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Understanding the Impact of Lime Stabilization on Expansive Soil for Grounding and Analysis Adopting LIBS

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ABSTRACT Stabilization of soil for grounding purposes has been performed by adding different wt% of lime. Electrical breakdown measurements under lightning impulse voltage showed a reduction in breakdown voltage and the time to breakdown with increment in the lime content in the soil. These parameters also increased with curing period for all concentrations of lime of stabilized soil samples. The results show that pH and electrical conductivity of the expansive soil increase as the lime content increases, but decrease with increasing the curing period. Mineralogical and micro-structural analysis has been made by means of X-ray diffraction (XRD) studies and by scanning electron microscope (SEM) analysis. The axial strength obtained from unconfined compressive strength (UCS) analysis, the plasma temperature and the electron number density calculated from Laser Induced Breakdown Spectroscopy (LIBS) data tend to increase with increment in lime content as well as the curing period of the lime stabilized soil. Using univariate analysis, the normalized intensity ratio of Ca II peaks from LIBS spectral data are correlated with lime content in the soil samples at various curing periods. The partial least squares regression (PLSR) technique is successful in determining the soil parameters with the help of LIBS spectral data, at different curing periods with an R^2 value greater than 0.95 and the percentage RMSE value smaller than 4%.

INDEX TERMS Electrical breakdown, grounding, lightning impulse, LIBS, PLSR, soil measurements.

I. INTRODUCTION

Proper grounding system design is essential in order to ensure reliable and safe operation of power system apparatus. The system earth impedance should be as low as possible to successfully protect the power system apparatus from the negative impacts of high currents due to faults and lightning [1], [2]. The behaviour of grounding electrodes under lightning impulse voltages/currents is an important factor to be considered while designing lightning protection systems for power systems as well as buildings. The grounding electrodes are generally buried under soil, which is a heterogeneous medium consisting of solid soil particles, ionic liquid as well as air gaps. In such mixture,

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electrical conduction is non-linear process which can be affected by density, frequency and amplitude of the ground current [2], [3]–[5]. Several researchers have found that when significant impulse currents are applied to the soil, the resistivity of the soil is greatly lowered [3], [6]. Soil ionization occurs when the grounding electrode is subjected to a sudden lightning impulse voltage and the resulting generated electric field exceeds a critical electric field value (E_c) [7]. The soil resistivity in the area around the grounding electrode is reduced as a result. Further research is needed to clarify the behaviour of soil under lightning from the point of view of soil composition and soil enhancement compounds for grounding purposes.

In most parts of south India, expansive soils can be found. These soils are prone to swelling and shrinkage as a result of temperature and moisture fluctuations. These characteristics

will affect the performance of grounding systems buried in such soils [8]. As a result, the expansive soils need to be stabilized for construction and grounding purposes, in order to improve certain properties such as workability, compressive strength, and durability, as well as reduce plasticity [9]. According to present research, expansive soils are being stabilized using structural, mechanical, and chemical-based stabilization methods, including calcium as well as non-calcium-based stabilizers [10]–[12]. Lime stabilization has been a feasible stabilization method for several decades, and it is the most widely used due to its cost-effectiveness. Short-term lime modification as well as long-term pozzolanic reactions are both caused by lime stabilization. Cation exchange and flocculation are examples of short-term lime modification phenomena, while soil-lime pozzolanic reactions are examples of long-term reactions. These processes arrest the expansive soils from changing volume and enhance engineering qualities like expansive soil shear strength. [9], [13], [14]. Therefore, hydrated lime ($\text{Ca}(\text{OH})_2$) has been selected in the study to explore its impact on the soil used for grounding purposes.

The physicochemical parameters such as clay mineralogy, clay composition and clay & pore fluid interactions have a substantial impact on the engineering behaviour of clay soils [15]. During lightning strikes, the ground current may be strong enough to induce electrical discharges that cause soil ionization and disintegration in the surrounding area. This results in the reduction of apparent impedance of the grounding system. The impulse characteristics of grounding systems and soil ionization have been the subject of extensive investigation. [3]–[5], [7], [16]–[18], and there are design guidelines in international standards [19].

Laser induced breakdown spectroscopy (LIBS) is an important tool for elemental characterization of various materials [20]–[24]. Because of the advantages it offers, such as quick measurement and remote measurement capacity, the LIBS approach has wide applications in a variety of fields [25]. Gondal *et al.* detected various ionic species from electrical wires using this approach [26]. It can also be potentially useful for determining the total concentrations of elements in soils. For material categorization, LIBS combined with multivariate analysis such as partial least squares regression (PLSR) and principal component analysis (PCA) are becoming popular tools [27]–[30]. Since, univariate calibration suffers due to matrix effects, the multivariate analysis of whole LIBS spectra effectively reduces the matrix effect [31]. Partial least squares regression (PLSR) is an effective and stable multivariate analysis for calibration of LIBS in soil sample analysis. Guo *et al.* have used LIBS analysis to determine 13 distinct elements in 17 different reference soils using PLSR and by support vector regression (SVR) [32]. Prediction of Mg, Fe, K, Na, Mn and Ca in 63 soil specimens using PLSR and SVR was compared using single and double pulse LIBS [33]. Other soil parameters, such as soil pH, were also determined using multivariate techniques [34], [35].

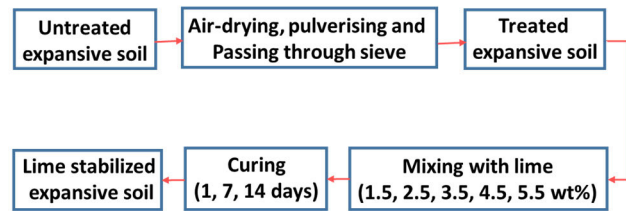


FIGURE 1. Schematic diagram representing the expansive soil treatment and its lime stabilization.

