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Mitigation of Adverse Effects of Sulfates in Cement Treated Marine Clay Subgrades

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ABSTRACT: Marine clays are normally characterized by high compressibility and low shear strength, which contribute to many geotechnical problems and, at times, necessitate the need to adopt stabilization with calcium-based stabilizers. However, calcium-based stabilization, when adopted on clays rich with sulfates, causes sulfate heaving, which impacts the strength of the soil. As a result of this heaving, severe damage has occurred to transportation infrastructure such as highways, runways, tunnels, canals, etc. Numerous pavement failures attributed to sulfate-induced heave in cement-treated sulfate-bearing clay subgrades have been documented by researchers worldwide. In this study, an attempt was made to prevent the sulfate attack in cement-treated clay by introducing barium hydroxide and sulphate resisting cement. Unconfined compressive strength, CBR, liquid limit and free swell index tests were conducted on treated clay samples to determine the effect of sulfates in cement-treated sulfate-bearing clays for prolonged curing periods. On the basis of the results obtained, the incorporation of barium hydroxide produced a high CBR value of 70% and a strength gain of 551 kPa in treated clay samples, indicating the effectiveness of barium hydroxide in mitigating the adverse effects of high sulfate content in soil. It was also determined that the sulphate-resistant cement was sufficient to lessen the impact of 0.5% sulfate content in soil, but it was unable to mitigate the impacts of 4% sulfate, resulting in a significant reduction in strength of 34%.

KEYWORDS: Sulfate, Expansive soils, Marine clay, OPC, SRC, and Barium Hydroxide.

