



Influence of Soluble Sodium Sulphate Contamination on Physical and Strength Behavior of Untreated and Lime Treated Soil

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ABSTRACT

The susceptible behavior of sulphatic soil with lime treatment is explored mostly by using gypsum. However, the effect of varying sulphate cations contamination in the behavior of chemically treated soils with calcium based stabilizers has not been given attention properly. Hence, the present study explores the influence of soluble monovalent sodium sulphate (Na₂SO₄) on the physical and strength behavior of untreated and treated expansive soil with lime. Further, research works are extended to establish the soluble Na₂SO₄ concentration that can affect adversely to the lime treatment in comparison to untreated soil and its mechanism is elucidated using detailed physicochemical and microanalyses. The results reveal that the soluble Na₂SO₄ contamination of both untreated and lime treated soil has shown an increase in the MDD and reduction in OWC with a marginal effect on the plasticity characteristics. The soluble Na₂SO₄ contamination accelerates the early strength in the treated soil as compared to the same with untreated soil. On the contrary, longer curing periods and higher sulphate concentrations beyond 20,000 ppm lead to distress in the strength of untreated and treated soil. Multiple factors that can control the behavior of both untreated and treated soil upon soluble Na₂SO₄ concentration such as cation position in lyotropic series, ionic reactions and its products are evidenced using the microanalyses (SEM, XRD, EDAX, TGA, FTIR) and physicochemical changes (pH and EC).

1. Introduction

Chemical stabilization using additives like lime and cement is considered widely as one of the effective soil improvement methods (Al-Mukhtar et al., 2012). However, the presence of sulphate can deteriorate lime stabilized soil permanently (Puppala et al., 2018). It has been mentioned that 20% of the land in the world is covered with soils containing sulphate which create several issues such as ground subsidence, karst formations, settlement, formations of cavity and cracks etc. (Yamamoto and Kennedy, 1969; Solis and Zhang, 2008). The existence of sulphatic compounds depends upon several aspects including geological formations, industrial induced activities, waste accumulation, sulphatic river streams, acid rains etc. (Sharma and Kumar, 2020). The solubility rates of most commonly available sulphatic salts [CaSO₄, Na₂SO₄, MgSO₄ and K₂SO₄] in the soils are 2.6 gm/L, 408 gm/L, 260 gm/L and 120 gm/L, respectively

(Mitchell, 1986; Hunter, 1988; Kota et al., 1996). Because of varying solubility rates as well as cationic configurations, each sulphatic salt behaves differently with the soils. The interactions of soil with sulphates in the presence or absence of any additive may influence the cationic exchanges and pozzolanic reactions. Past research indicates that the presence of sulphates causes destructive impacts to the soil in terms of abrupt swelling/heaving when treated with calcium-based stabilizers (Hunter, 1988; Mitchell, 1986; Kota et al., 1996; Puppala et al., 2019). Therefore, the influences of chemical constituents need to be understood prior to their applications.

Previous research works have focused mostly on the findings of the deleterious effects of gypsum in the lime treated soil (Mitchell, 1986; Hunter, 1988; Kota et al., 1996; Sivapullaiah et al., 2000; Puppala et al., 2003; Aldaood et al., 2014; Jha and Sivapullaiah, 2016; Gadouri et al., 2017). However, sodium sulphate (Na₂SO₄) having the highest solubility rate (i.e., 408 gm/L) has

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an ability to dissolve easily in the flowing water and, can migrate into the soils, leading to ground contamination (Blaser and Scherer, 1969; Ramesh et al., 1999). Very limited researchers (Sivapullaiah et al., 2006; Gadouri et al., 2017; Abdi et al., 2020; Raja and Thyagaraj, 2019; Ramesh and Manjunatha, 2020) have investigated the impact of Na_2SO_4 on the plasticity, compaction, strength, and swell properties of soils. It is reported that the use of Na_2SO_4 in the range of 0.20-2.00% with additives such as lime, rice husk ash, fly ash, etc. acts as a booster to improve the soil properties (Davidson et al., 1960; Blaser and Scherer, 1969; Marks and Halliburton, 1970; Shi and Day, 2000b). However, its excess dosage results in an adverse effect on the properties of soils (Sherwood, 1962; Celik and Nalbantoglu, 2013; Abdi et al., 2020; Raja and Thyagaraj, 2019). It is stated that the Na_2SO_4 dissolves in its own water of hydration during warm temperature (more than 90 °F) and then moisture evaporates from soil which leaves behind salts on the surface. During cold temperature (below 90 °F) moisture forms crystals that bind itself to the Na_2SO_4 , resulting in the volumetric expansion of soil.

The presence of sulphate in lime treated soil results in the formation of heave known as “Sulphate Induced heave”; while the presence of Na_2SO_4 in the soil causes in the formation of heave also known as “Salt Heave” (Blaser and Scherer, 1969; Fang et al., 2018). These salt heaves generate when the soil encounters the variable temperature range fluctuating between i.e., 35-100°F (Blaser and Scherer, 1969). One molecule of Na_2SO_4 can bind 10 water molecules, causing high expansion. The facts imply that Na_2SO_4 holds a variable approach towards the untreated and treated soil. Also, the amount of Na_2SO_4 , participating ions, types of soil, clay mineral, climatic conditions, availability of water, types of any additive used, curing days etc. govern the geotechnical behaviour of soil (Ogawa and Roy, 1982; Myneni et al., 1998; Damidot and Glasser, 1993; Little et al., 2005). Ramesh et al. (2009) reported that the strength of treated soil with lime improved initially but, decreased at longer curing periods in the presence of Na_2SO_4 due to the formation of ettringite. In the case of gypsum contamination, it has been established that the concentration of sulphate beyond 2,000 ppm causes adverse effects on soils treated with lime due to the formation of expansive minerals (i.e., ettringite or, thaumasite) (Hunter, 1988; Mitchell et al., 1992; Petry and Little, 1992; Berger et al., 2001; Little et al. 2005 and 2006; Harris et al., 2006; Puppala et al., 2018). However, no study has been reported the optimum Na_2SO_4 concentration to affect the untreated or lime treated soil adversely. Ettringite is reported as a calcium alumino-sulphate hydrate mineral that may have a needle-like structure or rounded fibrous that formed under high alkaline conditions (Moore et al., 1970; Petry and Little, 1992; Moon et al., 2007). One molecule of ettringite mineral can hold twenty-six molecules of water within it, leading to the high expansion (Hunter et al., 1988; Raja and Thyagaraj, 2019). The expansion due to high sulphate concentration are responsible for causing damage to subgrade and structures in many areas of the world. On contrary, the ettringite having its needle-like and fibrous structure acts as an interlocking and

reinforcing material within the soil samples, resulting in an enhancement in the strength (Mehta, 1983; Dermatas, 1995; Jha and Sivapullaiah, 2017). Kinuthia et al. (1999) reported that sulphates containing monovalent cations (Na^+ and K^+) have shown an adverse effect on the improvement of lime treated kaolinite soil over sulphates containing divalent cations (Ca^{2+} and Mg^{2+}). Further, previous research have focused mostly on the findings of the behaviour of soil containing sulphate upon lime treatment (Kinuthia et al., 1999; Shi and Day, 2000a; Gadouri et al., 2017). However, there are many possibilities where contamination in soils can occur due to the migration of water containing sulphates (eg., acid mine drainage, discharge of sulphate contaminated water by industries etc.), leading to the possible alteration in the its behaviour. The effects of migration of sulphate containing varying cations in the formation of ettringite and the behaviour of untreated and soil treated with lime are still debatable issues to be addressed.

The present study has aimed to investigate the geotechnical properties, physicochemical, and microanalyses of untreated and lime treated expansive soil subjected to wide ranges of soluble Na_2SO_4 concentrations. Geotechnical properties are assessed by performing physical (Atterber’s limits and compaction characteristics) and unconfined compressive strength (UCS) tests. Further, microanalyses of samples collected after completion of experiments are performed through X-ray diffraction (XRD), Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray (EDAX), Fourier Transform Infrared Spectroscopy (FTIR), and Thermogravimetric Analysis (TGA)-Derivative Thermogravimetry (DTG). The physicochemical examinations are assessed by measuring the alkalinity (pH) and electrical conductivity (EC).

2. Material and Methodology

2.1 Materials Used

2.1.1 Soil

The soil is obtained from the location of Jaipur in the Rajasthan State of India. The soil is taken out from a depth of 1.5 meters below ground level to avoid any kind of impurities. The oven dried soil passed through 425 microns IS sieve is used for experimental purposes [IS: 460-Part 1 (1978)].