Hence, in the current work, LIBS technique is employed for the elemental analysis of various concentrations of lime stabilized soils. The purpose is to (a) analyze soils using X-ray diffraction (XRD) and scanning electron microscopy (SEM) methods (b) determine the pH and conductivity of lime stabilized soils (c) perform electrical breakdown studies under lightning impulse voltages (c) determine axial strength by UCS analysis, (d) perform elemental analysis by LIBS and (e) determine the soil parameters by employing PLSR technique to LIBS spectral data.

II. MATERIALS AND EXPERIMENTAL SETUP

A. MATERIAL DETAILS

For the current investigation, expansive soil of the required quantity was taken from a depth of 2 m at Solagampatti village, near Tiruchirapalli, Tamil Nadu, India. The gravel fraction was removed from the expansive soil using a typical 4.75 mm sieve. The portion that passed through the 4.75 mm sieve was air-dried, pulverized, and passed through a normal 425 μm sieve. Through particle size analysis it is noticed that the un-stabilized soil has fine particle-content is 76%. The treated soil was then stored in big airtight containers and utilized for the laboratory testing. The soil is classified as an inorganic clay of high compressibility (CH), by the Unified Soil Classification System (USCS). For the expansive soil, the Eades and Grim protocol was employed to determine the initial consumption of lime (ICL). The ICL of expanding was determined to be 3.5%. In this experiment, laboratory grade lime ($\text{Ca}(\text{OH})_2$) with a purity of 90% was employed. Apart from the ICL value, the expansive soil was mixed with lime contents of 1.5% (ICL – 2%), 2.5% (ICL – 1%), 3.5% (ICL), 4.5% (ICL + 1%) and 5.5% (ICL + 2%) separately to account for any possible changes in the lime content in the field. The Schematic diagram representing the expansive soil treatment and its lime stabilization are depicted in Fig. 1.

B. ELECTRICAL BREAKDOWN STUDIES UNDER LIGHTNING IMPULSE VOLTAGE

In the present study, the experimental set-up for performing electrical breakdown studies under lightning impulse voltages, consists of a cubical test cell and standard lightning impulse voltage generator (Fig. 2). The cubical test cell is of dimension $8 \times 8 \times 8 \text{ cm}^3$ with the top open. Inside the cubical test cell, thin copper sheets are pasted on the walls and the bottom of the test cell. The copper sheet at

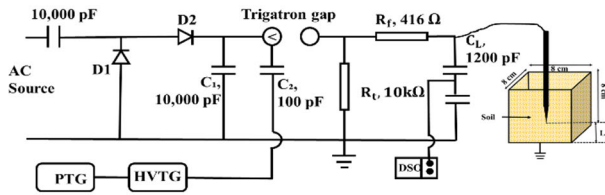


FIGURE 2. Experimental setup for electrical breakdown studies under lightning impulse voltage.

the bottom is grounded electrically and then the required soil sample is filled. A needle-plane electrode configuration is used to study the breakdown voltage of the soil and the soil ionization process. The stainless-steel needle electrode of diameter 6mm having a tip radius of curvature of 0.08 mm is inserted into the soil vertically and a constant electrode gap between the needle tip and bottom ground electrode is maintained.

Standard impulse voltage of $1.2/50 \mu s$ is applied between the two electrodes and measured using capacitance divider and digital signal oscilloscope. Initially, the soil was compressed by a static compressive force to maintain uniformity. Twenty five impulses were applied and the corresponding voltages and the time of breakdown were measured by maintaining a gap of 4 cm using the up and down method.

C. pH AND ELECTRICAL CONDUCTIVITY MEASUREMENT

The samples used to evaluate pH and electrical conductivity were dried in an oven at 50°C for 24 hours. The pH and electrical conductivity calculation of the expansive soil has been performed according to the standards IS 2720 (Part 26) and IS 14767 respectively. 30 g of soil and 75 ml of distilled water were combined in a 150 ml plastic container (soil to water ratio of 1:2.5). The mixture was briskly stirred every 10 minutes for 1 hour to ensure that the mix was homogenous at the time of testing. The average values of five measurements were reported in this work. All of the experiments were carried out at a temperature of $28 \pm 2^\circ C$.

D. XRD AND SEM ANALYSIS

The soil samples were kept on a clean glass slide and glycolated before being mounted on a Bruker AXS-D8-Discover X-Ray diffractometer for XRD investigation. A Cu K (= 1.5148) X-ray tube with a 30 kV input voltage and 30 mA current and a continuous scan speed of 1° per minute was used to produce XRD patterns. The samples were scanned from 15° to 85°. With the aid of X’pert highscore software, the results were recorded and evaluated.

E. UNCONFINED COMPRESSIVE STRENGTH (UCS)

To perform unconfined compressive strength (UCS) analysis, the soil specimens were compacted using a cylindrical mould of 76 mm height and 38 mm diameter. The compacted samples were wrapped in cling films and placed in desiccator

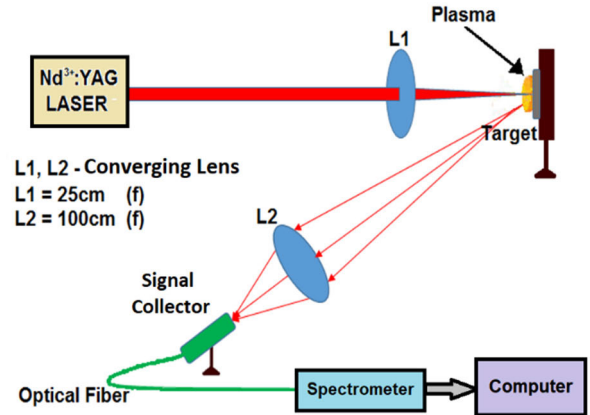


FIGURE 3. Experimental setup of LIBS analysis.

for curing. After the required time of curing (1, 7 and 14 days), the sample was tested for its compressive strength at a strain rate of 0.5 mm/min until failure. The experiment was conducted as a strain-controlled test. The displacement and load values are noted down for every 0.1 mm of displacement change and the stress is calculated from the measured load and area. The strain rate is chosen based on IS 2720-10 which states that the strain rate must be 8-12% of the maximum particle size of the soil.