1. INTRODUCTION

The swelling and shrinkage behavior of soft marine clay covering long stretches of the coastal belt and offshore areas of the world result in many geotechnical problems. These soft soil formations have very low bearing capacity and high compressibility characteristics. Consequently, they cause excessive settlements and create distress and damage to the structures founded on them (Por *et al.,* 2015; Bared and Marto, 2017). With the increase in population, people are forced to move into coastal areas and other soft soil formations, which initiated the need for improving the engineering properties of these soils. The geotechnical behavior of these expansive soils can be enhanced significantly by utilizing calcium based stabilizers such as lime and cement. Along with the obvious improvement in the strength and stiffness of the soils, the incorporation of cement reduces both the vertical free swelling strain and the areal shrinkage strain (Por *et al.*, 2017; Chompoorat *et al*. [2019, 2021a, 2021b, 2021c, 2021d, 2022]). The improvement in engineering properties of cement treated soils is related to the formation of cementitious products in the treated soil matrix, which were produced by soil cement reactions (Croft, 1967; Kezdi, 1979; Rajasekaran and Narasimha, 2002; Parsons and Milburn, 2003; Makusa, 2012; Muhmed and Wanatowski, 2013). Either naturally or by means of industrial effluent containing sulfates, marine clays may possess substantial levels of sulfates (Rajasekaran, 1994). Groundwater contamination from sulfates occurs during the oxidation of pyrite, caused by dredging marine sediments or reclamation of bay areas containing heavy metals. These reactions lead to the formation of iron hydroxide and sulfate ions in marine clays (Kawasaki, 1988 and Ohtsubo *et al.*, 1991). In the last two decades, numerous incidents of extensive pavement heaving and structural distress have been reported when sulfate bearing clays were treated with calcium-based stabilizers (Mehta and Klein, 1966; Mitchell, 1986; Hunter, 1988; Petry and Little, 1992; Kota *et al.*, 1996; Rollings *et al.*, 1999; Wild *et al.*, 1999; Rajasekaran, 2005; Sivapullaiah *et al.*, 2006; Yong and Ouhadi, 2007; Sivapullaiah and Ramesh, 2011; Puppala *et al.*, [2014, 2018]). Several pavements founded on sulfate bearing soils that are stabilized with lime or cement, have undergone distress and severe heaving issues shortly after construction. This can be attributed to the formation of ettringite [$Ca_6{\cdot}Al_2(OH)_{12}(SO4)_3{\cdot}26H_2O$] and thaumasite [$Ca_3{\cdot}Si(OH)_6{\cdot}(SO_4){\cdot}(CO_3){\cdot}12H_2O$], expansive in nature with very large expansion potential, in some cases as high as 250% (Mitchell, 1986; Hunter, 1988; Rajasekaran, 1994; Rajasekaran, 2005; Sriram and Thyagaraj, 2021). Sulfates of sodium (thenardite, $NaSO_4.10H_2O$), potassium (arcanite, K_2SO_4), calcium (gypsum or selenite, $CaSO_4.2H_2O$) and magnesium (epsomite, $MgSO_4.7H_2O$) are commonly available in the earth's crust, especially in regions of limited rainfall (Rajasekaran, 2005; Wild *et al.*, 1999). When these soluble sulfate minerals present in soil, along with free aluminium, react with calcium from the stabilizer in a moist environment at high pH a water sensitive mineral is formed known as ettringite. This process is termed as "Sulfate Induced Heave" (Dermatas, 1995; Puppala *et al.*, [2014, 2018]). At ideal temperature, humidity and elevated pH conditions, the ettringite thus formed gets enlarged in volume due to crystal growth and hydration. This leads to expansive stresses, resulting in the failure of pavements. Based on the previous studies, soil scientists from all over the world have warned against the use of cement in sulfate bearing clays. Given the uncertainty over the level of sulfate minerals and the broad acceptance of calcium- based stabilizers for improving weak soils, an in-depth study is highly desirable. Several mitigation agents have been suggested by various researchers to hinder the effect of sulfates on calcium based stabilized soils (Ferris *et al.*, 1991; Raja, 1990; Tsatsos and Dermatas, 1998; Anitha, 2014; Zhang *et al.*, 2015; Consoli *et al.*, 2019; Seco *et al.*, 2017; Bazyar *et al.*, 2017; Puppala *et al.*, [2003, 2004, 2005, 2018]; Eyo *et al.*, [2020a, 2020b, 2021]; Mahedi *et al.*, 2020; Caselles *et al.*, 2020; Chegenizadeh *et al.*, 2020; Adeleke *et al.*, 2020; Biswas *et al.*, 2021; Ehwailat *et al.*, 2021; Jang *et al.*, 2021; Chakraborty *et al.*, 2022; Ebailila *et al.*, 2022). The studies conducted to account for the detrimental effect of sulfates on cement treated marine clay are very limited when compared to lime treated marine clay with respect to prolonged curing periods. Therefore, the present study involves the addition of sodium sulfate to cement treated marine clays to study the adverse effects of high sulfate bearing clays for a period of 270 days of curing. The study also emphasizes the effect of drying on the strength improvement of the treated soil specimens. It is aimed at mitigating the detrimental effects of sulfates in cement treated sulfate bearing clays by using barium hydroxide ($Ba(OH)_2$). The study also utilizes sulphate resisting cement instead of Ordinary Portland cement (OPC) and barium hydroxide to prevent the sulfate induced heave. It also focuses on how the amount of sulfates present in soil plays a significant role in determining the type of mitigating agent used in counteracting the detrimental effect of sulfates.

2. MATERIALS AND METHODS

2.1 Characteristics of Materials

In this experimental study, an effort is made to study the mitigation of the effects of sulfate in Cochin marine clay

(a) by introducing barium hydroxide ($Ba(OH)_2$)
(b) by using sulphate resisting cement instead of OPC and $Ba(OH)_2$

