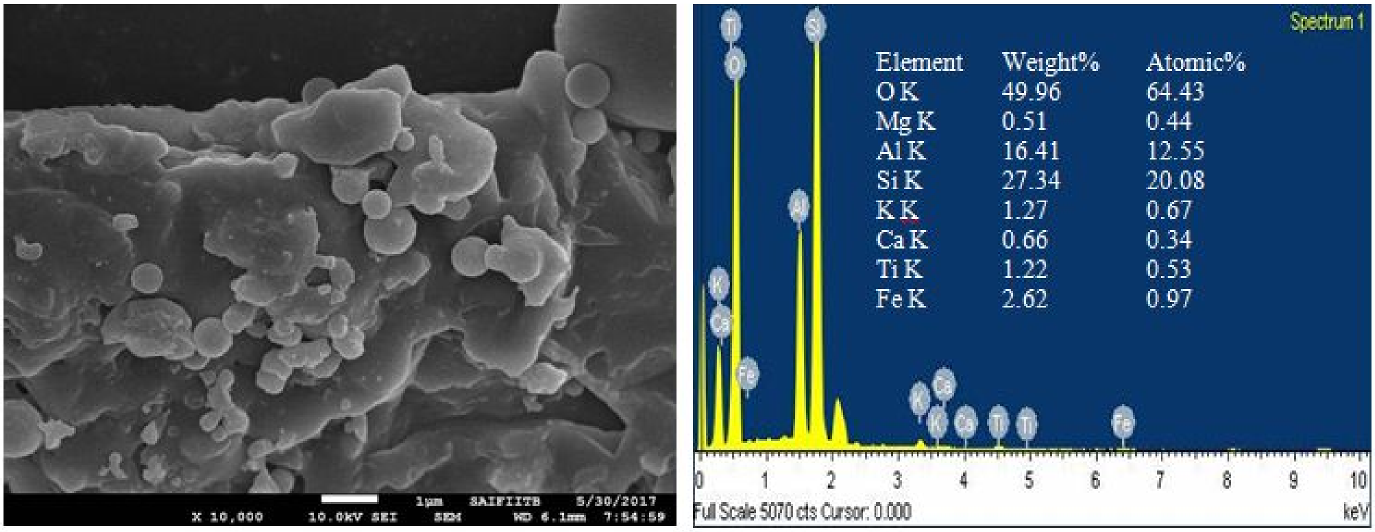
Element	Weight%	Atomic%
O K	50.35	65.29
Mg K	0.33	0.28
Al K	17.55	13.49
Si K	22.83	16.86
K K	0.75	0.40
Ca K	4.01	2.08
Ti K	0.82	0.35
Fe K	3.37	1.25

Figure 12. SEM image and EDS analysis of (85FA+15L) using spectrum



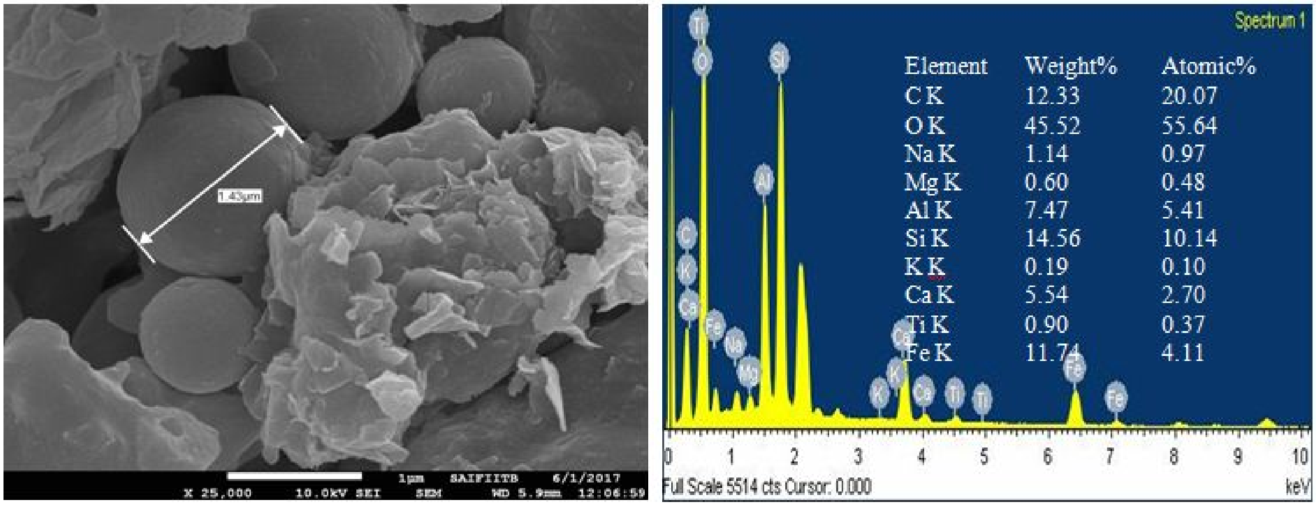
Element	Weight%	Atomic%
O K	56.08	69.43
Al K	11.40	8.37
Si K	29.81	21.02
K K	0.94	0.47
Ca K	0.22	0.11
Ti K	0.62	0.26
Fe K	0.93	0.33