The properties of the soil are shown in Table 1. The grain size analysis (IS: 2720-Part 4, 1985) of soil confirms the presence of sand (4.75 – 0.075 mm) (i.e., 11%), silt (0.075 – 0.002 mm) (i.e., 13%) and clay (<0.002 mm) (i.e., 76%). The specific gravity of soil is seemed to be lower (i.e., 2.37). This is attributed to the presence of some organic matter and porous particles. However, based on the plasticity chart of Indian Standard (IS), soil falls below A-line, and is designated as “Silt of Intermediate Plasticity (MI) or Organic soil of Intermediate Plasticity (OI)”. Further, it is mentioned clearly in IS: 1498 – 1970 (IS:1498, 1970) that the Black Cotton Soil (BCS) (i.e. common name of expansive soil in India) is also found to lie below the ‘A’-line even though, the soil is predominated with clay minerals. This again confirms the

Table 1. Geotechnical Properties of Untreated and Lime Treated Soil

Properties	Soil	Lime stabilized soil	Methodologies Followed
Sand (4.75 – 0.075 mm), %	11.00	-	IS: 2720-4 (1985)
Silt (0.075 – 0.002 mm),%	13.00	-	
Clay (< 0.002 mm),%	76.00	-	
Specific Gravity	2.37	-	IS: 2720-3 (1980)
Liquid Limit (%)	49.00	41.00	IS: 2720-5 (1985)
Plastic Limit (%)	29.20	39.60	
Plasticity Index (%)	19.80	1.40	
Shrinkage Limit (%)	11.65	21.96	IS: 2720-6 (1972)
Max Dry Density (MDD) (g/cc)	1.62	1.49	Sridharan and Sivapullaiah (2005)
Optimum Water Content (OWC) (%)	20.08	22.67	
Free swelling index (%)	70.00	40.00	IS 2720-40 (1977)
Free swell ratio (FSR)	1.70	-	Sridharan and Prakash (2000)
pH value	7.50	11.50	Eades and Grim (1966)
Soil Classification	MI or OI*	-	IS 1498 (1970)
Degree of expansion	Medium	-	
Swelling Percentage (%)	2.53	0.00	IS (Indian Standard) 2720 (Part XV) (1986)

*MI or OI: Silt of Intermediate Plasticity (MI) or Organic soil of Intermediate Plasticity (OI)

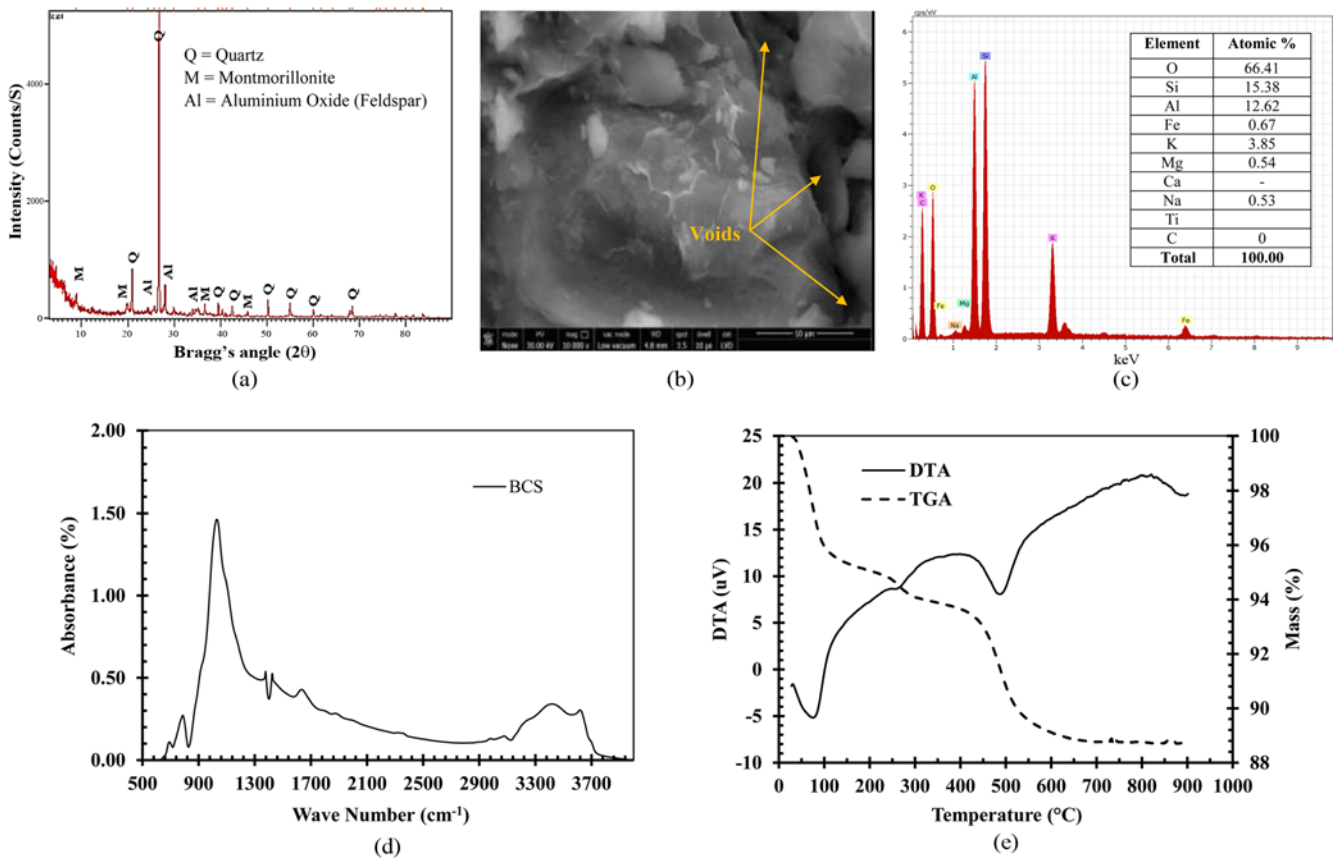


Fig. 1. Characterizations Using: (a) Mineralogical Analysis, (b) Microstructural Image, (c) Chemical Composition, (d) FTIR Analysis, (e) Thermal Analysis of Parent Soil

appearance of lower specific gravity of the present soil. The free swell index and liquid limit of soil are 70% and 49%, respectively.

Hence, the soil is classified as a “Medium” degree of expansion having a marginal degree of severity (Sridharan and Prakash,

2000).

The microanalyses of soil are presented in Figs. 1(a) – 1(e). The mineralogical analysis of soil reveals the montmorillonite, quartz, and aluminium oxide (feldspar) (Fig. 1(a)). The SEM image shows the staking of thin sheet-like particles and the voids within the soil mass (Fig. 1(b)), confirming the presence of montmorillonite. The chemical composition of soil reveals the presence of the predominant amount of Aluminium (Al) and Silica (Si) with potassium (K), magnesium (Mg) and sodium (Na) as minor elements (Fig. 1(c)). Further, chemical analysis confirms the absence of sulphur or sulphate in the soil.

The FTIR analysis of soil (Fig. 1(d)) revealed the existence of hydroxyl group (OH) and water at the $3,600\text{ cm}^{-1}$ and $3,417\text{ cm}^{-1}$ wavenumbers, respectively (Madejova et al., 2002). The band at $1,050\text{ cm}^{-1}$ represents the Si-O of clay mineral, endorsing the montmorillonite mineral in the soil. Further, the presence of quartz and combined Si-O and Al-O are indicated by the bands at the wavenumbers of 785 cm^{-1} and 620 cm^{-1} , respectively (Eisazadeh et al., 2012).

Thermal analyses (TGA and DTA) of soil are presented in Fig. 1(e). The total mass loss of soil subjected to a temperature up to $1,000^\circ\text{C}$ is observed to be 11.30%. Further, the major endothermic peaks of soil at $50 - 100$, $250 - 300$, and $450 - 550^\circ\text{C}$ reveal the losses of hygroscopic water, adsorbed water and, OH⁻ groups of smectite, respectively (Al-Mukhtar et al., 2014). However, the lower intensity endothermic peak at $850 - 900^\circ\text{C}$ confirms the change and recrystallization of quartz (Sudo and Shimoda, 1970).

2.1.2 Chemical Additives

Commercially available laboratory reagents i.e., hydrated lime [$\text{Ca}(\text{OH})_2$] and sodium sulphate [Na_2SO_4] are used as stabilizing and contaminating agents, respectively. The Na_2SO_4 is used to prepare the sulphate contaminating water of varying concentrations (0 to 30,000 ppm).

2.1.3 Sample Preparation

Detailed experiments are performed to examine the effects of sulphatic contaminations on the behaviour of untreated and lime treated expansive soil. Two categories of samples are prepared for tests i.e., untreated soil (i.e., without using lime) and lime treated soil (i.e., with using optimum lime content of 6%), respectively. The desired proportions of soil and lime are taken based on the dry weight of soil which is, then, mixed uniformly prior to wet mixing. Wet mixing of untreated and treated soil is conducted using laboratory synthesized sulphatic water of varying sulphate concentrations (i.e., 1,000, 3,000, 5,000, 8,000, 10,000, 16,000, 20,000 and 30,000 ppm). For the preparation of sulphatic water of a particular sulphate concentration, the required amount of Na_2SO_4 powder based on its molecular weight is dissolved in distilled water. A similar procedure as adopted by Shivanshi et al. (2021) to prepare the sulphate contaminated water using gypsum is followed in the present study. However, the properties of samples prepared by distilled water are also

determined to compare the same subjected to sulphate contamination.

