Influences of curing conditions on strength and microstructure of lime-amended fly ash

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ABSTRACT: The influence of curing conditions on strength and microstructure of lime-amended fly ash is presented in this paper. A series of compaction, UCS and CBR tests are carried out by varying the lime content from 0 to 12%. The curing of specimen is done for 0 to 60 days under temperature of 10ºC to 90ºC. The test results showed that an increase of either lime content or curing period increases the UCS and CBR values. The curing temperature also influences the rate of strength gain and formation of hydration products. Lower temperature cured specimens showed gradual increase in strength even up to 60 days whereas the specimen cured at higher temperature stabilized much earlier. Mineralogical analysis showed new amorphous products of CSH and CASH compounds. The microstructural study indicates formation of greater amount of needle like structure of ettringite which reduces the inter-particle voids and takes a major role in strength enhancement.

1 INTRODUCTION

The socio-economic growth of a country is mostly influenced by the different modes of transport piecing together. Antecedently, for various construction works like road base/sub-base, embankment the soil is used as a primary constituent. Rapid industrialization coupled with increasing power demand generates huge amounts of industrial wastes that cause serious environmental pollution and scarcity of valuable land for their disposal. To mitigate these problems, the industrial wastes are used as a substitute to the earthen material after augmenting the essential geotechnical parameters. In general, the road base/sub-base, embankment, etc. are directly exposed to the atmosphere and are subjected to different environmental conditions.

Several studies on the stabilization of fly ash with mineral and chemical admixtures, the effects of curing conditions and molding conditions on strength and durability have been reported in the literature. Application of fly ash in construction works like embankments, dykes, and road subgrade is demonstrated by DiGioia & Nuzzo (1972) and Gray & Lin (1972). Lav et al. (2000) observed that the cement and lime-stabilized fly ash specimens produce similar hydration products which are responsible for gain in strength over time because the available voids between the particles were filled by growing hydrates over time; this contributes an increase in strength. Ghosh & Subbarao (2001) studied the surface morphology of lime stabilized fly ash specimen and found that formation of gel-like substance of C-S-H and C-A-H occurs at higher doses of lime. Also, needle-like structures of ettringite are visible at long curing period. X-ray diffraction (XRD) studies are employed to identify the crystalline phases in the stabilized fly ash. Arora & Aydilek (2005) observed that the strength of a class F fly ash amended with the addition of soil-cement and soil-lime is highly dependent on the curing period, compaction energy, cement content, and water content. Stabilization of the low lime fly ash with lime and gypsum gives effective control over hydraulic conductivity and leachate characteristics Ghosh & Subbarao (2007). Singh et al. (2008) reported a CBR value of 105% for the compacted fly ash–GBFS–cement (52:40:8) mixture, indicating its suitability for use in base and sub-base courses in highway pavements with proper combinations of raw materials. Maitra et al. (2010) reported that the rate of decrease in free lime content for water cured compacts/samples was maximum up to 50–55 days, whereas in steam curing the rate of decrease in free lime content was maximum up to a curing period of 10 h. So, the order of the reaction and the reaction rate constant values were higher for steam cured samples compared to the water cured samples. Reddy & Gourav (2011) suggested that the lime-pozzolana reaction required very long curing period to achieve appreciable strength under ambient temperature conditions. The reaction rate and mechanism of pozzolanic reaction of fly ash vary with curing temperature. Elevated curing temperatures accelerate the rate of reaction linearly Narmluk & Nawa (2014).

Extensive research has been done on the utilization of fly ash in different fields of civil engineering. However, very few attempts have been made to evaluate the effect of temperature on the various geotechnical properties, and microstructural changes of the lime stabilized fly ash. In the present investigation an effort has been made to examine the effects of elevated curing temperature on strength and microstructure of lime-amended fly ash.

2 MATERIALS

Fly ash used for this study was obtained from Rourkela steel plant (RSP), India in dry condition. The physical properties and chemical composition of the fly ash are given in Table 1 and 2 respectively. The principal constituents of the fly ash are silica, alumina and iron oxide. Calcium present in the fly ash is less than 20%. So, according to ASTM specification C 618-89 (1992), this fly ash belongs to a Class-F category. The grain size distribution of the fly ash is determined as per IS: 2720 Part-IV, 1985 and the gradation curve is given in (Fig.1). It contains 84% fines (<75μm) and is non-plastic in nature. The Cu and Cc values are 8.34 and 2.08, respectively, indicating uniform gradation of the sample. The sample is classified as 'SP' as per unified soil classification system. Commercial grade hydrated lime of 90.2% purity was used in this study.