Figure 13. SEM image and EDS analysis of (99.5FA+0.5G) using spectrum



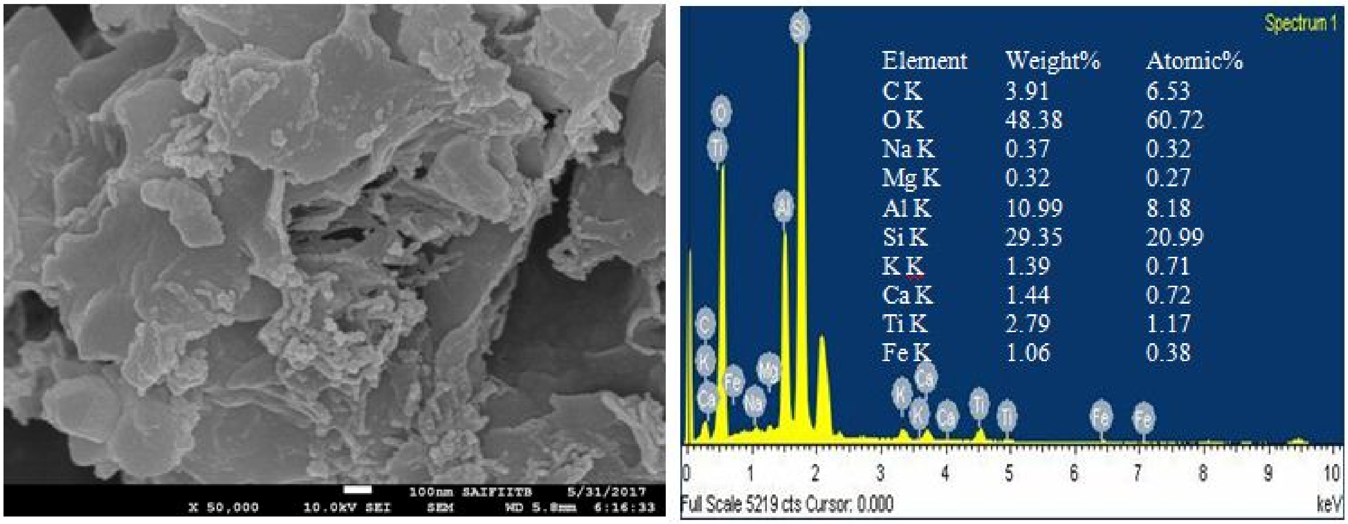
Element	Weight%	Atomic%
O K	49.96	64.43
Mg K	0.51	0.44
Al K	16.41	12.55
Si K	27.34	20.08
K K	1.27	0.67
Ca K	0.66	0.34
Ti K	1.22	0.53
Fe K	2.62	0.97