2.2 Methodologies Followed

2.2.1 Geotechnical Properties

The effects of expansive soil with and without lime at varying sulphatic molar solutions (i.e., 0 – 30,000 ppm) are investigated by performing Liquid Limit (LL), Plastic Limit (PL) and Shrinkage Limit (SL), compaction characteristics (MDD and OWC) and Unconfined Compressive Strength (UCS) at varying curing periods up to 28 days.

The specific gravity of parent soil is observed by the test conducted using the density bottle method as per Indian Standard (IS) 2720-Part 3 (1980). The Atterberg's limit tests are performed as per IS 2720-Part 5 (1985). The MDD and OWC of all the samples are determined using mini compaction procedures (Sridharan and Sivapullaiah, 2005).

The UCS tests are conducted as per IS 2720-Part 10 (1973). The specimen of height 76 mm and diameter 38 mm compacted at their respective MDD and OWC are prepared for the UCS test. Two identical specimens are prepared for each combination. To achieve the desired curing periods, the compacted samples are placed in zip-lock polybags which are then kept in a sealed desiccator to avoid any moisture loss. On completion of each curing period, the UCS tests are performed by maintaining a 1.2 mm/minute strain rate. The peak stress values from stress-strain curves are taken. If the difference between peak values of both identical samples is more than 10%, samples are rejected, else the average value of two tested samples is considered as a final UCS value.

2.2.2 Physicochemical Properties (pH and EC)

The pH tests as per procedures of Eades and Grim (1966) are followed to get the optimum lime content (OLC) to be used for soil treatment. According to this method, lime content beyond which the pH value is constant is taken as an OLC. The pH test is performed by taking the different percentages of lime from 2 – 10%. The mixtures to measure the pH are prepared in the ratio of 1:5 (Soil-lime: Distilled water). Standard buffer solutions of 4.0, 7.0, and 9.20 are used to calibrate the pH meter. The soil-lime mixtures are stirred in regular intervals for complete dilution of lime powder and the pH is measured after 1.5 hours. The pH becomes constant after 6% of lime is used in soil and hence, the same lime content is considered as an OLC to treat the soil. However, the pH and EC values of untreated soil are measured by following the procedures of IS 2720-Part 26 (1987) and IS 14767 (2000), respectively. Further, the changes in pH and EC of treated soil upon sulphate contamination and with curing periods are examined in the samples obtained from the fractured portion after the UCS test. Digital pH meter and portable EC meter devices are used to measure both physicochemical parameters of samples.

2.2.3 Microanalysis

The fractured samples of UCS are used for microstructural investigation including SEM, XRD, EDAX, FTIR, TGA, and

DTG.

The XRD is performed to know the mineralogical composition of the material. It is conducted by using Cu-K α radiation and graphite monochromator. The scanning angle (2θ) is kept between $3^\circ - 90^\circ$. The XRD patterns are developed by using software “X’pert Highscore”.

The SEM coupled with EDAX is performed by using a small quantity of oven-dried soils which are then placed on the aluminium mounting disk or SEM stubs by using carbon tape. The sample is coated with a 100-Å thin layer of gold-palladium for 38 s with polaron E5100 at 10^{-3} Torr Vacuum which eliminates the charging problem during analysis.

The FTIR of samples are performed by using Bruker’s Alpha II model which is performed for the frequency ranges measured in terms of wavenumbers of $5,000 - 500 \text{ cm}^{-1}$.

The TGA and DTG are used to determine the thermal properties of samples. TGA is a technique in which the mass (weight) of a sample is heated and then cooled at a controlled rate and, in a specified atmosphere is noted as a function of temperature or time. DTG describes the first derivative of the TG curve with respect to either time or, temperature.

3. Results and Discussion

3.1 Effect of Sodium Sulphate Contamination on Physical Properties of Untreated and Lime Treated Soil

The effects of Na_2SO_4 contamination on the physical properties (LL, PL, PI and SL) of untreated and treated soil with lime are presented in Fig. 2.

For untreated soil, it is observed that both LL and PL reduce instantaneously upon sulphate contamination and are observed to be constant thereafter (Fig. 2). The derivative of LL and PL, i.e., PI reduces from 20.44 to 13.13%. The variation in plasticity behaviour of untreated soil upon Na_2SO_4 contamination may be attributed to the intrusion of monovalent sodium (Na^+) ion which neutralizes the negative charge on the clay surface and also generates more hydrolyzed cations. The monovalent Na^+ ion due to its lower positive charge has a tendency to attract to the surface of clay which also increases hydro-spheres on the clay particles. This finally leads to the increase in the repulsion and thereby, reducing the particle-to-particle interaction significantly. Further, an increase in monovalent Na^+ ion concentration having a higher hydrolysing tendency allows the particle-to-particle interaction due to the compression in the diffuse double layer (DDL) within the clayey particles (Marks and Halliburton, 1970; Kinuthia et al., 1999). This phenomenon ultimately enhances the resistance to flow and subsequent shear stress, controlling the further reduction in the LL with an increase in Na_2SO_4 concentration. The reduction in LL is attributed to the possible growth of short-term reactions at intermediate concentrations which are later affected due to higher electrolytic concentration (Thyagraj and Zodinsanga, 2014). The result is also in the agreement with Rajasekaran (2005).

For lime treated soil, the LL and PL values at lower sulphate

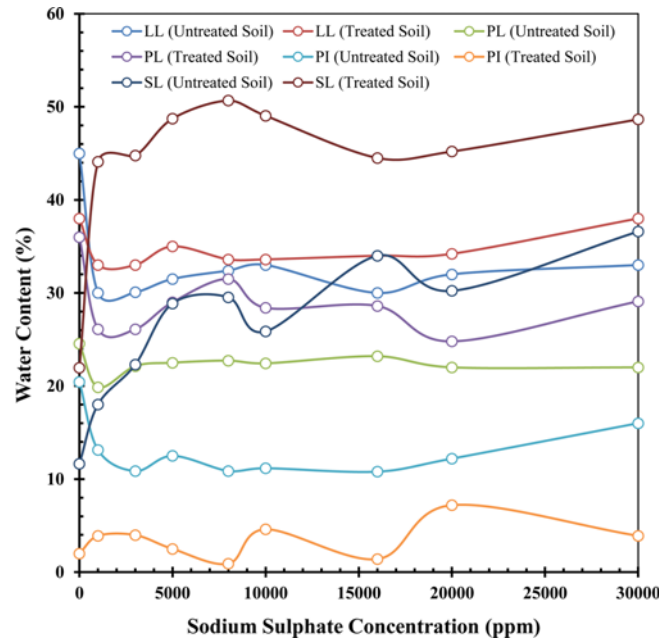


Fig. 2. Effect of Na_2SO_4 Contamination on the Physical Properties of Untreated and Treated Soil with Lime

concentrations of 1,000 ppm reduce from 38.00 to 33.00% and 36.00 to 26.10%, respectively (Fig. 2). This is due to the effect of lime supersedes sulphate immigration. Also, the concentration of Na_2SO_4 is low enough to cause any considerable disruption in the soil-lime flocculated matrix. However, the LL and PL values of lime treated soil are influenced marginally with a higher sulphatic concentration beyond 1,000 ppm. In comparison to untreated soil, both LL and PL of lime treated soil show a considerable increment in values at any sulphatic concentration.

The ionic valence and electronic charge control significantly to the DDL of the clay-water-electrolyte system. The ion having smaller in size and higher in charge imposes more rigid structure and vice-versa. Further, imposed rigid structure varies with the position of ions in the Hofmeister or lyotropic series (i.e., $\text{Li}^+ < \text{Na}^+ < \text{Mg}^{2+} < \text{Ca}^{2+} < \text{B}^{2+} < \text{Al}^{3+}$). Considering this series, the larger hydro-sphere of Na^+ results in more particle-to-particle separation as compared to Ca^{2+} , leading to the reduction in the LL. Thus, the influence of intrusion of Na^+ ion through soluble Na_2SO_4 is countered by the supply of Ca^{2+} ion, controlling the further reduction in LL of treated soil as compared to the same with the untreated soil.

It is observed that the SL of untreated soil is observed to be lesser as compared to treated soil irrespective of sulphate concentration (Fig. 2). Further, the SL values of both untreated and treated soil with lime increase up to 8,000 ppm sulphate concentration (Fig. 2). However, the reduction in SL of treated soil has been examined with sulphate concentration beyond 8,000 ppm. This is attributed to the alterations in the fabric of the soil due to reducing surface area and, thereby formation of flocculated structures. Similar results are also reported by previous researchers (Sivapullaiah et al., 2000; Thyagaraj and Zodinsanga,

2014; Raja and Thyagaraj, 2019).