F. LIBS ANALYSIS

The LIBS experimental setup consists of a Q-switched Nd³⁺:YAG laser (LAB-150-10-S2K, Quanta-Ray LAB series, Spectra Physics) which generates a 1064 nm laser beam with a repetition rate of 10 Hz and pulse duration of 10 ns (Fig. 3). It was focused on the target sample using a 25 cm focal length lens. The optical emission from the target is focused using a lens with 100 cm focal length and collected using a spectrometer (Ocean Optics USB2000+UV-VIS-ES), through an optical fibre with a numerical aperture of 0.22 NA and a core diameter of 400 μm. The spectral data was evaluated in the region of 200-900 nm in the current investigation. In the present study, the laser pulse energy was set at 40 mJ. The laser was focused with the help of a 25 cm focal length lens, to the target sample with a spot diameter of 0.5 mm. For this laser energy of 40 mJ and 0.5 mm spot diameter, laser fluence 203.72 kJ/m² is obtained.

III. RESULTS AND DISCUSSION

A. ELECTRICAL BREAKDOWN STUDIES

The variation in breakdown voltage and time to breakdown are represented in (t_{bd}) of the soil samples with respect to the lime content is represented in Fig. 4. The breakdown voltage and the time to breakdown are seen to reduce with increment in the lime content in the soil, which is an indication of the rise in the conductivity of the soil. To understand the impact of lime stabilization on conductivity, the electrical conductivity measurements have been performed, which were discussed

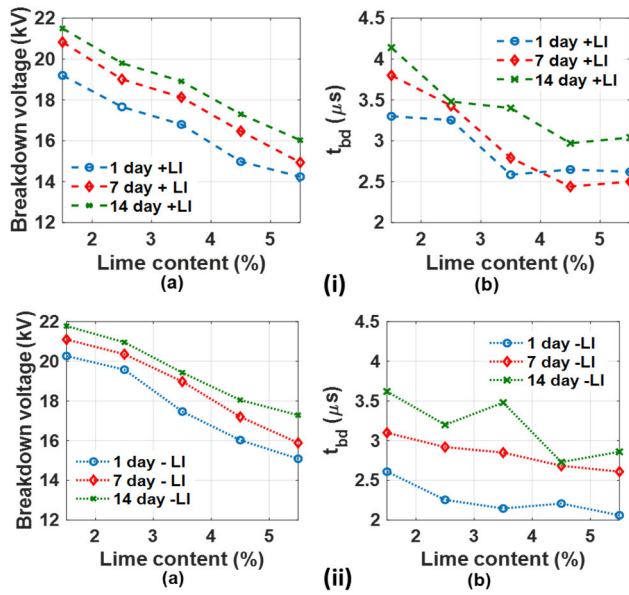


FIGURE 4. (a) Breakdown voltage and (b) time to breakdown of 1 day, 7 days and 14 days cured expansive soil under (i) positive lightning impulse and (ii) negative lightning impulse voltage.

in section III B. The breakdown voltage for negative-polarity LI voltage is higher than that for positive LI voltage for all lime contents and curing durations. With both polarities, the longer the curing period, the higher the breakdown voltage becomes, probably due to the reduction in the electrical conductivity of the soil with curing. The results also show that the time to breakdown becomes longer for increasing curing durations. For positive LI, the time to breakdown appears to stabilize after the lime content exceeds 4.5%. For the negative LI, the time to breakdown is slightly shorter than of the positive LI. Similar results have been observed by Nor *et al.* with higher breakdown voltage and marginally lower time to breakdown under negative LI compared to positive LI [16]. They have stated that the higher breakdown voltage is because of the higher threshold electric field (E_c) of negative LI when compared to positive LI and the time to breakdown is not significantly affected by the polarity of impulse voltage [16].

B. pH AND ELECTRICAL CONDUCTIVITY

The variation of pH and conductivity of the expansive soil with respect to the lime content at various curing periods is depicted in Fig. 5a and Fig 5b respectively. The pH and electrical conductivity of the soil increase with increment in the lime content. Since the lime is basic, the pH of the lime stabilized soil increases sharply at an approximately constant rate when the lime content is varied from 1.5 % to 3.5% for all curing periods. Above 3.5%, the pH continues a gradual increase but at a lower rate. Similarly, the conductivity increases largely with lime content for all curing periods. This confirms that the reduction in the electrical breakdown voltage with respect to lime content, is because of the

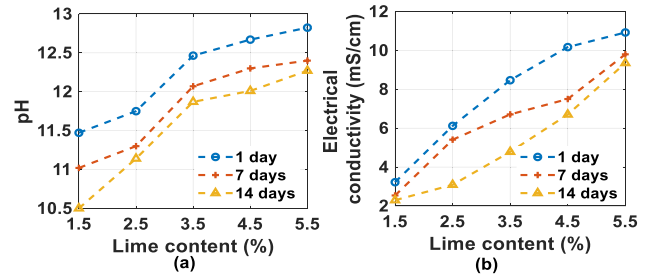
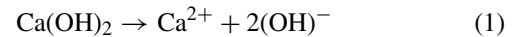


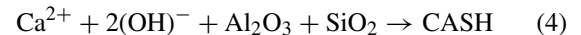
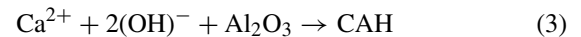
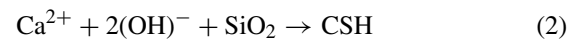
FIGURE 5. (a) pH and (b) electrical conductivity of 1 day, 7 days and 14 days cured expansive soil as a function of lime content.

increase in conductivity of the soil after stabilization. Both pH and conductivity of the soil samples tend to reduce with the duration of the curing period, as was confirmed by Rao *et al* [36].