Figure 14. SEM image and EDS analysis of (98.5FA+1.5G) using spectrum



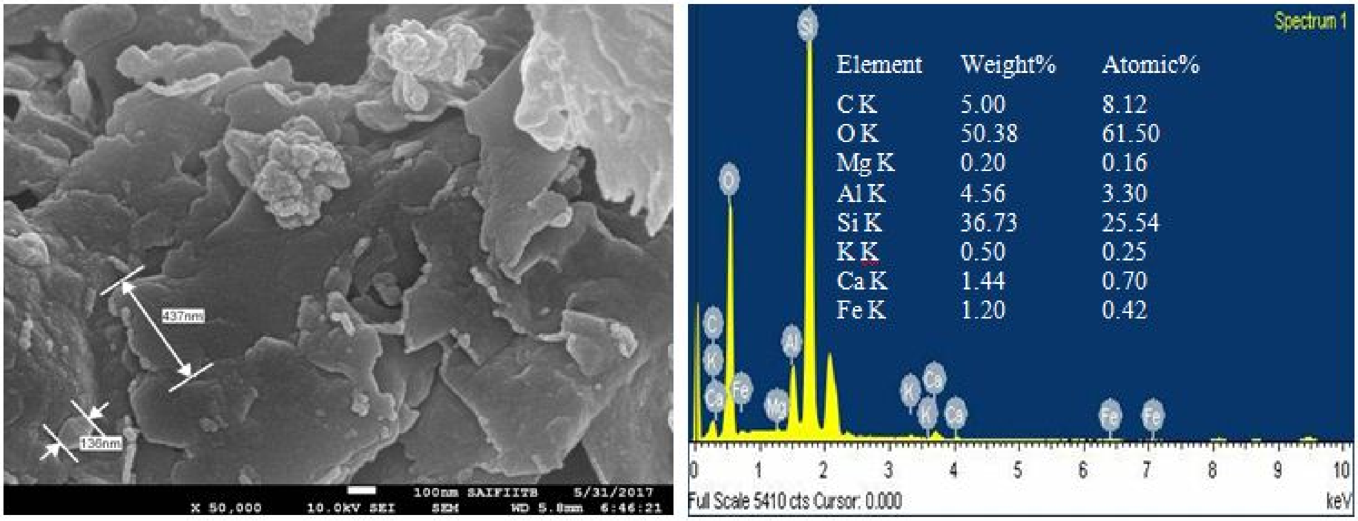
Element	Weight%	Atomic%
C K	12.33	20.07
O K	45.52	55.64
Na K	1.14	0.97
Mg K	0.60	0.48
Al K	7.47	5.41
Si K	14.56	10.14
K K	0.19	0.10
Ca K	5.54	2.70
Ti K	0.90	0.37
Fe K	11.74	4.11

Figure 15. SEM image and EDS analysis of (45FA+50B+5L) using spectrum



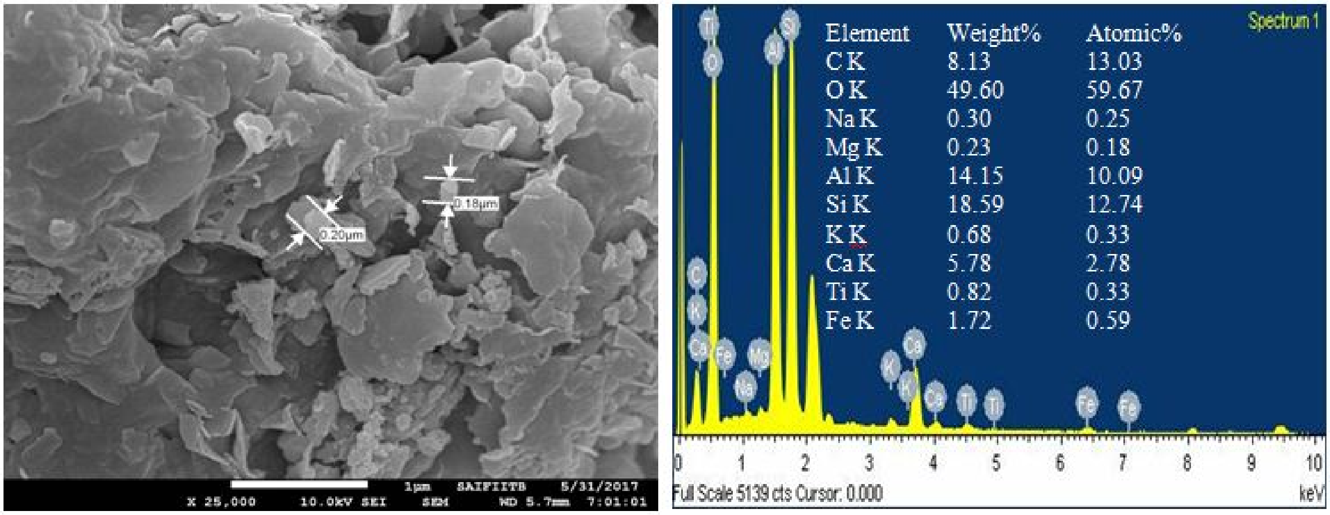
Element	Weight%	Atomic%
C K	3.91	6.53
O K	48.38	60.72
Na K	0.37	0.32
Mg K	0.32	0.27
Al K	10.99	8.18
Si K	29.35	20.99
K K	1.39	0.71
Ca K	1.44	0.72
Ti K	2.79	1.17
Fe K	1.06	0.38