3.2 Effect of Sodium Sulphate Contamination on Compaction Characteristics of Untreated and Treated Soil

The MDD and OWC of samples upon Na₂SO₄ contamination are illustrated in Fig. 3.

It is noticed that the lime treatment of soil reduces the MDD with an increase in OWC. This is a well-known fact that the flocculation of soil matrix caused due to the lime treatment increases the pore sizes and thereby lowering MDD and increasing the OWC. Sulphate contamination of both soils pronounces an increase in MDD and reduction in OWC (Fig. 3). However, the alterations in MDD and OWC of both untreated and treated soil with lime subjected to Na₂SO₄ contamination are pronounced to be marginal. Further, MDD and OWC of untreated soil are observed to be higher and lower than that of the treated soil with lime at any Na₂SO₄ concentration, respectively (Fig. 3). The mechanisms behind alteration in the compaction characteristics of untreated soil are mainly due to the particles-to-particles interaction upon sulphate contamination. As discussed in an earlier section, the contamination of untreated soil with soluble Na₂SO₄ concentration supplies an abundant amount of monovalent Na⁺ ions which enhance the particles-to-particles separation, deflocculation as well as dispersion. Thus, it reduces the resisting capacity of untreated soil against external compactive effort and, water holding capacity within the flocs, leading to the increase in MDD and reduction in OWC.

However, this is not a similar situation in the case of treated soil upon sulphate contamination. Multiple factors that can affect the compaction characteristics are; 1) reactions among ions, 2) cation position in lyotropic series, 3) pozzolanic reaction, and 4) ettringite formation. Treatment of soil with lime enhances the

flocculation and agglomeration due to short term reaction (i.e. ionic exchange). The flocculation and agglomerated soil structure can withstand the compactive effort as well as hold more water to flocs, leading to the reduction in MDD. However, migration of monovalent Na⁺ ions dominates the effect of soil-lime reactions which result in the increase in MDD and reduction in OWC in lime treated soil. As per cation position in lyotropic series, the size and electronic charge of monovalent Na⁺ ions are lower as compared to the same with divalent Ca²⁺ ions. Kinuthia et al. (1999) reported that the presence of monovalent ions like Na⁺ and divalent ions like Ca²⁺ restricts the reduction and increase in MDD, respectively. Owing to this fact, the increase in MDD of lime treated soil but its reduction as compared to untreated soil subjected to any Na₂SO₄ concentrations is due to the combined effect of monovalent Na⁺ and divalent Ca²⁺ ions. Gadouri et al. (2017) reported that Na₂SO₄ acts as an accelerator to enhance the pozzolanic reactions. The acceleration in pozzolanic reaction releases the silica during the formation and growth of ettringite by the reaction among aluminium-calcium-sulphate with water. The release of silica reacts with calcium to form silicate hydrated cementitious compounds which act as a gel to bind the particles and form a denser matrix, thereby reduction in the MDD of treated soil upon sulphate contamination (Fig. 3). Further, the growth of ettringite increase the size of pore within the soil matrix, resulting in disturbance in the matrix (Rajasekaran, 2005; Jha and Sivapulaiah 2015). Increased pore size and disturbance in the matrix lead to the increase in OWC and reduction in the MDD of treated soil subjected to Na₂SO₄ contamination. The results are in agreement with previous researchers (Kinuthia et al., 1999; Gadouri et al., 2017; Abdi et al., 2020; Raja and Thyagraj, 2019).

3.3 Effect of Sodium Sulphate Contamination on Strength of Untreated and Treated Soil

The UCS of untreated and treated soil upon Na₂SO₄ contamination at varying curing periods is shown in Figs. 4(a) – 4(b).

It is observed that the UCS of untreated soil varies upon both Na₂SO₄ concentration and curing periods. Fig. 4(a) shows the two peak bands of improvement in the strength of untreated soil cured for 7 and 14 days. In general, it is revealed that lower Na₂SO₄ up to 5,000 ppm has improved the strength of untreated soil up to 7 days curing periods. An increase in the Na₂SO₄ concentration has marginal or decline in the strength of treated soil up to 28 days. Further increase in the Na₂SO₄ concentration up to 16,000 ppm has improved the strength of untreated soil and declined marginally thereafter. It is interesting to observe that irrespective of sulphate concentration, an improvement in the strength is observed only up to 14 days of the curing period. The strength of untreated soil subjected to higher sulphate content beyond 10,000 ppm reduces significantly with the curing period of 28 days. The results are in line with the statement that several parameters such as ionic exchange, dispersion and flocculation of soil particles, and most importantly coating of sulphate ion on the surface of clay particles etc. control the beneficial and adverse strength of untreated soil (Holm et al., 1983).

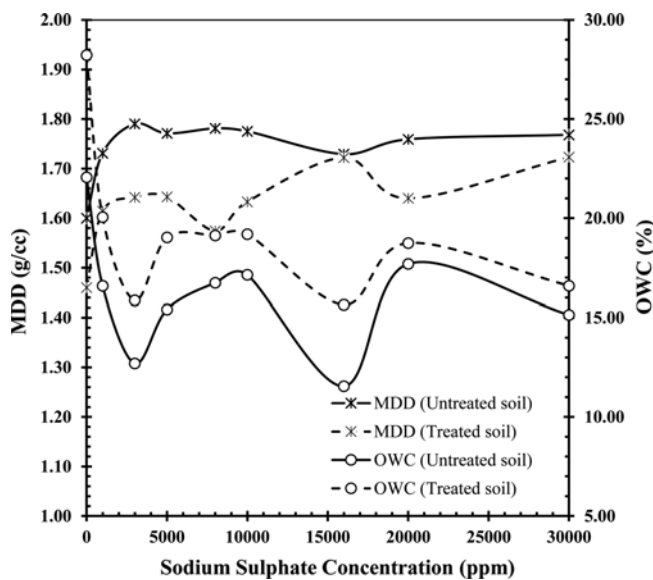


Fig. 3. Effect of Na₂SO₄ Contamination on Compaction Characteristics on Untreated and Treated Soil with Lime

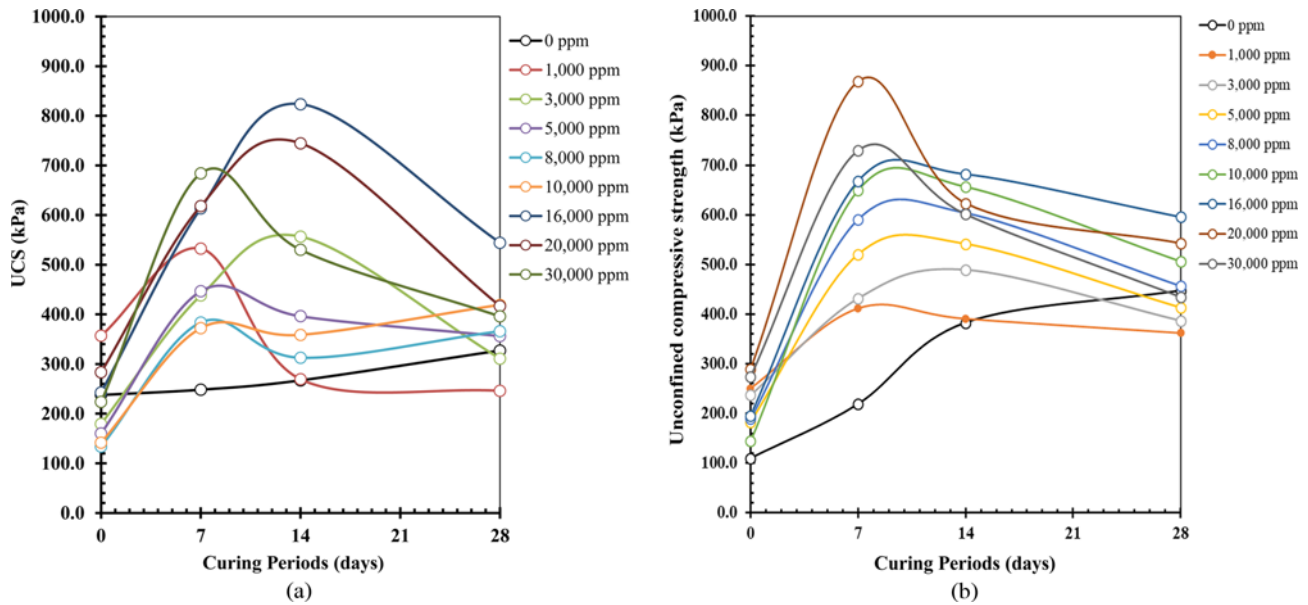


Fig. 4. Effect of Na₂SO₄ Contamination on UCS of: (a) Untreated Soil, (b) Treated Soil with Curing Periods

The UCS of treated soil subjected to different Na₂SO₄ concentrations up to 30,000 ppm are presented in Fig. 4(b). It is observed that the lime treatment improves the strength of soil with curing periods which is due to pozzolanic reactions and the corresponding formation of cementitious compounds. However, the strength of treated soil upon Na₂SO₄ contamination is observed to be susceptible. The sulphate contamination pronounces to be favourable in terms of early strength improvement in the treated soil. The strength of treated soil upon Na₂SO₄ contamination is accelerated significantly at 7 days. The ultimate improvement in the strength of treated soil with lime is observed up to 20,000 ppm. The strength acceleration in lime treated soil subjected to 20,000 ppm at 7 days is observed to be 3.96 folds more as compared to the same with treated soil without Na₂SO₄ contamination. The acceleration in the strength may be due to the reaction between Na₂SO₄ and lime thus forming gypsum (CaSO₄) and sodium hydroxide (NaOH) which enhance the alkalinity within the system, leading to the enhancement in the dissociation of silicate (SiO₂) and aluminate (Al₂O₃). The rapid dissolutions of SiO₂ and Al₂O₃ react with hydroxide (OH⁻) and calcium ion (Ca²⁺) as liberated from lime to form the silicate and aluminate hydrated cementitious compounds. Further, dissolution of Al₂O₃ in reactions with Ca²⁺ and SO₄⁻ ions form the ettringite crystals which act as a reinforcement to the lime treated soils, resulting in increase in the strength of the soil. Thus, the catalytic behaviour of Na₂SO₄ and thereby acceleration in the formation of cementitious gel/compounds and ettringite crystal contributes to early strength (Wild et al., 1993).