3 METHODOLOGIES

The engineering properties such as compaction characteristics, unconfined compressive strength, and California bearing ratio (CBR) value of different mixes of fly ash-lime are evaluated to examine the suitability of these materials in highway construction. The percentages of lime in the mix varied as 0%, 2%, 4%, 8% and 12%. The compaction characteristics of these mixes are determined using two different compactive energies i.e. 595 kJ/m³ and 2674 kJ/m³. From the compaction curves the OMC and the maximum dry unit weight are found out for different mixes and energy levels. The specimens prepared at OMC with lime contents of 0%, 2%, 4%, 8% and 12% with compaction energy of 595 kJ/m³ are designated as L0, L2, L4, L8, and L12 whereas these are designated as H0, H2, H4, H8, H12 for a compaction energy of 2674 kJ/m³. The specimens for UCS tests are cured at different temperatures under sealed and unsealed conditions. Accordingly the specimens are labeled as a following common coding system. For example the sealed and unsealed specimen prepared at 2% lime are designated as SL_2 and UL_2 respectively. Similarly for ST_{10}/UT_{10} represents specimen prepared at a sealed/unsealed condition and are cured at temperature of 10°C whereas SC7/UC7 represents the sealed/unsealed specimen cured for 7 days respectively.

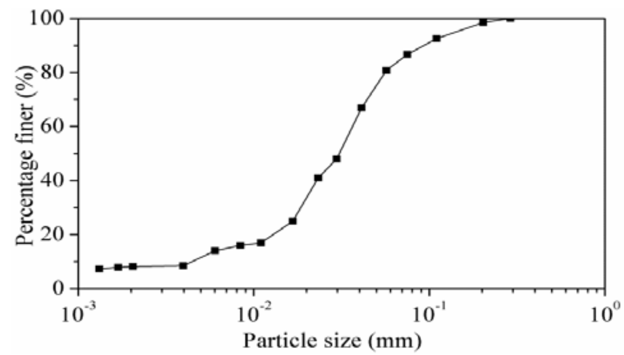

Figure 1. Gradation curve of fly ash

Table 1. Physical properties of fly ash

Physical parameters	Properties
Colour	Light grey
Shape	Rounded/sub-rounded
Silt & clay size (%)	84
Fine sand size (%)	12
Medium sand size (%)	0
Coarse sand size (%)	0
Uniformity co-efficient (Cu)	8.34
Coefficient of curvature (Cc)	2.08
Specific gravity	2.38
Plasticity index	Non-plastic
Classification (USC)	SP (poorly graded sand)

Table 2. Chemical composition of fly ash

Elements	Composition (%)
SiO_2	59.2
Al_2O_3	17.9
Fe_2O_3	9.5
CaO	3.2
MgO	1.3
SO_4	1.2
Unburnt carbon	7.0
Others	0.7

3.1 *Compaction test*

The moisture content, dry unit weight relationships for different fly ash-lime mixes were found as per IS: 4332 (Part-II and III), 1967. The compaction tests were carried out using two different compactive energies i.e. 595 kJ/m³, and 2674 kJ/m³. From the dry unit weight and moisture content relationship, optimum moisture content (OMC) and maximum dry unit weight were determined for different fly ash-lime mixes at both the compaction energies.

3.2 *UCS test*

Specimens for UCS tests were statically pressed to their respective maximum dry unit weights at OMC. For each condition six identical test specimens are prepared. These are of size 50 mm in diameter and 100 mm in height. Three samples are cured under unsealed conditions and other three are in sealed conditions. Sealed samples were wax coated for 10^0C, 25^0C, 45^0C temperature and covered with heat resistant wrapper for 90ºC temperature and cured for 0, 7, 15, 30, and 60 days in a temperature controller unit. All the specimens were sheared at an axial strain rate of 1.25 mm/min till failure of the sample and the average value was reported. All these tests are performed according to IS 4332 (Part V) 1970.

3.3 *CBR test*

For CBR test the specimens are prepared in a rigid metallic cylindrical mould having diameter of 150 mm and a height of 175 mm. The samples are statically compressed to their respective MDD at OMC. These specimens are cured for 7 and 30 days under moist environment at an average curing temperature of 27^0C. For determination of soaked CBR value the specimens are soaked in water for 4 days before testing. The tests are conducted according to IS: 2720(Part XVI)-1987 at a uniform strain rate of 1.2 mm/min.

3.4 *Microscopic study*

The X-ray diffraction tests are used to determine the hydration product that appeared in the lime treated specimen after specified curing periods by using Philips X'PERT high score equipment. The samples are prepared from the fractured UCS test specimens and then soaked in an anhydrous ethanol to stop further hydration. Microscopic study were undertaken to examine the morphology and microstructure of hydrated specimens. These are done by a JEOL 6480 LV scanning electron microscope. XRD is used to detect any new crystalline phases formed, and SEM/EDAX techniques are employed to follow changes in microstructure and composition during curing.