Figure 16. SEM image and EDS analysis of (40FA+50B+10L) using spectrum



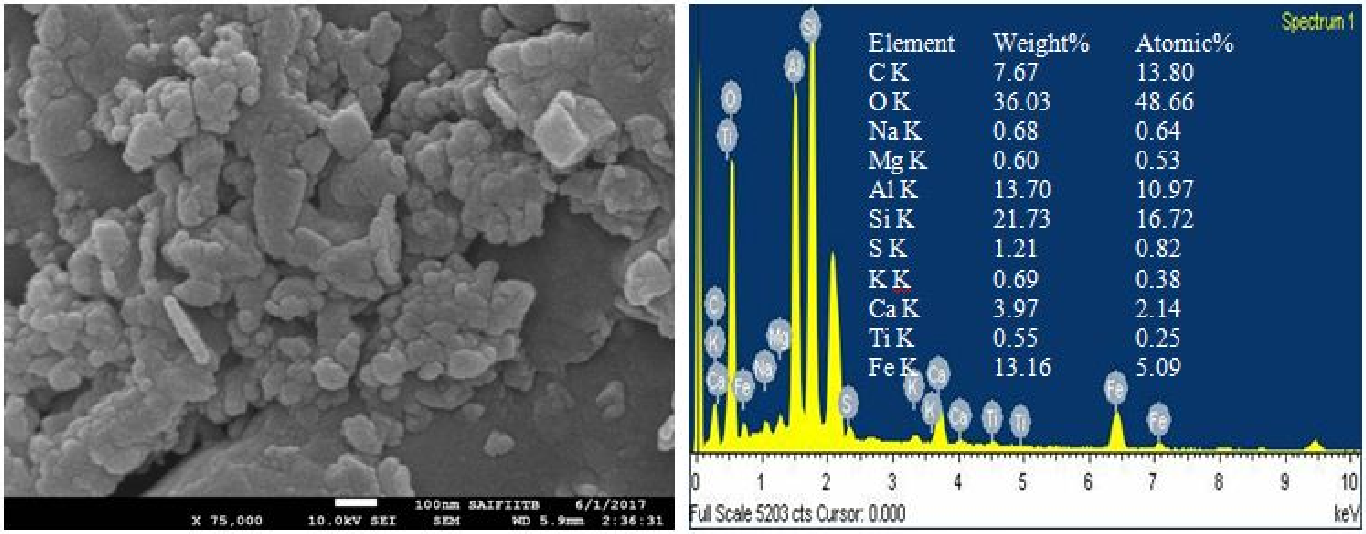
Element	Weight%	Atomic%
C K	5.00	8.12
O K	50.38	61.50
Mg K	0.20	0.16
Al K	4.56	3.30
Si K	36.73	25.54
K K	0.50	0.25
Ca K	1.44	0.70
Fe K	1.20	0.42

Figure 17. SEM image and EDS analysis of (35FA+50B+15L) using spectrum



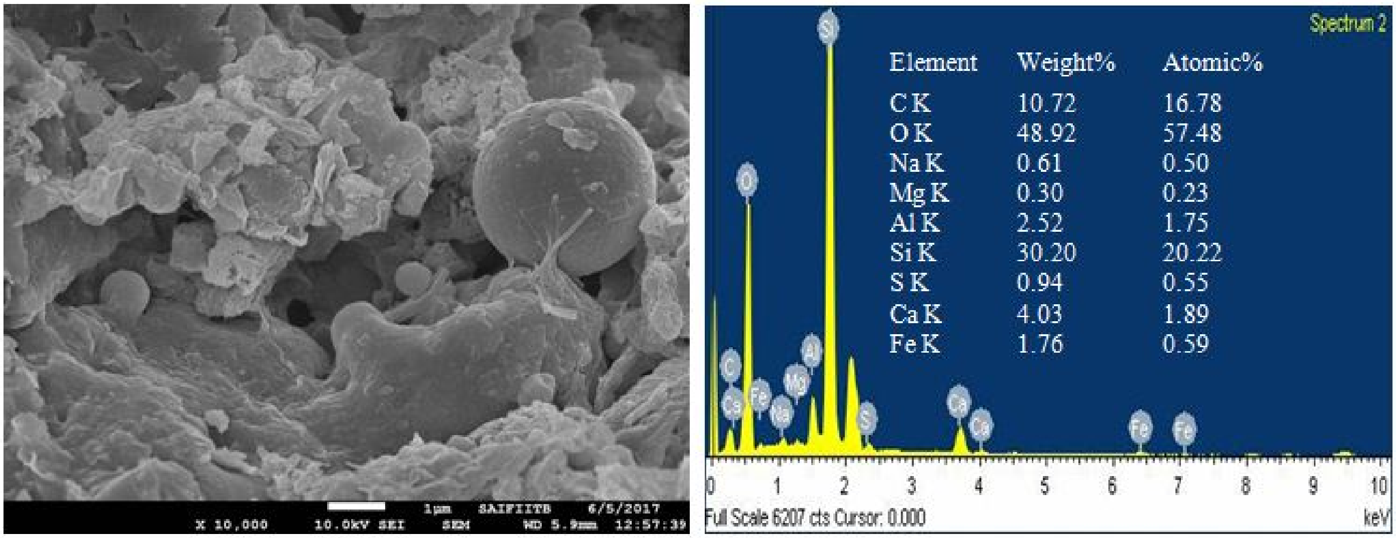
Element	Weight%	Atomic%
C K	8.13	13.03
O K	49.60	59.67
Na K	0.30	0.25
Mg K	0.23	0.18
Al K	14.15	10.09
Si K	18.59	12.74
K K	0.68	0.33
Ca K	5.78	2.78
Ti K	0.82	0.33
Fe K	1.72	0.59