Various experimental studies point out that the clays exhibit superior sulfate-induced swelling than that in sands under comparable environmental conditions (Sherwood, 1962 and Puppala *et al.,* 2004). Marine clay that covers the long stretches of coastal regions is characterized by low strength and high compressibility. Therefore, the soil chosen for the study was Cochin Marine Clay and it was taken from Kadavanthara, Kochi, Kerala. Studies were performed on samples collected from a bore hole using the shell and auger method. The samples collected from a depth of 12-15 m were mixed thoroughly and transferred to polythene bags immediately. They were sealed tightly to preserve them under humid conditions. The main physical characteristics of Cochin Marine Clay were determined based on procedures laid down by the American Society for Testing and Materials (ASTM) and Indian Standards. To study the effect of drying on the strength improvement of cement treated clays, the clays were sun dried and their properties were also determined. The properties and grain size distribution of moist, air dried and oven dried samples of Cochin marine clay are summarized in Table 1 in order to study the effect of drying on the index properties of this soil.

Table 1 Effect of drying on the physical properties of the soil

Property	Test values		
	Moist soil	Air dried	Oven dried
Natural Moisture content (%)	99	-	-
Liquid Limit (%)	121	75	49
Plastic limit (%)	46	31	28
Plasticity Index (%)	75	44	21
Shrinkage Limit (%)	23	26	27
Natural sulfate content (%)	0.53	0.53	0.53
Grain size distribution: Clay size	40	29	12
Silt size	39	43	53
Sand size	21	28	35

The method for ascertaining the total soluble sulfate concentration of soil is outlined in IS 2720 Part 27 and Anitha *et al.,* 2011. The total soluble sulfate content of marine clay was determined in the current study using the volumetric approach, which depends upon the formation of insoluble barium sulfate when barium chloride is added to the solution (Anitha *et al.,* 2011). The obtained value of sulfate content in Cochin marine clay as per the procedure mentioned in IS 2720 Part 27, was 0.53%. The X-ray diffraction studies carried out to determine the mineralogical composition of the marine clay chosen for the present study indicated the presence of clay minerals montmorillonite, illite and kaolinite, along with non-clay minerals, quartz and afghanite. These minerals are associated with high swelling in a moist environment (Figure 1).

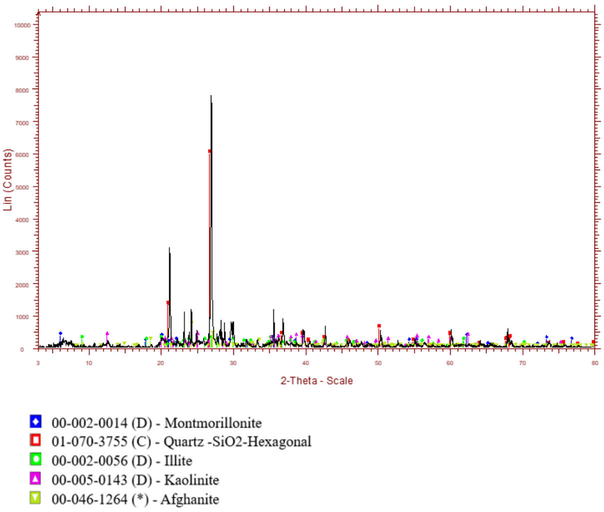

Figure 1 X-ray diffraction pattern of Cochin marine clay

Ordinary Portland Cement and Sulphate Resisting Cement were the two types of cement employed in the study. Sulphate resisting cement is anticipated to prevent sulfate attack on clays that have been treated with cement due to its low C_3A concentration (Puppala *et al.,* [2003, 2004, 2014]). Therefore, it was proposed to employ sulphate resisting cement to counteract the effects of sulfates in clay. The cement bags were preserved in an airtight bin to avoid any change in properties with time of storage. The properties of Ordinary Portland Cement and Sulphate Resisting Cement are presented in Table 2. All the tests were carried out based on ASTM standards.

Table 2 Physical properties of the OPC and SRC used in the study

Property	Test values of OPC	Test values of SRC
Initial setting time (minutes)	176	177
Final setting time (minutes)	278	240
Standard consistency (%)	36	30
Compressive strength (N/mm^2), 7 days	31.2	28.7
Compressive strength (N/mm^2), 28 days	53.2	43.5

The chemical compositions of both Ordinary Portland cement (OPC) and Sulphate resisting cement (SRC) are also reported in Table 3, respectively.