4 RESULTS AND DISCUSSIONS

4.1 *Moisture content ~ dry density relationship*

The changes in OMC and maximum dry unit weight with the lime content are given in (Figs 2-3) respectively. Addition of low amount of lime (2%) to the fly ash results in a substantial increase of OMC value, further increase of lime reduces the OMC value marginally. However, these OMC values are higher than that of the virgin fly ash. Similarly, addition of low amount of lime (2%) to the fly ash results in a substantial decrease of maximum dry unit weight, further increase of lime increases the maximum dry unit weight value marginally. However, these maximum dry unit weight values are higher than that of the virgin fly ash. Addition of lime brings about the colloidal reactions and minor flocculation or aggregation occurs in the sample. This results in a reduction of maximum dry unit weight value and increase of OMC value. As expected an increase in compaction energy causes an increase of maximum dry unit weight values and reduction in OMC values. The maximum dry unit weight of the virgin fly ash is found to change from 1.12 to1.236 kN/m^3 with change in compaction energy from 595 kJ/m^3 to 2674 kJ/m^3, whereas the OMC is found to decrease from 40.5 to 33 %.

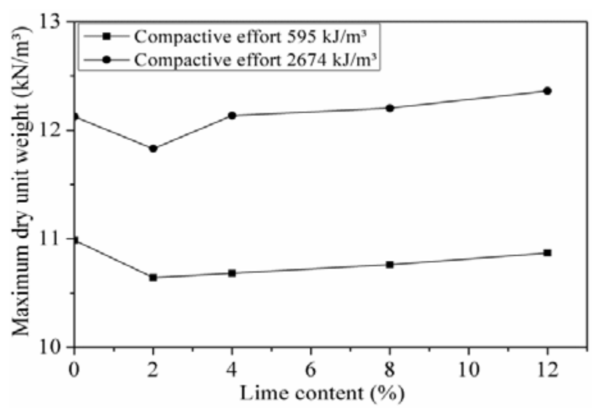

Figure 2. Variation of dry unit weight with lime content

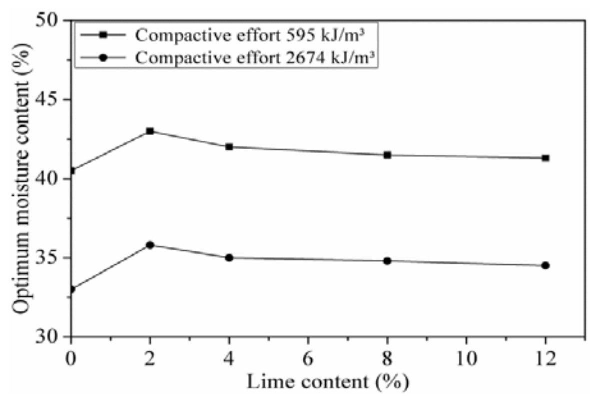

Figure 3. Variation of OMC with lime content

4.2 *Unconfined compressive strength*

4.2.1 *Effects of compaction energy*

The effect of compaction energy on the unconfined compressive strength with varying lime content and curing period is presented in (Fig.4). The specimen prepared at 595 kJ/m^3 energy and cured for 60 days with 12% lime give a UCS value of 3.5 MPa whereas specimen prepared at 2674 kJ/m^3 energy give 4.5 MPa strength. The higher UCS value of the specimen with higher compactive effort is attributed to the closer packing of particles, resulting in an increase of interlocking between the particles. A closer packing is also responsible in increasing the cohesion component in the sample that results in a gain in strength. Furthermore, the doses of lime as well as curing period play a vital role to gain appreciable unconfined strength. At low lime content (< 4%) the amount of lime added mostly utilized in colloidal reaction and sufficient amount of lime is not left for the pozzolanic reaction. Hence, at low lime contents the gain in UCS value not appreciable even though the samples are cured for longer time.