The consumption of hydroxyl ions during the pozzolanic reactions, is responsible for the decrease in pH and electrical conductivity, as described in equations (2)-(4) [37]. The lime addition to the soil makes it highly alkaline, which results in dissolution of silica (Si^{4+}) and alumina (Al^{3+}) ions. Pozzolanic reactions tend to occur between free Ca^{2+} ions from lime and dissolved Si^{4+} and Al^{3+} ions from soil, which lead to the formation of calcium-silicates and aluminates, which upon curing transforms into hydrates and form cementitious compounds such as calcium silicate hydrate, calcium aluminum hydrate and calcium aluminum silicate hydrate (CSH, CAH and CASH) in the presence of adequate moisture. The dissociation of hydrated lime into its constituent ions is as follows:



Calcium silicate hydrate and calcium aluminum hydrate formation:



The type and availability of reactive clay minerals in the soil determine the occurrence of pozzolanic reactions, which result in a gradual rise in the strength, stiffness, and durability of stabilized soil. This is referred to as long-term stabilization. To confirm the occurrence of pozzolanic reactions as well as the formation of cementitious compounds, XRD and SEM analysis have been performed and were discussed in section III C.

C. XRD AND SEM ANALYSIS

X-ray diffraction spectra of the expansive soil samples before and after lime stabilization are represented in Fig. 6. X-ray diffraction spectra mostly showed the montmorillonite peaks (ICDD-PDF-002-0239) and quartz peaks (ICDD-PDF-001-0649). The montmorillonite peaks are observed at 20.20° , 39.87° and 76.10° . The quartz peaks are

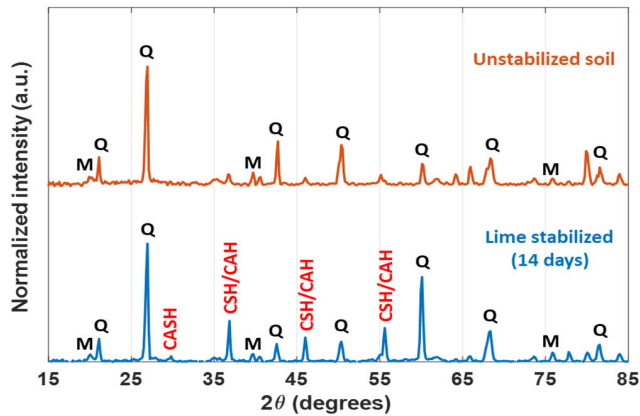


FIGURE 6. XRD patterns of soil specimens before and after lime stabilization; M: Montmorillonite, Q: Quartz, CSH: Calcium silicate hydrate and CASH: Calcium aluminum silicate hydrate.

observed at 20.91°, 26.63°, 50.07°, 60.02° and 67.89°. The XRD spectra after lime stabilization reflected a significant increment in the peaks at 36.65°, 46.03° and 55.66°, which represent calcium silicate hydrate (ICDD-PDF-003-0247) as well as calcium aluminum hydrate (ICDD-PDF-011-0203). Also, the appearance of new peak at 30.12°, which represent the formation of calcium aluminum silicate hydrate (ICDD-PDF-011-0589).

The SEM micrographs of non-stabilized expansive soil and lime stabilized soil samples (with 3.5 wt% of lime and cured for 1 day, 7 days and 14 days) at 20% moisture content are represented in Fig. 7. From Fig. 7, it is noticed that the formation of aggregates is not significant after 1 day of curing. The aggregates are identified clearly in case of 7 days and 14 days cured samples. Upon addition of lime, the hydrous diffuse double layer that surrounds clay particles is modified [38], [39]. This causes flocculation and agglomeration of clay particles by altering the electrical charge density surrounding the soil particles. The aggregation of the clay soil leads to the conversion of a portion of clay particles into the silt-sized particles, causing a change of the plasticity characteristics [12]. This indicates that the expansive nature of the soil is reduced after lime stabilization. It can be concluded that the soil classification was changed from CH (high compressible clay) to ML (low compressible silt) after lime stabilization of the expansive soil.

D. UCS ANALYSIS

Fig. 8 represents the compressive strength values of lime stabilized soil specimens with respect to lime content at various curing periods. As can be seen in the Fig. 8 the compressive strength increases with increment in lime content. This indicates the increase in the strength of the soil, after lime stabilization. The compressive strength also increases with curing time, reflecting that the soil becomes hardened after curing. The expansive soil used in the present study contains a high percentage of clay. Clay is identified as a good pozzolan as it possesses silica and alumina,