Table 3 Chemical composition of the OPC and SRC used in the study

Chemical composition	Content percentage in OPC	Content percentage in SRC
SiO_2	22.03	20.74
Al_2O_3	5.15	4.34
Fe_2O_3	4.86	5.17
CaO	65.41	64.66
MgO	1.20	1.97
K_2O	0.34	0.2
Others	1.01	2.92

Jose *et al.* (1991) investigated the effectiveness of various additives for stabilization of Cochin marine clay and the experiments revealed that lime and cement 6% by dry weight of soil gave remarkable gains in strength. Considering 6% to be optimum, both the cements, OPC and SRC were mixed with 6% by dry weight of clay in this study. Soil scientists worldwide have reported that even a minimal amount of 0.05% sulfates can cause deleterious effects on calcium based stabilized soil. In India, a maximum sulfate content of 4.10% is reported for soil samples from Madras (Rajasekaran *et al.*, 1997). Therefore, the effect of sulfates on cement treated clay has been studied on artificially prepared high sulfatic clays by adding sodium sulfate in clays. Some researchers (Raja, 1990; Ferris *et al.*, 1991; Tsatsos and Dermatas, 1998; Anitha, 2014) reported that barium compounds were found to be effective in eliminating the ettringite formation in lime treatedsoil containing sulfates. The inclusion of barium compounds in sulfate bearing soils resulted in the formation of barium sulfates, removing all the sulfate ions available in the soils. Therefore, with no available sulfate ions, the formation of swelling ettringite mineral was prevented, and no heaving was observed in pavement subgrades. Also, barium hydroxide showed impressive results compared to barium chloride for counteracting the sulfate induced damage. Hence, it was proposed to use barium hydroxide as an additive for extenuating the effect of sulfates in clay. The long-term effects on unconfined compressive strength and CBR of the sulfate bearing cement treated clays were also investigated.

2.2 Specimen Preparation and Testing

Portions of clay samples collected from the site were mixed thoroughly and stored in polythene bags under moist conditions. ASTM D 698 (2012) describes the standard procedure for light compaction tests in order to obtain the optimum moisture content and maximum dry density of clay. It was conducted by adopting both methods, viz. the wetting method and the drying method. In the wetting method, the air dried clay was compacted by adding small quantities of water at each stage of compaction. While in the drying method, the water content was reduced and compaction was done at each stage of reduction. Figure 2 presents the compaction curves obtained for Cochin marine clay by the wetting and drying methods. The optimum moisture content and maximum dry unit weight obtained from the wetting method were 26% and 14.2 kN/m^3, respectively. In the case of the drying method, the optimum moisture content and maximum dry unit weight were 36.8% and 13.3 kN/m^3, respectively.

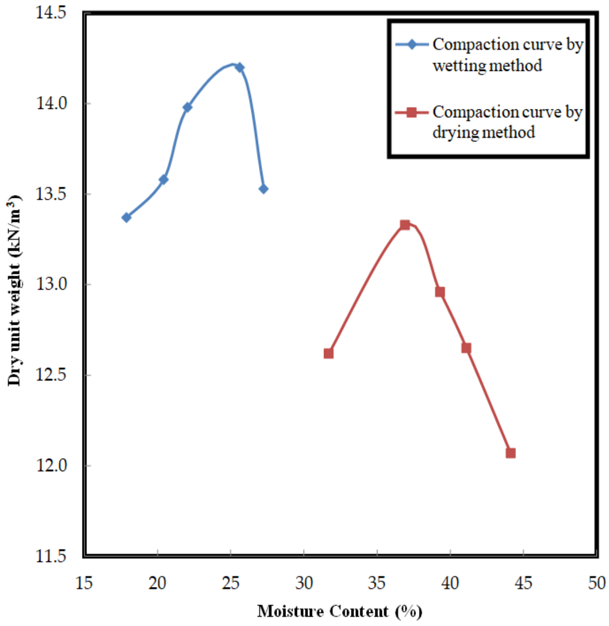

Figure 2 Compaction curve by wetting method and drying method

Specimens for Unconfined Compressive strength tests and California Bearing Ratio tests were prepared in different combinations of additive and the combinations with sample designations are as listed:

1. Clay + 6% OPC (S1)
2. Clay + 6% SRC (S2)
3. Clay + 6% OPC + 4% Na_2SO_4 (S3)
4. Clay + 6% SRC + 4% Na_2SO_4 (S4)
5. Clay + 6% OPC + 4% Na_2SO_4 + $Ba(OH)_2$ (S5)

The amount of barium hydroxide required to mitigate the sodium sulfate in the sample can be calculated by balancing the chemical equation given below.

$$Ba(OH)_2 + Na_2SO_4 \rightarrow BaSO_4 + 2NaOH \tag{1}$$

The UCC samples were prepared at 90% of the maximum dry unit weight of the clay (by wetting method) and at a moisture content of 40% (moisture content corresponding to its saturated condition). The estimated amount of OPC, SRC, sodium sulfate and barium hydroxide as a percentage by dry weight of the soil was then added according to the categorized soil samples (S1, S2, S3, S4 and S5). The clay specimens prepared for UCS were sealed in polythene bags and kept for predetermined periods of curing (0, 7, 30, 60, 90, 180 and 270 days) under moist conditions. Unconfined compressive strength tests were carried out on soil specimens in accordance with ASTM D 2166 (2016). The CBR samples were prepared at the optimum moisture content and maximum dry density of the clay (by the wetting method). The additives were added in predetermined quantities to the soil in accordance with the classified soil samples (S1, S2, S3, S4, and S5). The specimens preparedfor CBR tests were also sealed and kept for curing (0, 7, 30, 90 and 270 days) under moist conditions. ASTM D 1883 (2016) was followed to perform CBR tests on prepared soil specimens. Liquid limit and free swell index tests were also conducted for all treated soil specimens at 0, 7, 30, 60 and 90 days of curing periods. The method proposed by Sridharan and Rao, 1985 was used for the determination of free swell index. For this test, a moist sample weighing 10 g of equivalent dry weight was placed in a graduated 100 ml cylinder with 40 ml of distilled water. The suspensions were completely combined with a glass rod after being continuously agitated and increased to the 100 ml level with the addition of distilled water. The soil was allowed to settle. The free swell index in cc/g represents the sediment volume per gramme of dry soil. Tables 4 & 5 summarizes the different types of treated soil samples and the experimental tests that were conducted in this study.

Table 4 Operation details of the experiments conducted in the study

Tests	Condition	Operation	No. of repetitions
Unconfined Compression Test	Air dried	ASTM Standards	3
CBR			2
Liquid limit			2
Free Swell Index		Method proposed by Sridharan and Rao, 1985	2

Table 5 Experimental program for study of properties of treated soil samples

Sample Designation	Sample description	Test conducted	Curing period
S1	Clay + 6% OPC	Liquid limit, Free swell index	0 day, 7 days, 30 days, 60 days, 90 days
S2	Clay + 6% SRC		
S3	Clay + 6% OPC + 4% Na_2SO_4		
S4	Clay + 6% SRC + 4% Na_2SO_4		
S5	Clay + 6% OPC + 4% Na_2SO_4 + $Ba(OH)_2$		
S1	Clay + 6% OPC	UCC, CBR	0 day, 7 days, 30 days, 60 days, 90 days, 180 days, 270 days
S2	Clay + 6% SRC		
S3	Clay + 6% OPC + 4% Na_2SO_4		
S4	Clay + 6% SRC + 4% Na_2SO_4		
S5	Clay + 6% OPC + 4% Na_2SO_4 + $Ba(OH)_2$		