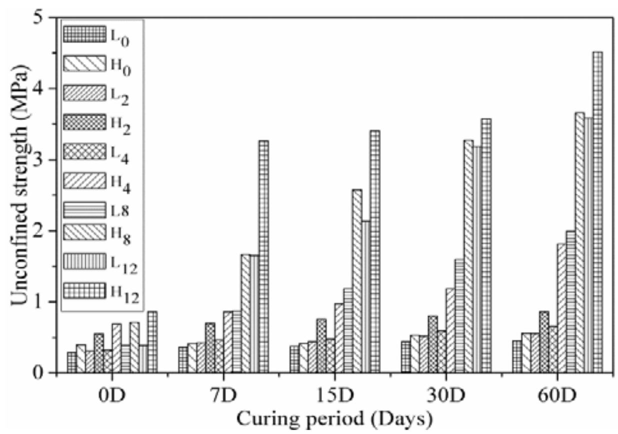

Figure 4. Variation of UCS value with lime content and curing period

4.2.2 *Effects of lime content*

The effect of lime content on UCS values of the sealed and unsealed specimens at varying curing temperature is as shown in (Fig. 5). It is observed that in both sealed and unsealed case at low lime content of 0 to 4%, the unconfined compressive strength is found to change from 0.5 to 2 MPa indicating that the gain in strength is not so remarkable with lesser dosages of lime. In the other way at higher dosage of lime a noticeable unconfined compressive strength is found beyond 4% which enhances the strength from 2.5 MPa to 10 MPa. This conveys that about 4% of lime is used for colloidal type of reaction and lime in excess to this amount is utilized for pozzolanic reaction. Furthermore, it is clearly visible that the unsealed specimen gives better strength than sealed specimens with higher amount of lime content. Since in case of sealed samples the molding water content is not sufficient to complete the pozzolanic reaction process; but in case of unsealed specimens it consumes additional moisture from the external source which helps to complete the whole reaction process. Also, the higher percentages lime facilitates better pozzolanic reaction and formation of more cementitious gel of CSH and CASH which binds the particles together which causes strength improvement. The figure shows almost a linear trend of strength gain up to 12% lime signifying that the reaction process is still continuing and it may possibly be go further with more incorporation of lime.

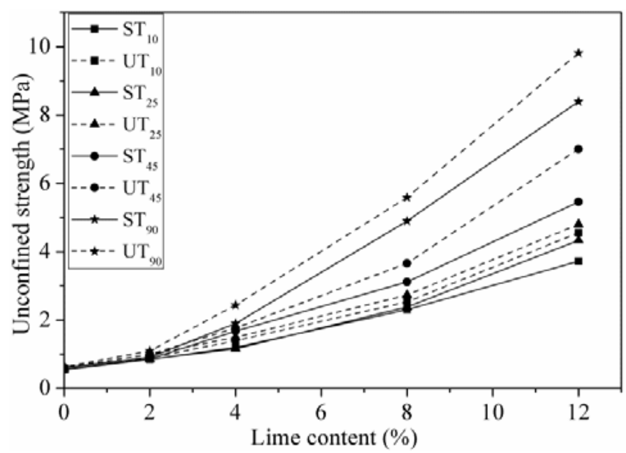

Figure 5. Variation of UCS values with lime content

4.2.3 *Effects of curing period*

The effects of curing period on UCS values at two different curing temperatures of 10^0C and 90^0C are summarized in (Figs 6-7). From (Fig. 6) it is observed that for both cases (sealed and unsealed) the early age of curing (≤ 15 days) is practically insignificant to give reasonable amount of strength, even, at higher dosage of lime. As the curing period increases with higher dosage of lime more vigorous pozzolanic reaction takes place which improves the strength with time. Moreover, it is seen that the specimens cured at low temperature give a gradual rising slope of strength up to 60 days of curing and this trend describes that the optimal state of stabilization has not been reached, with further increment in curing period it may possibly offers optimum state of stabilization.

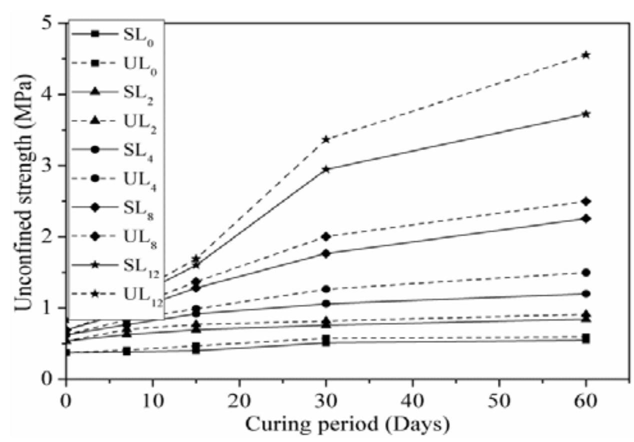

Figure 6. Variation of UCS values with curing period for specimens cured at 10^0C

In the other hand, from (Fig. 7) it is noticed that the unconfined compressive strength increases with increase in curing period for both sealed and unsealed condition. Whereas, unsealed specimens deliver better pozzolanic reaction than sealed one at higher temperature (90^0C) and longer curing period. For both the cases (sealed and unsealed) due to high temperature, there is a substantial improvement in strength observed at an early age of curing. Approximately similar trend of strength continues up to 60 days of curing at higher dosage of lime content, which attributes an optimal stage of stabilization. But at low lime content minor increasing slope is noticed which indicates that the specimen needs more time to complete the total reaction process to achieve better strength. In addition to that, high temperature makes a favorable condition for better pozzolanic reaction and results in early age of strength.