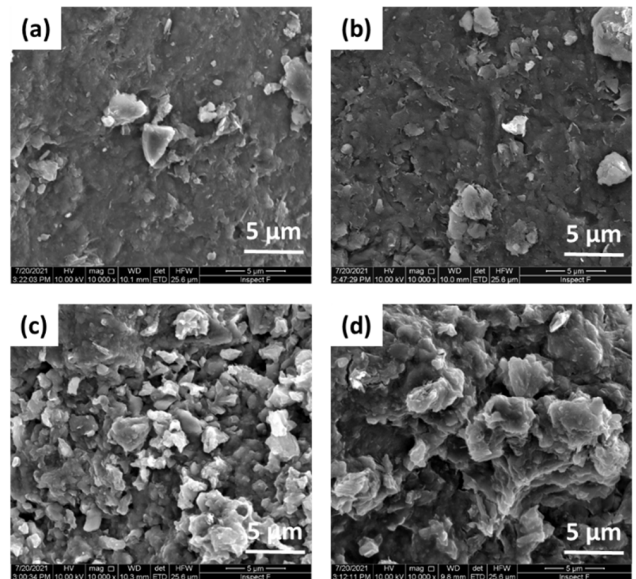


FIGURE 7. SEM micrographs of (a) non-stabilized expansive soil (b) 3.5 wt% lime stabilized soil cured for 1 day, (c) 7 days and (d) 14 days.

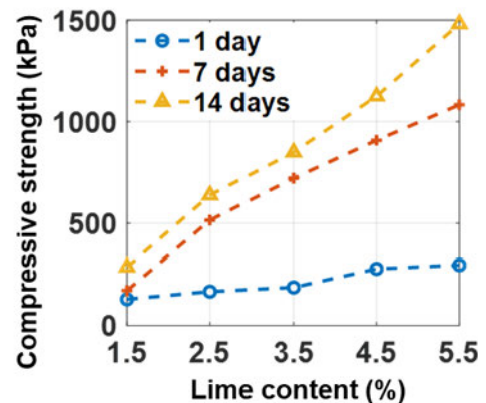


FIGURE 8. Comparison of compressive strength as a function of lime content for 1, 7 and 14 days of curing.

which on treatment with lime can give rise to cementitious compounds that initially form as a gel. This gel has the ability to combine the lumps of clay and upon crystallization forms cementitious compounds. The formed cementitious compounds bind the aggregates together, and form an interlocking structure upon crystallization, thereby increasing the strength of the soil [40]. From the SEM micrographs shown in Fig. 7, the formation of the aggregates can be identified clearly after 14 days of lime stabilization. This is due to crystallization of lumps of clay forming cementitious compounds. Thus, the increment in the compressive strength of the lime stabilized soil with respect to curing period is due to the hardening of the soil by the formation of cementitious compounds.

E. LIBS ANALYSIS

The LIBS intensity spectra of the soil specimens have been shown in Fig. 9. With the use of the NIST database [41],

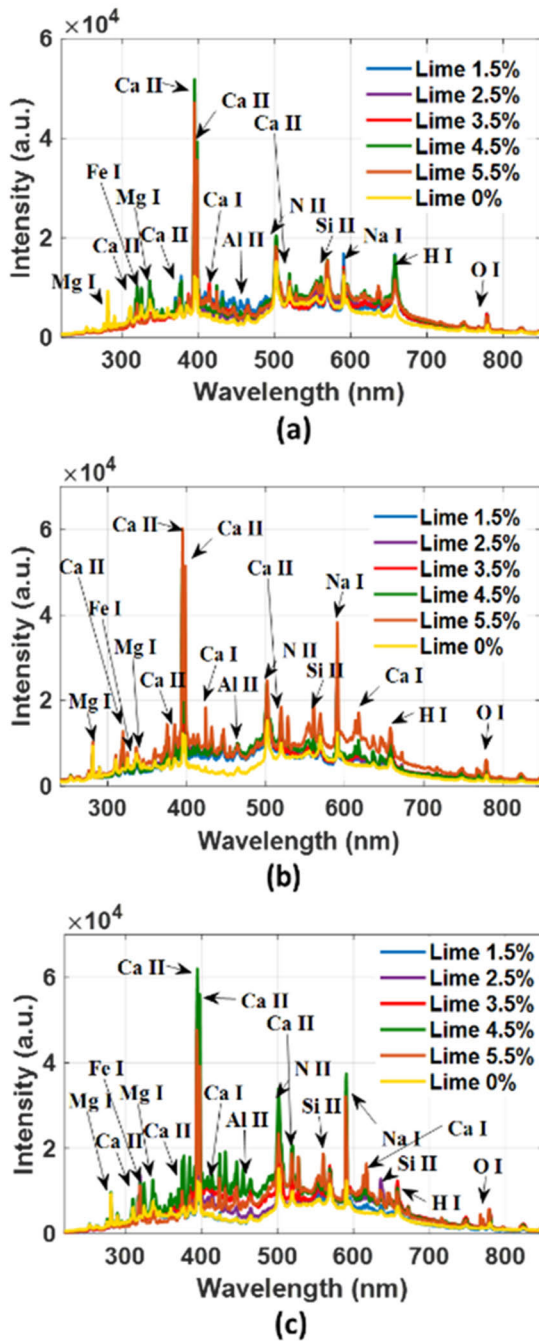


FIGURE 9. LIBS emission spectra of (a) 1 day cured (b) 7 days cured and (c) 14 days cured expansive soil.

peaks corresponding to aluminium (Al), calcium (Ca), silicon (Si), iron (Fe), Sodium (Na), hydrogen (H), and oxygen (O) are recognized in the spectral data. With increasing the curing duration, no new peaks in the LIBS spectra of the test specimens were identified. The Boltzmann-Saha equations may be used to calculate the plasma temperature (T_e) and electron number density (n_e) of the test specimens [29].

$$T_e = 1.44 \frac{E_2 - E_1}{\ln \left(\frac{I_1 \lambda_1 A_2 g_2}{I_2 \lambda_2 A_1 g_1} \right)} \quad (5)$$

TABLE 1. Details of spectral lines used for calculating plasma temperature.