3. RESULTS AND DISCUSSIONS

3.1 Effect of Drying on the Strength of Marine Clays

The phenomenal improvement in the index properties of air-dried clays compared to moist clays is depicted in Table 1. The liquid limit of marine clay was reduced by 38% on air drying and 59% on oven drying, as seen in Table 1. From the table, a considerable influence on Atterberg limits and grain size distribution can be noticed. The major cause of this peculiar change can be attributed to the aggregation of particles upon drying (Jose *et al.*, 1987). It resulted in a reduction in the percentage of clay content from 40% to 29% and an increment in the proportion of silt and sand sizes. The sand content increased from 21% to 28% and the silt percentage from 39% to 43%. The liquid limit was lowered to 75% upon drying, while the plastic limit was lowered to 31%. As a result, the plasticity index also dropped, reaching a value of 44%. The values of the shrinkage limit were found to have increased from 23% to 26%.

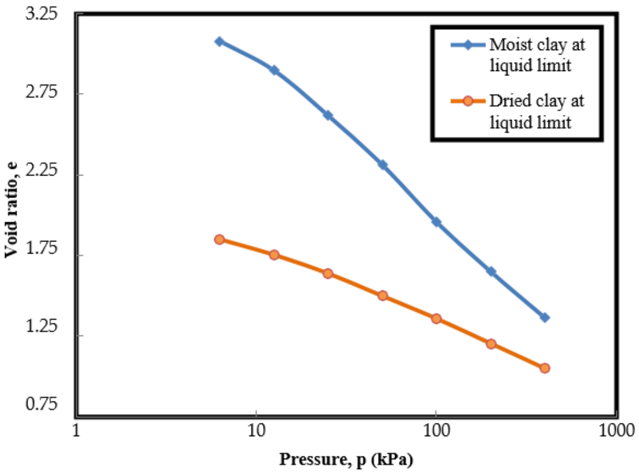

Figure 3 e-log p curve of moist clay and air dried clay at liquid limit

Consolidation tests were conducted to obtain the compressibility behavior of both moist and air-dried clays as per the standard procedure mentioned in ASTM D (2004) 2435. The test was done on both moist and air-dried clay samples at their liquid limits and at their natural water content. In the case of the air-dried sample, water was added to attain its saturated state, and then it was kept for consolidation. The e-log p curves of both air-dried and moist clays at their liquid limits and at their natural water content were plotted and illustrated in Figures 3 and 4. The coefficient of consolidation determined from the square root time method for both wet and dried samples is listed in Table 6. The table also provides compression index values obtained from the e-log p curves of the respective clay samples. Figures 3 and 4 clearly indicate that the compressibility characteristics have improved remarkably for air-dried samples compared to the natural clay sample.

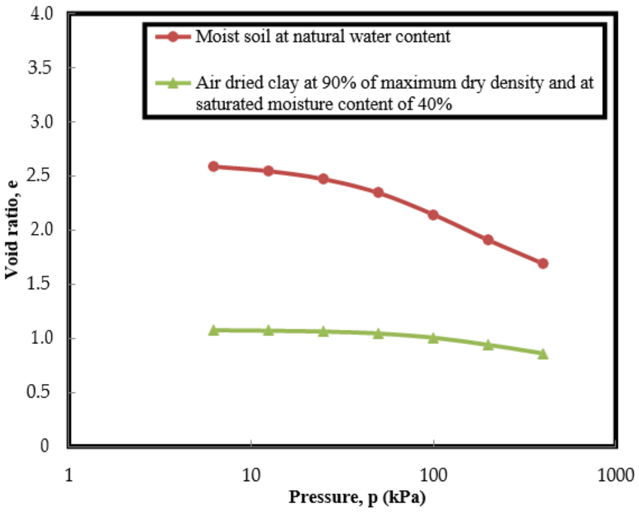

Figure 4 Comparison between e log p curves of moist clay at natural moisture content and air dried clay at 90% of maximum dry density & a saturated water content of 40%

Drying makes the clay less compressible than the moist clay sample. This result throws light on the significance of drying in improving compressibility characteristics. Unconfined compressive strength tests on air-dried clays produced a value of 50 kPa, whereas moist clay at natural moisture content yielded only a compressive strength of 13 kPa. It implies that drying also improves the strength remarkably.