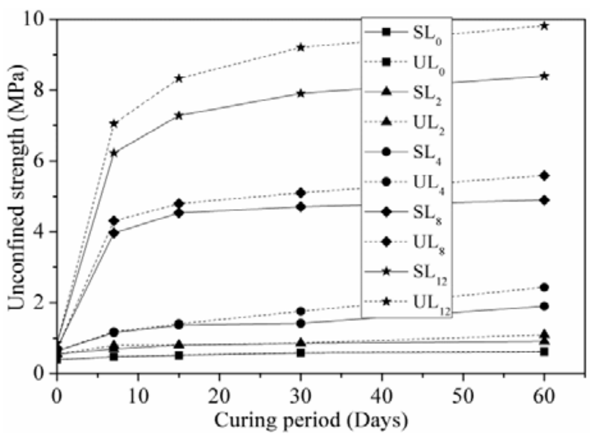

Figure 7. Variation of UCS values with curing period for specimens cured at 90^0C

4.2.4 *Effects of curing temperature*

The effects of curing temperature over the unconfined compressive strength are illustrated in (Fig. 8). The unconfined compressive strength is very much susceptible to curing temperature and it is found to influence the strength of both the sealed and unsealed specimens. It is observed that at low temperature (10^0C) the unconfined strength changes from 1 to 2.5 MPa with a change in the curing period from 7 to 60 days. It shows that longer curing period helps to complete the pozzolanic reaction at low temperature. In contrast to that, the 60 days compressive strength changes from 2.5 to 6 MPa when the curing period is changed from 25 to 90^0C. A crossover effect is noticed at about 60^0C (Fig. 8) between sealed and unsealed specimens; indicating that at high temperature favors a better pozzolanic reaction and the water added during moulding of specimens are insufficient to complete the pozzolanic reactions in the sealed specimens. According to test results it is expected that with further increase in temperature may increase the unconfined strength or it will show an optimal state of stabilization.

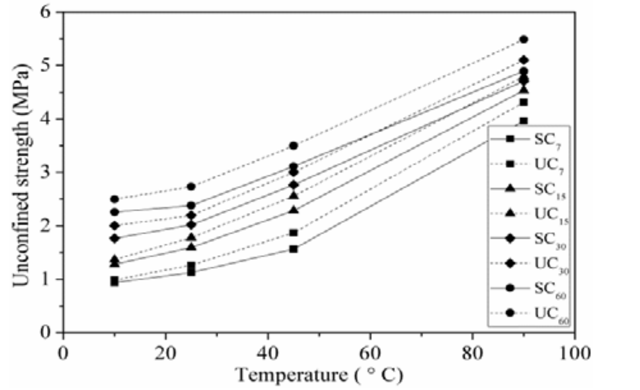

Figure 8. Variation of UCS values with curing temperature at 8% lime content

4.3 *California bearing ratio*

The variations of CBR value with increase in lime content and curing period are presented in (Figs 9-12). It is clearly demonstrates that the CBR value increases with increase in compaction energy, lime content as well as curing period. The 7 days soaked CBR value of virgin fly ash is relatively low ranging from 1.3% to 5.8% as compaction energy increases from 595 to 2674 kJ/m^3. For a given compactive energy the unsoaked CBR value is found to be more than the soaked CBR value. This phenomenon occurs mainly due to higher frictional resistance of fly ash particles under unsoaked conditions in addition to the development of pseudo-cohesion forces that develops in the unsaturated fly ash specimen. This offers a higher penetration to the CBR plunger and thus higher CBR values. On the contrary, when the same fly ash samples are soaked by maintaining the similar placement conditions, they exhibited very low values of CBR. This can be attributed to the destruction of capillary forces under soaked conditions. However, the fly ash treated with 8% of lime gives comparatively higher CBR value of 53.4% and 120.5% at compaction emerges of 595 to 2674 kJ/m^3 respectively after 7days of soaking. Further, when the sample subjected to a curing period of 30 days the CBR value considerably increases due to more pozzolanic reaction of lime with increase of curing period.