Element	Wavelength, λ (nm)	Transition probability, A (s^{-1})	Statistical weights, g	Energy level, E (cm^{-1})
Ca II	317.9	3.60e+08	5/2	56858.46
Ca II	393.3	1.35e+08	3/2	25414.40

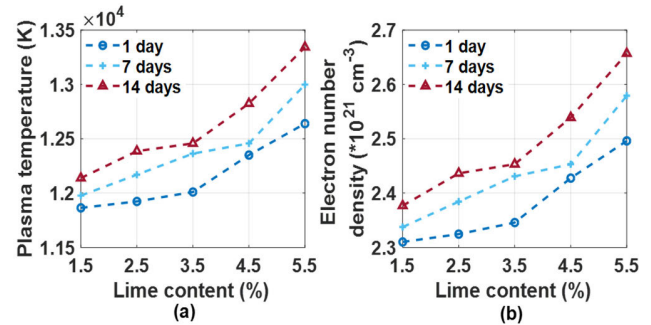


FIGURE 10. (a) Plasma temperature and (b) electron number density of 1 day, 7 days and 14 days cured expansive soil as a function of lime content.

$$n_e = 6.6 \times 10^{21} \frac{I_1 A_2 g_2}{I_2 A_1 g_1} \exp \left(- \frac{E^{ion} + E_2 - E_1}{T_e} \right) \quad (6)$$

where E_1 and E_2 are energies corresponding to excited energy levels 1 and 2, I_1 and I_2 represent intensities of particular atomic species at λ_1 and λ_2 wavelength respectively, A_1 and A_2 represent transition probabilities of states, g_1 and g_2 indicate statistical weights, E^{ion} is the ionization energy, T_e is the plasma electron temperature and n_e is the plasma electron density under the local thermodynamic equilibrium.

The persistent line of Calcium (Ca II) at a wavelength of 317.9 nm and the persistent line of Calcium (Ca II) at a wavelength of 393.3 nm are used to calculate T_e and n_e values. Table 1 lists all of the information corresponding to Calcium (Ca II) spectral lines. From Fig. 10, T_e and n_e values of the soil samples are represented as a function of lime content at different curing periods. Irrespective of curing period, the plasma temperature and electron number density of the soil samples tend to increase with increasing lime content. Also, the T_e and n_e values at each lime content increase with curing period. The plasma temperature is an indirect measurement for estimating the hardness of the material [42]. The harder material tends to resist the external damage more when compared to less harder material. Similarly, the energy absorption efficiency of the harder sample ablated by laser pulse is lower, resulting in less plasma formation during ablation. As a result, when the same laser pulse energy is used on a less ablative (harder) material, the energy is concentrated and the plasma temperature rises [42]. Aberkane *et al.* have correlated plasma temperature calculated through LIBS with the hardness of the material [43]. The rise in the plasma temperature with increasing lime content as well as curing period is an indication that the soil has become harder. Also, from UCS analysis it is noticed that the axial strength of the

TABLE 2. R² and RMSE values of soil samples while estimating different parameters by PLSR method.

Estimated parameter	Calibration			Testing		
	R ²	RMSE	%RMSE	R ²	RMSE	%RMSE
1 day cured soil						
Lime wt%	1.000	0.0014	0.0254 %	0.959	0.2898	5.27 %
Mass fraction of Ca	1.000	0.0007	0.0167 %	0.959	0.1555	3.71 %
pH	1.000	0.0002	0.0016 %	0.960	0.0581	0.45 %
Electrical Conductivity	1.000	0.0027 mS.cm ⁻¹	0.0246 %	0.976	0.4379 mS.cm ⁻¹	3.99 %
Electron number density	1.000	6.34×10 ¹⁶ cm ⁻³	0.0025 %	0.963	1.55×10 ¹⁶ cm ⁻³	0.62 %
Plasma temperature	1.000	0.2638 K	0.0021 %	0.962	65.07 K	0.52 %
7 days cured soil						
Lime wt%	1.000	0.0005	0.0091 %	0.991	0.1326	2.41 %
Mass fraction of Ca	1.000	0.0003	0.0072 %	0.991	0.0716	1.71 %
pH	1.000	0.0002	0.0016%	0.979	0.0525	0.42%
Electrical Conductivity	1.000	0.0005 mS.cm ⁻¹	0.0094 %	0.987	0.1289 mS.cm ⁻¹	2.42 %
Electron number density	1.000	2.19×10 ¹⁶ cm ⁻³	0.0008 %	0.996	0.58×10 ¹⁶ cm ⁻³	0.22 %
Plasma temperature	1.000	0.0913 K	0.0007 %	0.996	24.48 K	0.19 %
14 days cured soil						
Lime wt%	1.000	0.0016	0.0290 %	0.998	0.068	1.24 %
Mass fraction of Ca	1.000	0.0008	0.0200 %	0.998	0.0367	0.88 %
pH	1.000	0.0007	0.0056 %	0.997	0.0391	0.32 %
Electrical Conductivity	1.000	0.0030 mS.cm ⁻¹	0.0320 %	0.997	0.1351 mS.cm ⁻¹	1.44 %
Electron number density	1.000	2.2949×10 ¹⁶ cm ⁻³	0.0086 %	0.994	1.3251×10 ¹⁶ cm ⁻³	0.49 %
Plasma temperature	1.000	0.5532 K	0.0040 %	0.998	17.69 K	0.13 %