The strength of treated soil, irrespective of Na₂SO₄ concentration, has declined continuously after 7 days (Fig. 4(b)). The decrement in the strength is pronounced to be more with an increase in the higher sulphate content. The results reveal that optimum Na₂SO₄ contamination up to 20,000 ppm is identified as beneficial for

early strength improvement of lime treated soil. The decrease in strength may be due to the reduction in the pozzolanic ability of cementitious gels due to the adsorption of sulphate ions on their surfaces (Mehta, 1983; Sridharan et al., 1995; Shi and Day, 2000a; Gadouri et al., 2016). It is also observed that a significant decrement in strength is visible at higher curing days. Further, formation of pores, development of cracks and precipitation of gypsum crystal having lower density due to the continuous growth of ettringite crystals, rapid hydration process and reactions between Ca and SO₄ lead to the decline in the strength of lime treated soil upon higher Na₂SO₄ concentration at longer curing periods (Hunter, 1988; Sridharan et al., 1995; Tsatsos and Dermatas, 1998; Jha and Sivapullaiah, 2015; Gadouri et al., 2017).

3.4 Effect of Sodium Sulphate Contamination on Physicochemical Properties of Untreated and Lime Treated Soil

The physicochemical characteristics control the plasticity behaviour and fabric of soil subjected to different pore fluids which affect ultimately the geotechnical properties to a great extent (Sivapullaiah et al., 2000; Raja and Thyagaraj, 2019). Thus, physicochemical analyses (i.e., pH and EC) of both untreated and treated soil with lime subjected to varying Na₂SO₄ concentrations are conducted on samples collected after UCS tests. The pH and EC values are presented in Figs. 5(a) and 5(b).

The variation in pH of untreated soil has been observed up to a lower Na₂SO₄ concentration of 5,000 ppm (Fig. 5(a)). The pH of cured untreated soil increases initially at 1,000 ppm and reduces thereafter up to 5,000 ppm. Further increase in the Na₂SO₄ concentration has shown no effect in the alkalinity of untreated soil. This may be due to the fact that Na₂SO₄ is considered a neutral salt having a pH of 7. The variation in pH of untreated soil initially with lower Na₂SO₄ concentration may be due to the change in

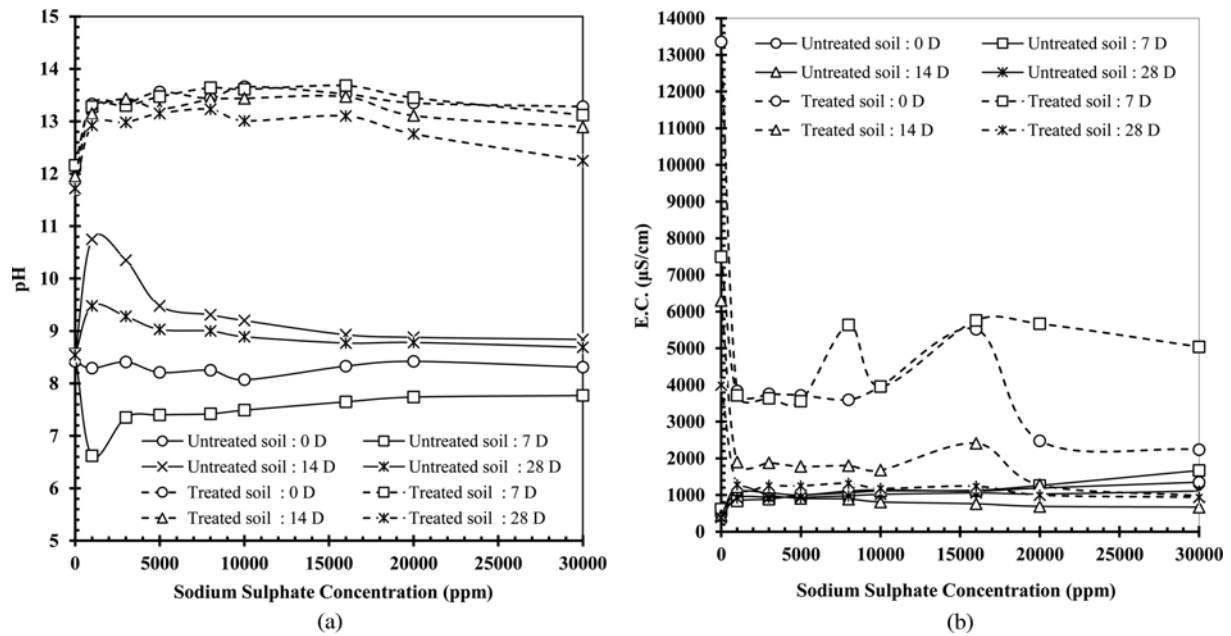


Fig. 5. Effect of Na₂SO₄ Contamination on: (a) pH, (b) EC of Untreated and Lime Treated Soil

the electrolyte solution. Because, ionization of Na₂SO₄ releases both Na⁺ cation and anion into the system which contributes information of the sodium hydroxide (NaOH) (i.e., strong base), and sulphuric acid (H₂SO₄) (i.e., strong acid). Further, EC of untreated soil cured for different periods has also shown marginal variation with Na₂SO₄ concentration (Fig. 5(b)). This neutral behaviour of Na₂SO₄ has pronounced the least effect on the DDL and corresponding change in the fabric of soil, resulting in marginal variations in the plasticity of untreated soil (Fig. 2).

The pH of treated soil increases upon Na₂SO₄ contamination up to 16,000 ppm and reduces afterwards. This is attributed to the dominant effect of the formation of NaOH over H₂SO₄. Further, it is to note that the pH of the samples reduces with the curing periods. This confirms the consumption of OH⁻ ions in the pozzolanic reactions and the formation of ettringite. Similar behaviour is observed by Raja and Thyagaraj (2019). However, decrement in the pH of treated soil upon contamination with higher sulphate concentration beyond 16,000 ppm at any curing periods may be due to the formation of H₂SO₄ with the supply of excessive SO₄ ions. Similar behaviour with a decrement in EC of treated soil is observed with curing period and increase in the concentrations of Na₂SO₄ (Fig. 5(b)). The increase in alkalinity results in the formation of a more flocculated matrix and a reduction in pH with curing periods indicates the cementitious compound formation. Thus, binding and filling of the flocculated soil matrix with cementitious compounds results in the development of a stronger matrix, leading to the increase in the UCS of lime treated soil (Fig. 4).

4. Microanalyses

4.1 Mineralogical Analysis

The XRD analyses of samples subjected to Na₂SO₄ contamination

at different curing periods of 14 and 28 days are presented in Figs. 6 and 7, respectively.

It is observed that the XRD of untreated soil subjected to 16,000 ppm sulphate concentration at 14 days curing period pronounces the Sodium Silicate Hydrate (SSH), Sodium Aluminum Silicate (SAS), Sodium Silicate (SS), Sulfur (S) along with Montmorillonite (M), and Quartz (Q) peaks (Fig. 6(a)). However, peaks related to cementitious compounds have not appeared, revealing the influences of ionic exchange, dispersion, and flocculation of soil particles in the strength improvement (Fig. 4(a)). Similar observations are pronounced in the XRD analysis of the same sample cured for 28 days (Fig. 7(a)). The intensity of the related peaks is appeared to be weaker than that of the peaks of the sample cured for 14 days. This confirms the alteration in the mineralogy with curing periods.