Table 6 Compressibility characteristics of both moist and air-dried clay sample

Type of Sample	Compression index, Cc	Coefficient of consolidation, c_v(x 10^{-4})		
		Pressure range		
		50-100kPa	100-200kPa	200-400kPa
Moist clay at natural water content	0.72	1.09	1.76	1.51
Moist clay at liquid limit	1.02	1.17	0.97	1.42
Air dried clay at liquid limit	0.51	0.87	1.33	1.46
Air dried clay at 90% of maximum dry density and a saturated moisture content of 40%	0.26	2.42	2.67	2.74

3.2 Effect of Curing Period on Unconfined Compressive Strength of Cement Treated Clay

The assessment was made based on a set of unconfined compression tests conducted on artificially prepared soil specimens at 0, 7, 30, 60, 90, 180 and 270 days curing periods as per the method in ASTM D (2016) 2166. A suitable longer curing period was preferred in order to understand the effect of sulfates on cement treated clays. Marine clay treated with cement (S1) was chosen as the control sample to establish baseline data for comparison with cement treated clay. The incorporation of barium hydroxide is a proven mitigation method for sulfate induced heave as it leaves behind an insoluble residue of barium sulfate (Raja, 1990; Ferris *et al.*, 1991; Tsatsos and Dermatas, 1998; Anitha, 2014).

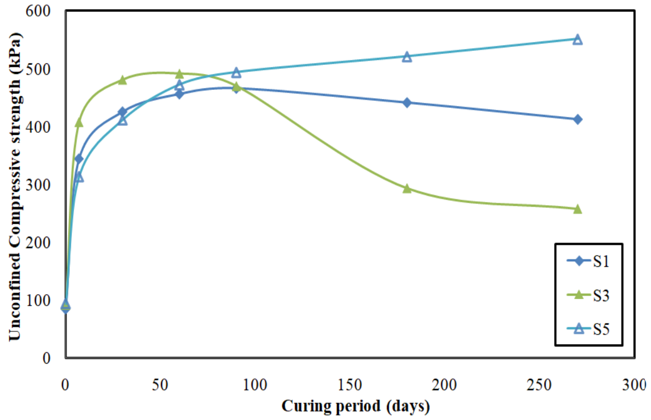

Figure 5 Variation in strength of samples treated with OPC and additives on curing

Figure 5 compares the strength behavior of S1, S3, and S5 as the curing time increases. It was observed that clay samples prepared with OPC and sodium sulfate (S3) exhibited greater strength during the initial period, but then this strength eventually declined as a result of the formation of ettringite. The decrease in strength of S3 occurred after 60 days of curing, and as the curing period increased to 270 days, the strength reduced from 491 kPa to 257 kPa, showing a drastic drop of 90%. The adverse effect of higher sulfate content was clearly evident from the reduction in strength of S3 by 60%, when compared to S1, at the end of 270 days. S1 initially exhibited an ascending trend as well, but after 90 days of curing, the strength declined. The primary cause behind this peculiar decrease in strength was the presence of 0.53% natural sulfate. The figure also illustrates that S5 (Clay + 6% OPC + 4% Na_2SO_4 + $Ba(OH)_2$) exhibited a steady increase in strength up to 551 kPa over time, despite the fact that the rate of strength improvement during the early period was gradual.