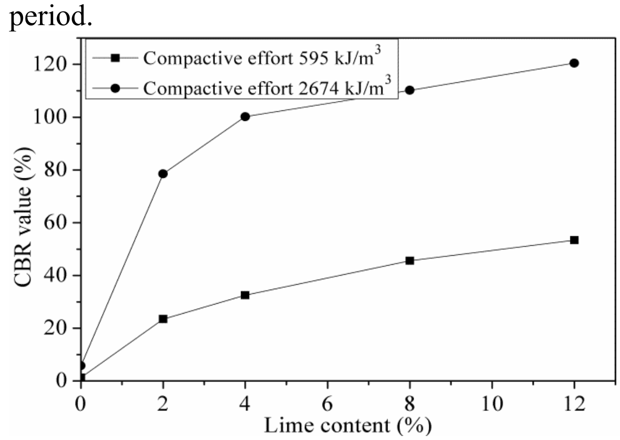

Figure 9. Variation of soaked CBR with lime content at 7days curing

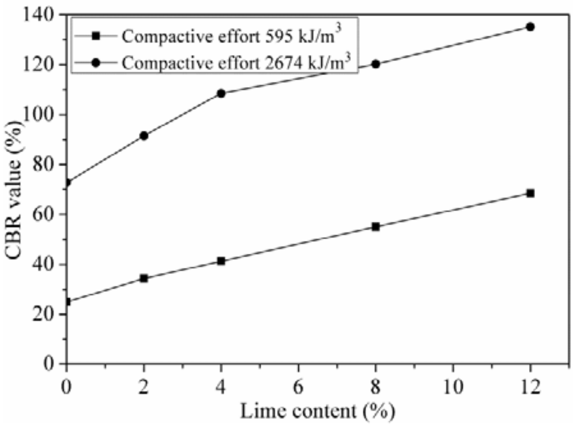

Figure 10. Variation of unsoaked CBR with lime content at 7days curing

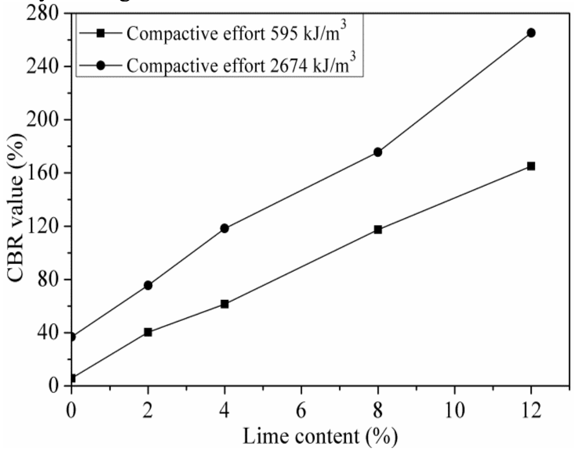

Figure 11. Variation of soaked CBR with lime content at 30days curing

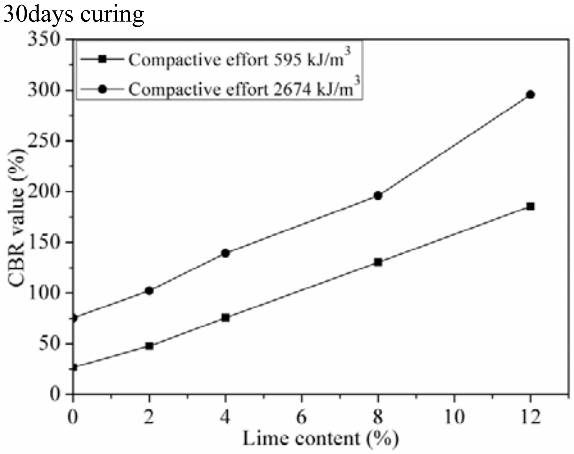

Figure 12. Variation of unsoaked CBR with lime content at 30days curing

4.4 *Chemical composition and micro-structure*

4.4.1 *X-ray diffraction study*

The X-ray diffractographs gave inconclusive substantiation with respect to the formation of new crystalline phases. These X-ray diffractographs of fly ash specimen treated with 8% lime at cured at 10^0C, 25^0C, 45^0C and 90^0C temperatures for 30 days are presented in (Fig.13). It is observed that the samples stabilized at lower temperature (10^0C) give very weak reflection of crystalline phase ; more number of ettringite peak ($2\Theta = 33.2^0$, 40.37^0 and d-spacing=2.696Å, 2.23 Å) and showing very few peaks of calcium aluminum silicate hydrate ($2\Theta = 47.55^0$ and d-spacing=1.912Å). An increasing in the curing temperature from 25^0C to 45^0C results additional peaks of low intensity of ettringite. Those peaks of ettringite get disappeared at higher temperature (90^0C) and it transformed to a gel like substance of calcium silicate hydrate/ calcium aluminum silicate (d-spacing = 1.541Å, $2\Theta = 60.003^0$).

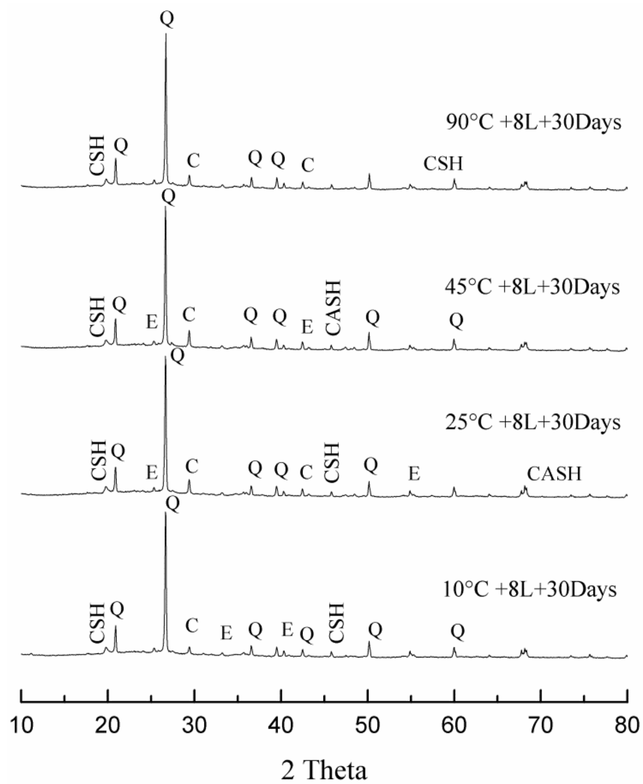

Figure 13. X-ray diffraction pattern of lime stabilized fly ash specimens cured at different temperature (Q – Quartz, E – Ettringite, C – Calcium carbonate, CASH – Calcium aluminium silicate hydrate, CSH – Calcium silicate hydrate