The XRD analyses of treated soil subjected to Na₂SO₄ contamination cured for 14 and 28 days are shown in Figs. 6(b) – 6(d) and 7(b) – 7(f), respectively. The XRD analyses show the formations of cementitious compounds (CASH, CASHH, and CSHH) and ettringite (E). In comparison, the broader peaks of CSHH and ettringite along with SS have been observed in treated soil with 20,000 ppm (Fig. 6(c)). The formations of silicate hydrated cementitious compound and SS reveal the release of silica during the formation of ettringite. Hence, the formation of CSHH acts as a cementitious gel to bind the particles, and to fill the pores whereas the formation of ettringite within the soil matrix reinforces the soil particles, resulting in an increase in the strength of lime treated soil at shorter curing periods (Fig. 4(b)). The contamination of treated soil with a higher sulphate concentration of 30,000 ppm at 14 days shows the weaker cementitious compound peaks, confirming the coating of sulphate on the surface of the cementitious compound

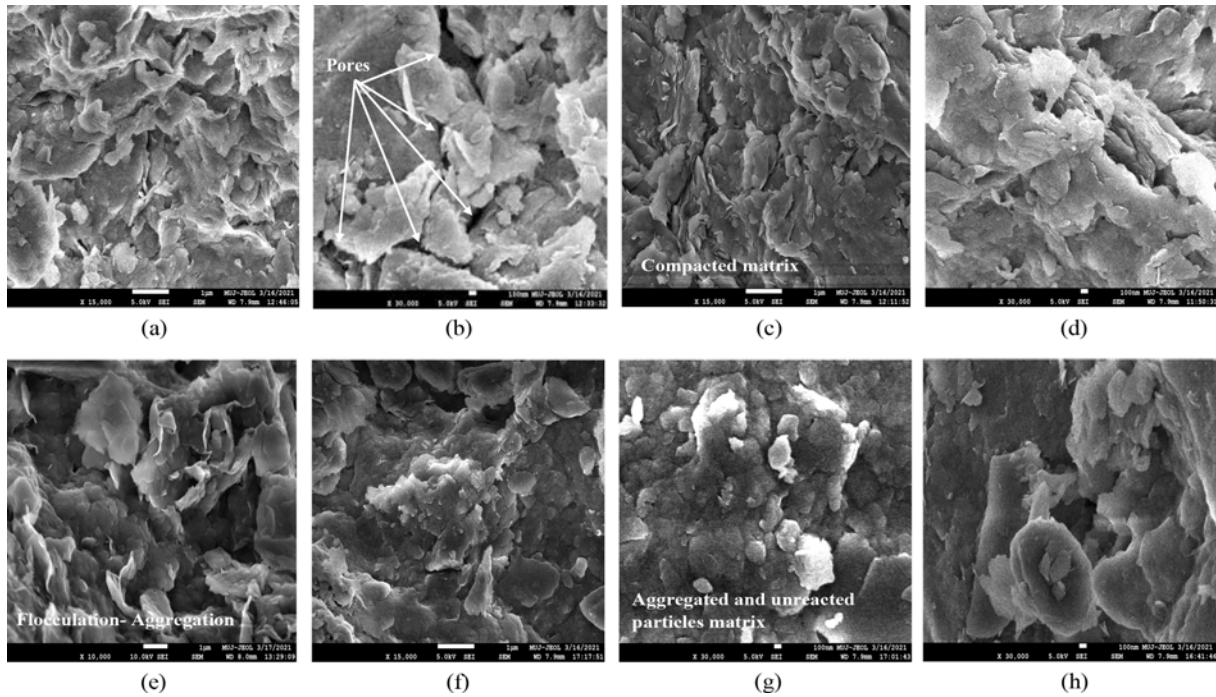


Fig. 8. SEM Images of Untreated Soil at Different Concentration of Na_2SO_4 Solution; 16,000 ppm: (a) 0 D, (b) 7 D, (c) 14 D, (d) 28 D; 20,000 ppm: (e) 0 D, (f) 7 D, (g) 14 D, (h) 28 D

EDAX results (Table 2). However, the formation of ettringite needles and cementitious gels take place simultaneously depending upon sulphate concentration and curing periods. It is observed that the SEM images of treated soil at 1000 ppm at different curing periods (Fig. 9(a) – 9(c)) have shown aggregated matrix with the formation of the lower amount of ettringite crystal. An increase in Na_2SO_4 concentration up to 10000 ppm has shown the nucleation and growth of ettringite needles within the pores/voids of treated soil (Figs. 9(d) – 9(i)). However, SEM images of treated soil upon sulphate contamination at longer curing periods show the disturbed matrix (Figs. 9(f) and 9(i)), leading to the reduction in strength (Fig. 4(b)).

The SEM images of treated soil upon sulphate contamination of 16,000 and 2,000 ppm at different curing periods up to 28 days are illustrated in Figs. 9(j) – 9(m) and 9(n) – 9(q) and have appeared similar microstructure development. The size and amount of fibrous ettringite needles are pronounced at lower curing periods. However, an increase in the curing periods diminishes the formations of ettringite needles and cementitious compounds (Figs. 9(l) and 9(m) and 9(p) and 9(q)). Whereas, it is observed clearly that samples cured for 7 days shows the compacted matrix with the formation of ettringite crystals within pores which act as reinforcing materials (Figs. 9(k) and 9(o)), resulting in an ultimate enhancement in the strength at lower curing periods (Fig. 4(b)). It is interesting to illustrate that microstructural images of treated samples subjected to a higher sulphate concentration of 30,000 ppm have pronounced the aggregated matrix with formations of ettringite crystals, particularly at 14 days curing period (Fig. 9(s)), leading to the increase in strength.

However, the presence of sulphate ions leads to the reduction in the cementing ability of silicate hydrated gel, resulting in restriction in the enhancement in strength.

4.3 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analyses of samples for curing days 14 and 28 days are shown in Figs. 10(a) and 10(b).

The spectrums of both soils are analyzed within the band of $500 - 4,000 \text{ cm}^{-1}$. It is observed that the change in sulphatic concentrations and lime treatment to soil results in the intensity of transmittance and bandwidth. However, the characteristic bands of untreated and treated samples are observed at around the same wavenumber (cm^{-1}) at any curing period. FTIR analysis explains the molecular alteration that takes place in the soil in presence of additives (Sharma et al., 2018). The characteristic bands between $3,500 - 4,000 \text{ cm}^{-1}$ indicate the presence of structural hydroxyl (OH^-) (Madejova et al., 2002). The sharpness of bands between $3,500 - 4,000 \text{ cm}^{-1}$ reduces with the Na_2SO_4 concentration. The band at $2,500 \text{ cm}^{-1}$ represents the CO_2 peaks that may be derived freely from the environment. Two bands of high intensity between $1,500$ to $2,000 \text{ cm}^{-1}$ illustrate the H-OH in the interlayer regime of montmorillonite and CO_3^{2-} compounds (Grim et al., 1961; Morris, 1963; Bahmani et al., 2014). The intensity of bands decreased with increasing sulphate concentration. Also, the intensity and bandwidth are reduced with the increasing curing period (Latifi et al., 2013). For both untreated and treated samples, a stronger band around $1,030 - 1,050 \text{ cm}^{-1}$ has appeared which is associated with the stretching vibration of the Si-O group and is due to the sheet of alumina-silicate-lattice (Smectite