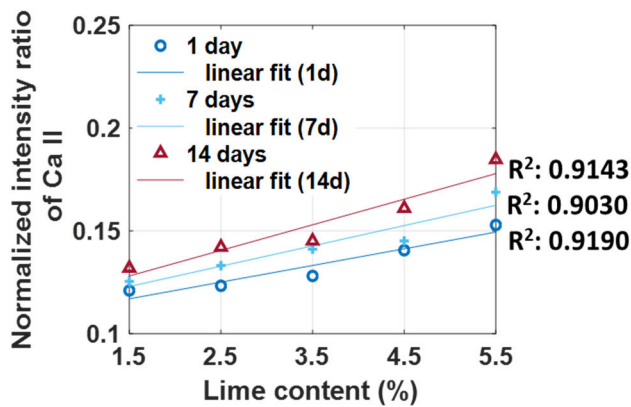


FIGURE 11. Normalized intensity ratio of 1 day, 7 days and 14 days cured expansive soil as a function of lime content.

soil samples tend to increase with respect to lime content as well as curing period. It can be concluded that the plasma temperature values and the axial strength values obtained from the UCS analysis follow similar trends with respect to lime content at different curing periods.

Fig. 11 shows the variation of the normalized intensity ratio of lime-stabilized soil specimens with lime content at different curing periods. The normalized intensity ratio of the LIBS spectral lines, Ca II at a wavelength of 317.9 nm and Ca II at a wavelength of 393.3 nm, were correlated with the lime content, and a direct correlation is obtained for all curing

periods. The univariate analysis gave a correlation coefficient (R²) greater than 0.9 for all curing periods. Also, it is noticed that the normalized intensity ratio increases with curing time.

F. DETERMINATION OF SOIL PARAMETERS FROM LIBS ASSISTED PLSR ANALYSIS

The LIBS spectral data information that cannot be recovered using conventional data processing methods. The multivariate method, partial least squares regression (PLSR) has been applied to the LIBS spectral data to predict various soil parameters such as lime content, mass fraction of calcium content in the soil, pH and conductivity. PLSR is a dimensional reduction approach that first creates a new reduced set of features that are linear combinations of the original features, and then uses these new features to fit a linear model using least squares [31]. The LIBS spectrum with wavelengths 200-900 nm was used for PLSR analysis of all soil samples in this investigation. This technique has been implemented in MATLAB for obtaining calibration models as shown in Fig 12. The LIBS spectra were taken as input and are randomly split into training and test dataset in the percentages 80% and 20% respectively. At each lime content, 50 LIBS spectra were selected for the analysis. Accordingly, the input dataset with dimensions 250 × 2048 is divided into 200 × 2048 for training and 50 × 2048 for testing. The measured/actual output dataset has the dimensions of 200×1 for training and 50×1 for testing. The predicted output values are correlated with the actual output values to obtain

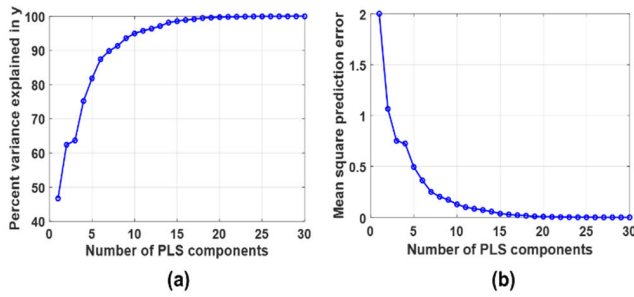


FIGURE 12. (a) Percentage explained and (b) mean square error during training of soil samples by PLSR method.

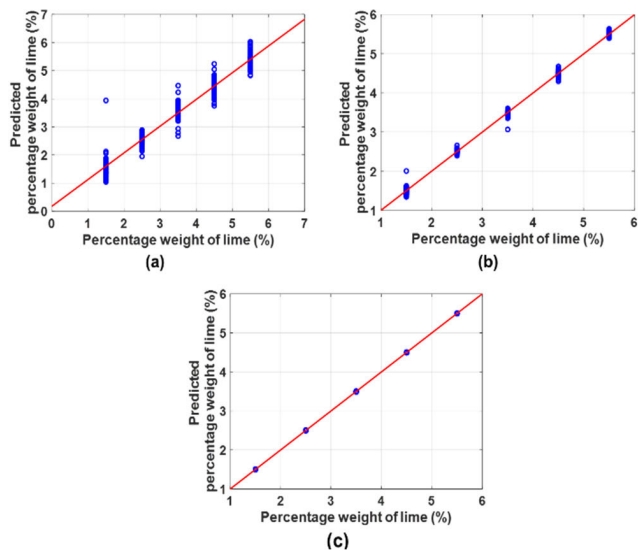


FIGURE 13. Regression plots during training of soil samples by PLSR method with (a) 10, (b) 20 and (c) 30 PLS components.

the correlation coefficient (R^2) and these values are compared to obtain the root mean square error (RMSE) during training as well as testing.

Fig. 12 displays the percentage variance explained and mean square error during calibration of lime stabilized soil data after 1 day of curing period. The percentage variance is almost covered i.e. near to 100% at 30 PLS components. Also, the mean square error of the calibration is lower than 0.01 at 30 PLS components. The regression plots during calibration of lime stabilized soil data after 1 day curing period with 10, 20 and 30 PLS components are depicted in Fig 13a, b and c respectively.

It is noticed that the predicted lime weight percentages are exactly equal to actual lime contents when 30 PLS components are used. Hence, the number of PLS components is selected as 30 for 1-day cured soil samples. Similarly, the number of PLS components that provide proper calibration results are 20 for both 7 days and 14 days cured soil samples.