4.4.2 *Scanning electron microscope study*

The surface morphology of the fly ash specimens treated with 8% lime and cured for 30 days at 10^0C, 25^0C, 45^0C and 90^0C temperatures are analyzed and are presented in (Fig. 14). It is observed that an interconnecting network of the pozzolanic reaction products has been formed in the specimens containing 8% lime that are cured at 90^0C. The needle-like crystals of ettringite exist in the hydration product at lower temperature of 10^0C which get gradually disappeared with increase in temperature from 25^0C to 45^0C and then 90^0C. These compounds are already found in texture analysis. Intensification in the curing temperature enhances the pozzolanic reaction and makes the environment favorable for formation of hydration products. It gives an idea that the amount of gel produced was proportional to the amount of lime reacted.

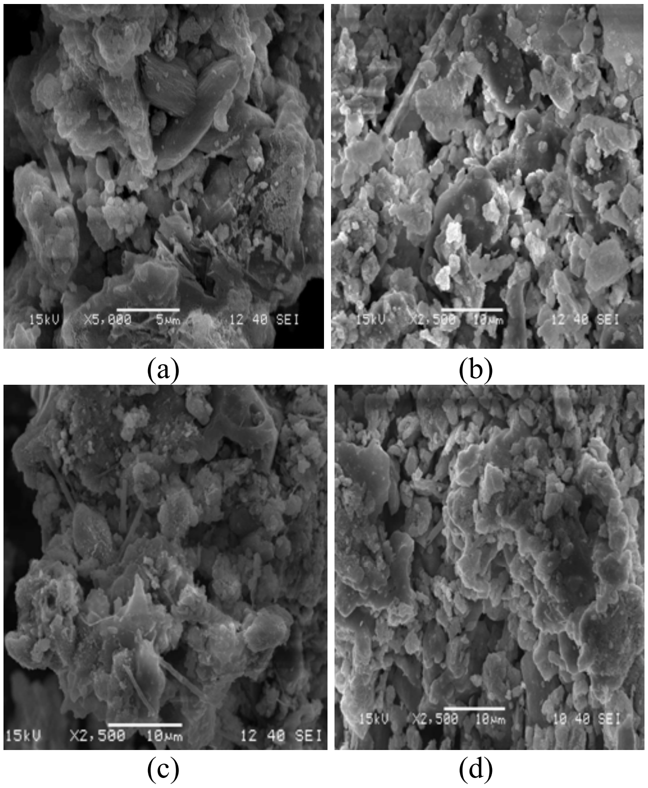

Figure 14. Surface morphology fly ash specimens stabilized with 8% lime for 30 days at curing temperatures of (a) 10°C (b) 25°C (c) 45°C (d) 90°C

4.4.3 *Energy-dispersive x-ray microanalysis*

An EDX spectrum of the specimens containing 8% lime and cured at temperature of 10^0C, 25^0C, 45^0C and 90^0C for 30 days are presented in (Fig. 15). Table 3. Represents the percentage weight ratios of Ca:Si and Al:Si. It is revealed from the EDAX study that the effect of temperature influenced the composition of the hydration products at the grain boundary. It is clearly visible that as the curing temperature increases from 10^0C to 90^0C the Ca:Si ratio decreases which may be attributed to CSH or CASH formation because the high temperature favors faster pozzolanic reaction. From this, it is concluded that more amount of calcium reacted with un-reacted silicon in the initial stage as the curing temperature increase the utilization of calcium is more. These parameters indicate the formation of C-S-H phase with an increase in curing temperature.