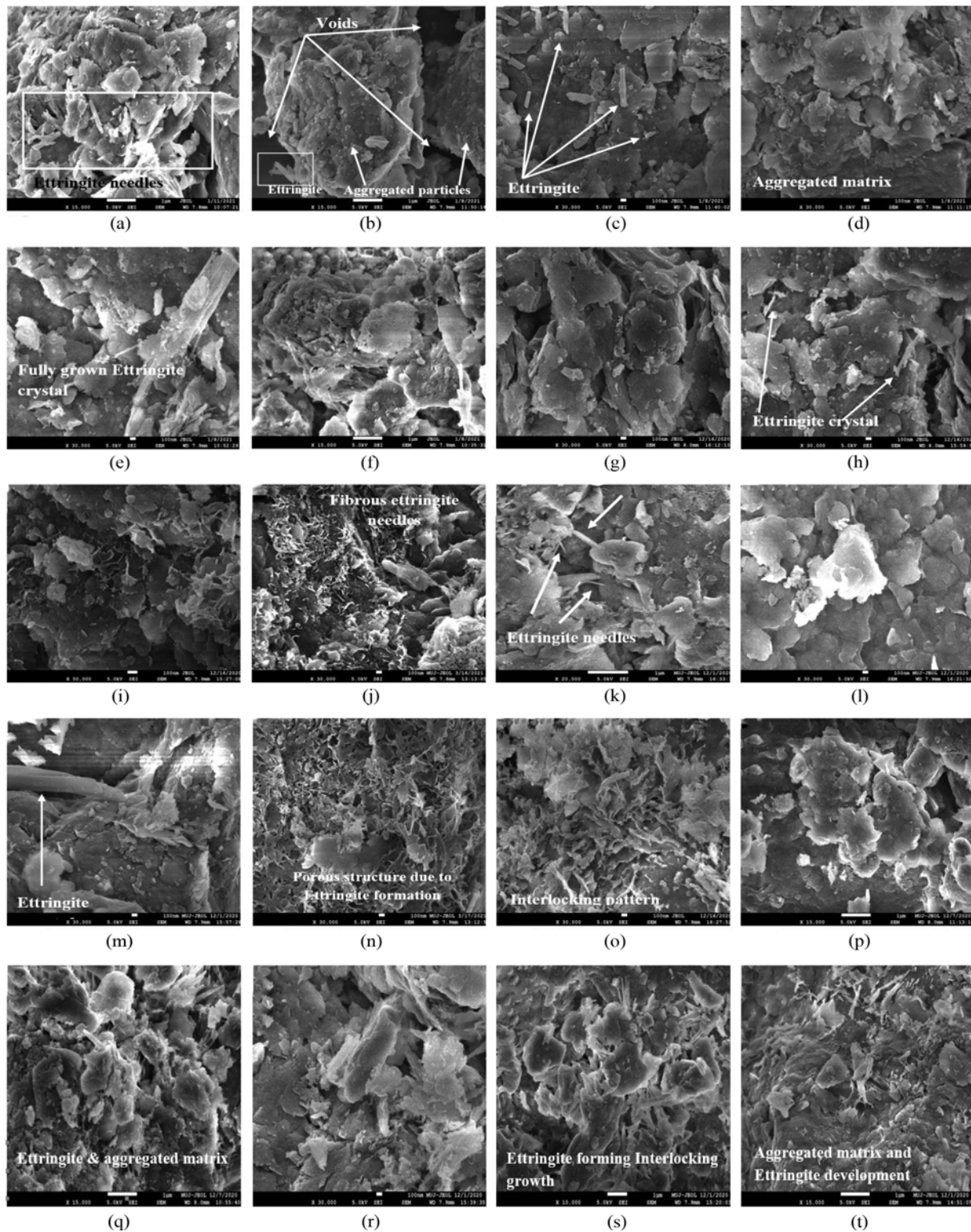


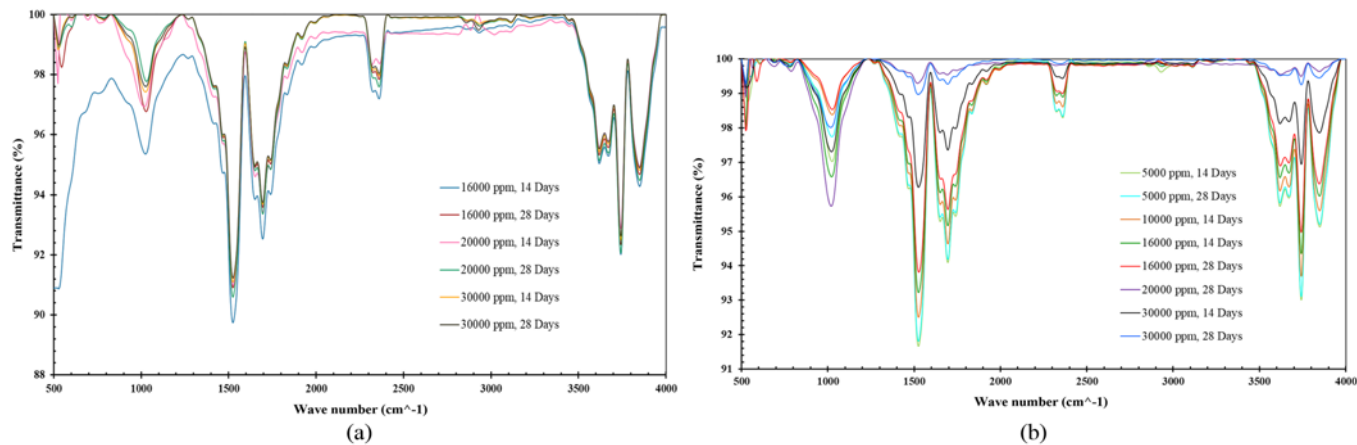
Fig. 9. SEM Images of Lime Treated Soil Subjected to Na_2SO_4 Solution; 1,000 ppm: (a) 7 D, (b) 14 D, (c) 28 D; 5,000 ppm, (d) 7 D, (e) 14 D, (f) 28 D; 10,000 ppm, (g) 7 D, (h) 14 D, (i) 28 D; 16,000 ppm, (j) 0 D, (k) 7 D, (l) 14 D, (m) 28 D; 20,000 ppm: (n) 0 D, (o) 7 D, (p) 14 D, (q) 28 D; 30,000 ppm, (r) 7 D, (s) 14 D, (t) 28 D

group) (Bahmani et al., 2014). Further, it has also been reported that the band near 1011 cm^{-1} also identifies the presence of sulphate (SO_4^{2-}) ion for ettringite (Scholtzova et al., 2015). The

observed band is sharper in the treated soil with the increasing concentration of Na_2SO_4 (Fig. 10(b)). The characteristic spectrums of untreated and treated soil show the group of Al-O-Si bending

Table 2. EDAX Analysis of Untreated and Treated Soil with Lime in Presence of Sulphate Contamination at Different Curing Period

Samples	Sodium Sulphate conc., ppm	Curing Days	Atomic %											
			O	Na	Mg	Al	Si	S	Ca	Si/Ca	Al/Ca	Si/Al	S/Na	
S	16,000	0	68.6	0.8	3.1	9	15.6	1.5	1.4	11.1	6.4	1.7	1.9	
		7	61.3	0.6	2	10.7	19.9	3.0	2.5	7.8	4.2	1.9	5.0	
		14	68.4	0.9	2.5	9.5	15.9	1.9	0.8	19.9	11.9	1.7	2.1	
		28	62.4	0.9	2.6	11.7	19.1	2.3	1.1	17.4	10.6	1.6	2.6	
	20,000	0	67.3	0.5	1.9	9.5	18.0	1.8	1	18.0	9.5	1.9	3.6	
		7	67.6	0.7	2.1	9.8	17.2	1.7	0.8	21.5	12.2	1.7	2.4	
		14	58.8	0.6	0.8	2.6	34.9	1.8	0.5	69.8	5.2	13.4	3.0	
		28	66.5	0.7	2.2	10.0	17.7	1.6	1.3	13.6	7.7	1.8	2.3	
S+L	1,000	7	69.5	0.8	1.9	8.9	16	0.6	2.4	6.7	3.71	1.8	0.7	
		14	71.3	0.2	3.0	8.4	13.1	1.2	2.9	4.5	2.90	1.5	6.0	
		28	66.0	0.5	2.1	9.7	16.9	1.3	3.6	4.7	2.69	1.7	2.6	
		5,000	7	61.8	0.7	2.1	9.4	18.3	2.2	5.6	3.3	1.68	1.9	3.1
			14	66.6	0.5	1.8	8.7	17.0	1.2	4.2	4.0	2.07	1.9	2.4
			28	71.2	0.3	2.1	7.6	15.1	1.6	2.0	7.5	3.80	1.9	5.3
	10,000	7	64.5	2.2	1.6	9.9	17.0	1.7	3.1	5.5	3.19	1.7	0.7	
		14	63.3	0.4	2.2	9.8	16.9	2.5	4.8	3.5	2.04	1.7	6.2	
		28	64.7	0.5	1.8	10.1	15.5	2.1	5.3	2.9	1.91	1.5	4.2	
		16,000	0	69.8	0.8	1.9	7.8	14.0	1.5	4.1	3.4	1.90	1.8	1.9
			7	68.5	0.7	1.5	5.5	10.8	1.0	12.2	0.9	0.45	1.9	1.4
			14	66.7	0.7	2.0	7.4	18.0	1.3	3.9	4.6	1.90	2.4	1.8
	28		68.0	0.8	1.8	8.2	16.0	1.2	4.0	4.0	2.05	1.9	1.5	
	20,000		0	67.2	0.6	1.3	3.4	10.8	2.2	14.4	0.7	0.24	3.2	3.6
			7	69.1	0.6	0.9	3.0	21.9	1.3	3.3	6.6	0.91	7.3	2.1
		14	64.6	0.8	2.1	7.4	21.0	1.1	3.0	7.0	2.47	2.8	1.3	
		28	70.6	0.8	1.3	5.2	12.4	0.9	8.8	1.4	0.59	2.4	1.1	
		30,000	7	71.0	1.0	1.8	6.9	15.9	1.1	2.3	6.9	3.00	2.3	1.1
			14	67.9	0.6	1.5	7.7	14.8	1.2	6.3	2.3	1.22	1.9	2.0
	28		68.4	1.2	1.8	7.1	15.1	0.7	5.8	2.6	1.22	2.1	0.6	

**Fig. 10.** FTIR Analysis: (a) Untreated Soil, (b) Lime Treated Soil

vibration near 520 cm^{-1} and Al-O and Si-O out-of-plane vibrations around 620 cm^{-1} (Eisazadeh et al., 2012). This indicates the presence of the CSH matrix. The presence of quartz is indicated at 791 cm^{-1} (Eisazadeh et al., 2012). It is observed from the FTIR spectrum of cured samples that the impact of lime alters the functional group of soil marginally than that of sulphate concentration.