Figure 18. SEM image and EDS analysis of (64.5FA+30B+5L+0.5G) using spectrum



Element	Weight%	Atomic%
C K	7.67	13.80
O K	36.03	48.66
Na K	0.68	0.64
Mg K	0.60	0.53
Al K	13.70	10.97
Si K	21.73	16.72
S K	1.21	0.82
K K	0.69	0.38
Ca K	3.97	2.14
Ti K	0.55	0.25
Fe K	13.16	5.09

Figure 19. SEM image and EDS analysis of (54FA+40B+5L+1.0G) using spectrum



Element	Weight%	Atomic%
C K	10.72	16.78
O K	48.92	57.48
Na K	0.61	0.50
Mg K	0.30	0.23
Al K	2.52	1.75
Si K	30.20	20.22
S K	0.94	0.55
Ca K	4.03	1.89
Fe K	1.76	0.59

Figure 20. SEM image and EDS analysis of (53.5FA+40B+5L+1.5G) using spectrum

V. CONCLUSION

An experimental study is carried out to investigate the potential of fly ash after stabilized with bentonite and lime modified with pure gypsum ($CaSO_4.2H_2O$). The compaction and plasticity characteristics of fly ash stabilized with bentonite (20–50%), lime (5–15%) and pure gypsum (0.5–1.5%) alone or in combination with these materials in different proportions including raw materials are studied through laboratory experiments. Compaction tests are conducted by standard Proctor compaction energy. The mineralogical and microstructural analysis of these materials after compaction is made as an analytical analysis by XRD and scanning electron microscope (SEM) for the detailed study of mineralogy and microstructure. Based on the experimental as well as analytical results following conclusion may be outlined:

- The obtained dry densities with optimum moisture contents are not abrupt; this nature is functional for field application and in a construction site.
- In general, MDD shows increasing trend of all the mixes, which is correlated with flowing properties of soil media to provide a liner in the landfilling site to prevent the contamination of leachate materials from the ground surfaces,where OMC does not follow any specific trend with respect of MDD as well as mixes. The variation of dry density with moisture content of fly ash stabilized with bentonite of 20–50% and lime of 5–15% modified with pure gypsum of 0.5–1.5% by dry weight show more efficient results compare to that of unstabilized fly ash of this study.
- The mineralogical and microstructural analysis of raw materials with few mixes of fly ash-bentonite, fly ash-lime, fly ash-gypsum, fly ash-bentonite-lime, and fly ash-bentonite-lime-gypsum shows the structural position and changes of minerals and chemical components produced with different shapes and sizes at different peaks.
- Different percentages of stabilizers such as bentonite(20–50%), lime(5–15%) with modifier gypsum(0.5–1.5%)selected for stabilization is on the basis of compaction effect, economy, time duration and environmental impact.
- The addition of gypsum only enhances the whole process quickly rather than considerable changes in the properties of samples.
- Composite is very much effective for field applications as land fill, embankment and road sub-base; mix contains maximum percentage (i.e., 33.5-99.5%) of fly ash which is waste materials and abundantly available with free of cost, and very little percentages of bentonite (i.e.,20-50%), lime (i.e., 5.0-15%) and gypsum (i.e., 0.5-1.5%) added to it. It's a new concept and very much useful in the field of geotechnical engineering.

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