Table 3. EDX analysis of lime stabilized fly ash at different temperature

Mix	Ca:Si	Al:Si
10°C+8%L+30D	1.33	0.888
25°C+8%L+30D	0.382	0.648
45°C+8%L+30D	0.199	0.552
90°C+8%L+30D	0.095	0.192

* C = temperature, L = percentage lime, D = curing period

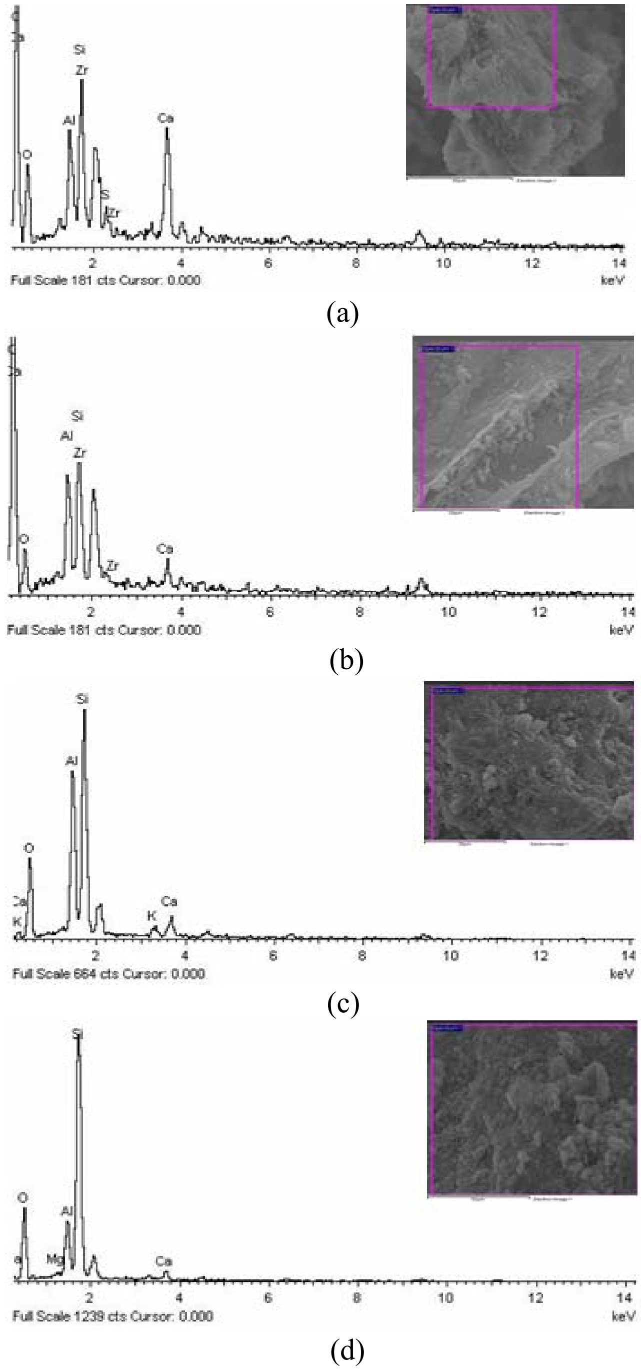

Figure 15. EDAX spectrum of fly ash specimens stabilized with 8% lime for 30 days at curing temperatures of (a) 10°C (b) 25°C (c) 45°C (d) 90°C

5 CONCLUSIONS

Based on the results of the present experimental work, the following conclusions are made:

An increase in lime content marginally reduces the dry unit weight and the dry unit weight of lime stabilized specimen is comparably lower than similarly graded natural inorganic soils. This is advantageous in constructing lightweight embankments over soft, compressible soils. An increase in compactive effort increases the dry unit weight.

An increase of either lime content or curing period shows an improvement in the UCS value. The unconfined compressive strength is very much susceptible to the curing temperature. A higher temperature favors better pozzolanic reaction than a lower temperature especially when the lime content is high. This results in early strength gain. At higher lime content and longer curing period the unsealed specimen shows greater strength than the sealed one; this indicates that the molding water content is not sufficient to complete the pozzolanic reaction especially when the lime content is more. Hence it is recommended that fly ash stabilized with higher amount of lime should either be compacted wet of OMC or sufficient water be added subsequently for proper curing.

The CBR value of compacted virgin fly ash is very much susceptible to the degree of saturation. Substantial reduction in CBR value is observed with increase in degree of saturation. A slight increase in CBR value of virgin fly ash with curing period indicates the presence of free lime in the fly ash which undergoes pozzolanic reaction in the presence of moisture. Both the unsoaked and soaked CBR values are found to increase with increase in lime content as well as curing period. This indicates that the reaction of lime with fly ash is slow at room temperature (27^0C) and a higher curing period is needed to accelerate the pozzolanic reactions. A maximum unsoaked CBR value of 300% is obtained after 30 days curing (12% lime) at 2674 kJ/m^3 compaction energy whereas the soaked CBR value for the above mentioned conditions is 265%.

The diffractographs of lime stabilized fly ash specimens give new amorphous humps of calcium aluminium silicate hydrate, calcium silicate hydrate and ettringite with lower intensity. Additionally, from the surface morphology study it is clearly visible that abundance of needle-like structures of ettringite is found which is mostly responsible for higher strength.

After augmenting the physical and mechanical parameters by treating with suitable doses of lime, this industrial waste material can be used as a road base/sub-base layer, embankment fill, etc as a substitute to the conventional natural materials. This will help in conserving the natural resources side by side disposing off successfully the industrial waste products.

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