4.4 Thermogravimetric Analysis (TGA)

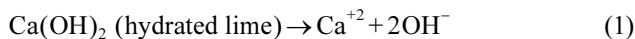
TGA analyses of samples upon sulphate contamination at different curing periods are shown in Fig. 11.

It is observed that mass loss in the untreated soil is lower as compared to lime treated soil upon sulphate contaminations which reveal the formation of cementitious gels and ettringite in the treated soil, validating the mineralogical and microstructural examination. Further, loss in mass is higher with curing periods, and up to sulphate concentration of 20,000 ppm. This reveals that the formations of cementitious gels and ettringite crystals are due to the increase in curing periods and sulphate concentration, respectively. The loss in mass is continued in general till 700°C in both untreated and treated soil with lime. Identical observation is shown by Al-Mukhtar et al. (2012).

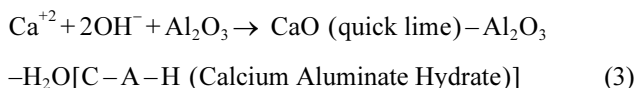
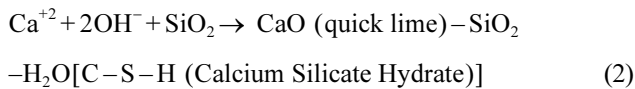
5. Mechanism of Strength Behaviour of Lime Treated Soil upon Sodium Sulphate Contamination

The improvement in the strength of soil and related mechanisms is a well-known phenomenon. However, when lime treated soil is attacked by sulphate migration, the pozzolanic reactivity of lime-soil affects significantly depending upon its concentration.

Lime in the presence of water releases Ca^{+2} cations, and OH^- anions (Eq. (1)).



High alkaline medium due to OH^- ions causes the release of aluminates and silicates from soil which then reacts with calcium ions to form aluminate and silicate hydrated cementitious compounds, respectively (Eqs. (2) – (3)).



When Na_2SO_4 migrates in the soil, it dissociates to Na^+ ions and SO_4^{2-} ions; however, lime treated soil has marginal influence due to the presence of lower Na_2SO_4 content (Eq. (4)).



Further, dissociated SO_4^{2-} and Na^+ ions react with Ca^{2+} and OH^- result in the formation of calcium sulphate (CaSO_4) and sodium hydroxide (NaOH) which have minimal influence over the strength improvement (Eqs. (5) – (6)). The phenomenon of

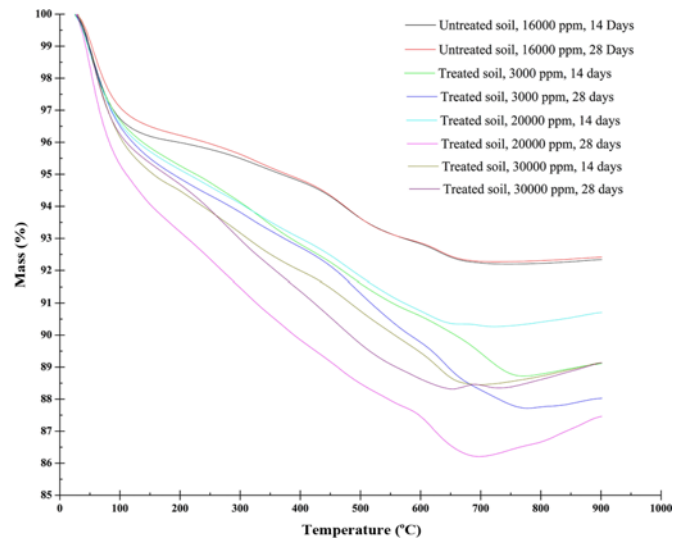
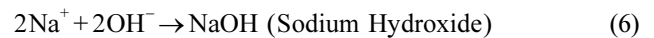
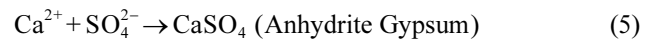
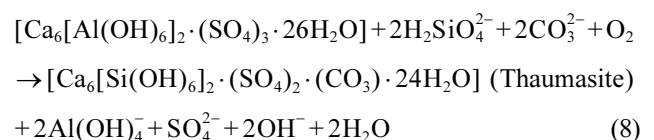
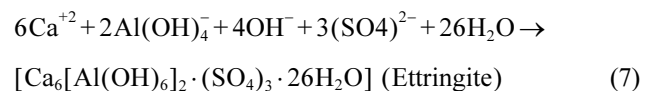


Fig. 11. TGA Analysis of Untreated and Lime Treated Soil for Cured of 14 and 28 Days

lime stabilization and sulphate intrusion are explained through chemical reactions as reported by previous researchers (Hunter, 1988; Kinuthia et al., 1999; Little et al., 2005; Puppala et al., 2005; Gadouri et al., 2016).



An increase in the concentration of sulphate hinders the mechanism of lime treatment to the soil. It is due to the consumption of the majority of the Ca^{2+} ions to form CaSO_4 with SO_4^{2-} ions (Eq. (5)). Since CaSO_4 has low solubility, it remains in the system in its own form. Simultaneously the formation of NaOH increases the alkalinity of medium, leading to the release of Si and Al ions from the soil as described by Sridharan et al. (1995). Afterwards, the Si and Al ions combine with Ca^{2+} from lime in the presence of SO_4^{2-} and water to form a highly expansive compound named ettringite $[\text{Ca}_6[\text{Al}(\text{OH})_6]_2 \cdot (\text{SO}_4)_3 \cdot 26\text{H}_2\text{O}]$ / thaumasite $[\text{Ca}_6[\text{Si}(\text{OH})_6]_2 \cdot (\text{SO}_4)_2 \cdot (\text{CO}_3) \cdot 24\text{H}_2\text{O}]$ depending upon temperature (Eqs. (7) and (8)).



The ettringite fills the pores and along with a small amount of pozzolanic compounds together contributes to acceleration in the early strength improvement at intermediate concentrations. This process of formation of ettringite begins the domination of the pozzolanic reactions with the increasing concentration of sulphates. This is attributed to the consumption of Ca^{2+} ions in

the ettringite formation, causing an insufficiency of calcium ions for the pozzolanic reactions.

Further, most sulphate ions are utilized in the formation of ettringite. However, some unreacted Na_2SO_4 in the system may be available at higher sulphatic concentrations. Therefore, strength at a higher curing period reduces due to the inability of the ettringite crystals to act as reinforcing agents (Jha and Sivapullaiah, 2015; Gadouri et al., 2017). Also, lower sulphatic content and shorter curing period restrict the magnitude of ettringite formation due to deficiency in availability of sulphate ions (Raja and Thyagaraj, 2020).

6. Conclusions

The following conclusions can be drawn from the present study:

1. Influence of soluble sodium sulphate contamination in the plasticity characteristics of untreated and lime treated expansive soil is marginal. In general, the contamination of untreated and treated soil with varying sulphate concentrations reduces both liquid limit and plastic limit with marginal influence on plasticity index. Further, the shrinkage limit of lime treated soil is observed to be higher as compared to untreated soil upon soluble sodium sulphate contamination. However, compaction characteristics of both untreated and lime treated soil show an increase in MDD with the reduction in OWC upon sulphate contaminations.
2. The contamination of untreated soil with sodium sulphate concentration up to 16,000 ppm accelerates the early strength of untreated soil and reduces thereafter up to 30,000 ppm. However, the long-term strength of untreated soil deteriorates upon any sulphate concentrations in general. Further, rapid acceleration in the lime treated soil subjected to sodium sulphate contamination up to 20,000 ppm is observed at a lower curing period of 7 days. Further, the strength of lime treated soil reduces with an increase in the higher sulphate content at longer curing periods. Hence, optimum Na_2SO_4 contaminations up to 16,000 and 20,000 ppm play a beneficial effect for early strength improvement of both untreated and lime treated soil at lower curing periods, respectively.
3. The variation in pH of untreated soil has been observed up to a lower Na_2SO_4 concentration of 5,000 ppm. The pH of lime treated soil increases upon Na_2SO_4 contamination up to 16,000 ppm and is reduced afterwards with a further increase in its concentration. While sodium sulphate contamination affects the EC of untreated soil marginally. However, the EC of lime treated soil reduces with an increase in the concentration of Na_2SO_4 and curing periods.
4. Microanalysis reveals that the presence of sulphate resulted in the aggregated matrix in untreated soil while cementitious gel and ettringite needles are witnessed in lime treated soil. The increase in strength at a shorter curing period is due to the formation of ettringite crystals which act as reinforcing materials and cementitious compounds. Further, strength enhancement at a longer curing period is restricted due to

inference in pozzolanic reactions due to excess sulphate ions at higher sulphatic concentrations.

Overall, the soluble sulphate contamination affects the behaviour of untreated and lime treated soil significantly. However, further studies need to be performed to examine the alteration in sulphate concentration through sorption capacity and intrinsic shear strength parameters for better understanding the behaviour of soils on sulphate contamination.

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