With LIBS spectral data as input and the percentage lime weight percentage added to expansive soil as output, a regression model has been calibrated and tested with the

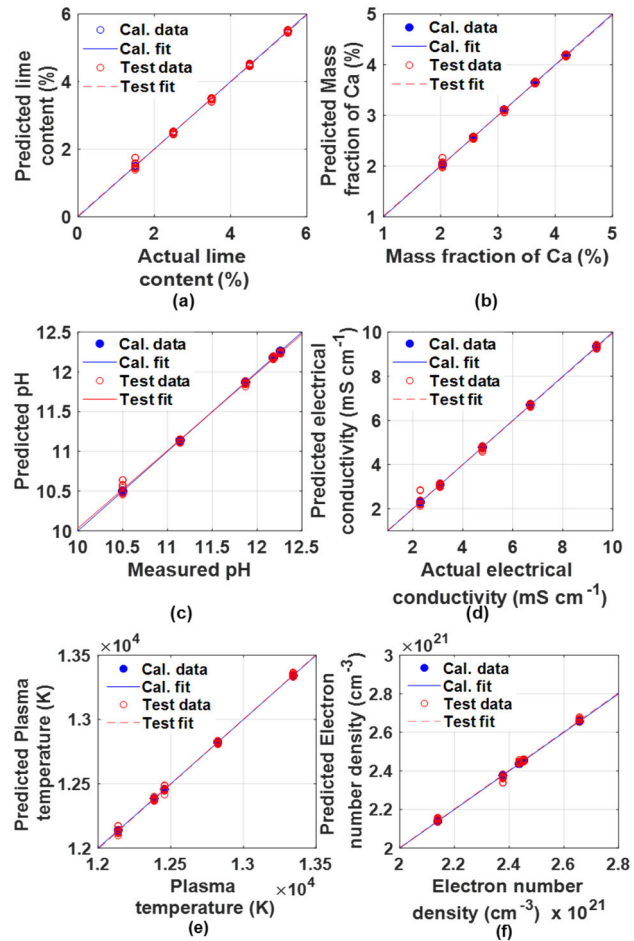


FIGURE 14. Regression plots during calibration and testing of soil samples by PLSR method for determination of (a) percentage lime content added, (b) mass fraction of Ca, (c) pH, (d) electrical conductivity, (e) plasma temperature and (f) electron number density.

help of PLSR technique. For 1 day cured samples the R^2 and the % RMSE values are 1 and 0.025% during calibration and 0.9585 and 3.98% while testing. For 7 days and 14 days cured soil samples also, the %RMSE values during testing are 2.41% and 1.24% respectively. The regression plots of calibration and testing data of 14 days cured soil samples are shown in Fig 14a. A proper correlation can be seen between actual values and the predicted values of lime content with R^2 values of 1 and 0.998 for calibration and testing respectively. For all curing periods, the percentage lime added to the soil has been successfully determined for LIBS spectral data with the of PLSR technique. Similarly, the PLSR technique has been adopted to determine the values of mass fraction of Ca, pH, electrical conductivity, plasma temperature and electron number density at different curing periods of lime stabilized soils. The R^2 , RMSE and the % RMSE values of the calibration and testing data for determination of different soil parameters at different curing periods of lime stabilized soils are represented in Table 2. From EDAX results, it is noticed that the mass fraction of Ca in the non-stabilized soil is 1.22% [12]. With respect to increment in lime content,

the mass fraction of 1.5, 2.5, 3.5, 4.5 and 5.5 wt% of lime stabilized soils are calculated as 2.03, 2.57, 3.11, 3.65 and 4.19% respectively.

The regression plots of calibration and testing data of 14 days cured soil samples, while determining mass fraction of Ca are shown in Fig 14b. R^2 values of 1 and 0.9977 have obtained between actual and predicted values for calibration and testing respectively. The measured values of pH and electrical conductivity are used as output data for calibration and the predicted values during testing show values of %RMSE lower than 0.5% and 4% respectively. Fig 14c and 14d show the regression plots of calibration and testing data of 14 days cured soil samples, while determining pH and electrical conductivity respectively. Similarly, the T_e and n_e values calculated from the LIBS analysis are considered as output data for calibration and the predicted values during testing are found to have the %RMSE values lower than 0.6% and 0.7%. The regression plots of calibration and testing data of 14 days cured soil samples, while determining T_e and n_e are shown in Fig 14e and 13f respectively. A proper correlation between actual values and the predicted values of T_e and n_e is noticed for both parameters with R^2 values 0.998 and 0.994 respectively. Overall, the soil parameters have been successfully determined from the LIBS spectral data using the PLSR technique.

IV. CONCLUSION

This study enables drawing the following important conclusions:

- The breakdown voltage and the time to breakdown tend to reduce with increment in the lime content of the soil, which is an indication of the rise of the soil conductivity that has also been noticed from the electrical conductivity measurements. The breakdown voltage and the time to breakdown increase with curing period for all concentrations of lime stabilized soil specimens.
- Increasing the lime content tends to increase the pH and electrical conductivity of the soil. Also, it is observed that the pH and the electrical conductivity of soil samples tends to reduce with the curing period.
- Stabilization of soil samples using lime has been confirmed from the formation of CSH, CAH and CASH compounds, which are identified through X-ray diffraction analysis. Formation of aggregates/ flocculation has been observed from SEM analysis after lime stabilization.
- The axial strength increases with increment in curing period as well as the lime content of the soil.
- The plasma temperature and the electron number density calculated with the help of LIBS spectral data tends to increase with increment in lime content as well as the curing period of the soil. A direct correlation is obtained between axial strength from UCS analysis and the plasma temperature.

- Using univariate analysis, the normalized intensity ratio of the Ca II peaks from LIBS spectral data were correlated with lime content in the soil samples at different curing periods.
- PLSR technique is successful for determination the soil parameters with the help of LIBS spectral data, at different curing periods with an R^2 value greater than 0.95 and the percentage RMSE value lower than 4%.

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