Figure 6 illustrates the impact of the curing period on the strength behavior of S1, S2, and S4. Even though S4 (Clay + 6% SRC + 4% Na_2SO_4) exhibited a steady increase in strength up to 90 days of curing, it gradually showed a considerable reduction of 34%, from its highest strength value. A much similar trend was observed in the strength values for S2 (Clay + 6% SRC) as well, but there was only a slight decline of 9% at the end of the curing period. When compared to S1 (Clay + 6% OPC), S4 displayed a strength drop of 37% after 270 days of curing, whereas only a 6% reduction in strength was observed for S2.

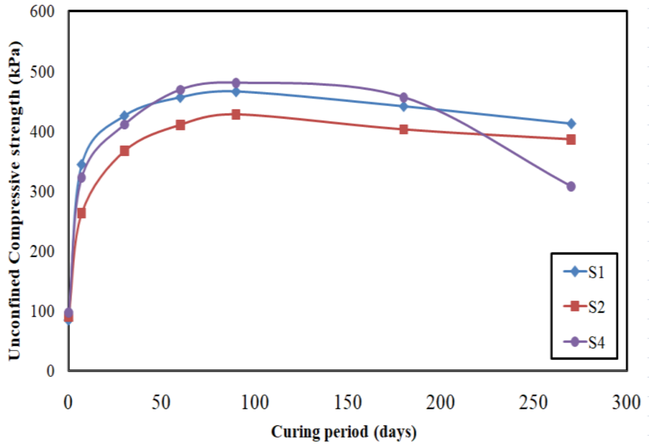

Figure 6 Variation in strength of sulfate containing samples treated with SRC and OPC on curing

These results indicate that sulphate resisting cement could be capable of arresting the adverse effects of cement treated clays bearing low sulfate content. The ettringite is formed by the reactions between calcium, sulfate, alumina and hydroxide in the presence of water. Owing to the low alumina content in sulphate resisting cement, the formation of ettringite can be prevented, and this can throw light on the effectiveness of sulphate resisting cement for stabilizing sulfate bearing clays (Puppala *et al.*, [2003, 2004, 2014]). However, if alumina is supplied by clay minerals, the cement treated clay will be susceptible to sulfate induced heave and this effect cannot be prevented by sulphate resisting cement (Sherwood, 1962).

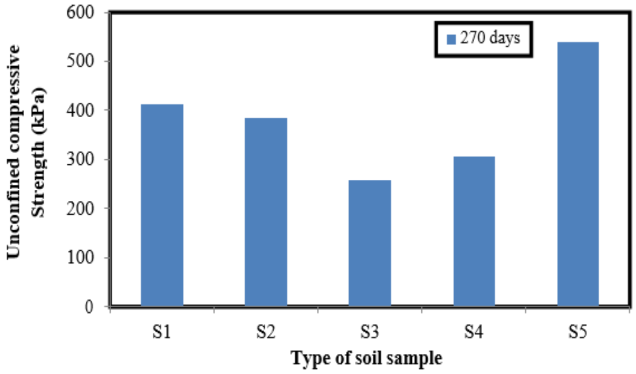

Figure 7 UCS of treated clay samples at 270 days of curing

Figure 7 brings out the drastic reduction in strength gain for S3 (Clay + 6% OPC + 4% Na_2SO_4) compared to all other soil specimens, emphasizing the need for mitigation measures. Given that S4 also displayed a sizable decrease in strength gain, this illustrates the fact that sulphate resisting cement cannot completely counteract sulfate induced heave when there is a considerable amount of sulfates in the soil. The figure also shows that S5 attains the greatest strength compared to all other treated clay specimens at the end of 270 days of curing. The increase in strength may be due to the conversion of sodium sulfate into barium sulfate, and thus proving the potential of barium hydroxide in arresting the effect of sulfates on cement treated marine clays.

3.3 Effect of Curing Period on CBR Values of Cement Treated Clay

A series of CBR tests were performed on all the prepared combinations of soil specimens at predetermined curing periods such as, 0, 7, 30, 90 and 270 days as per the procedure laid down in ASTM D (2016) 1883. The CBR values of S1, S3 and S5 with respect to the increasing curing period are presented in Figure 8.

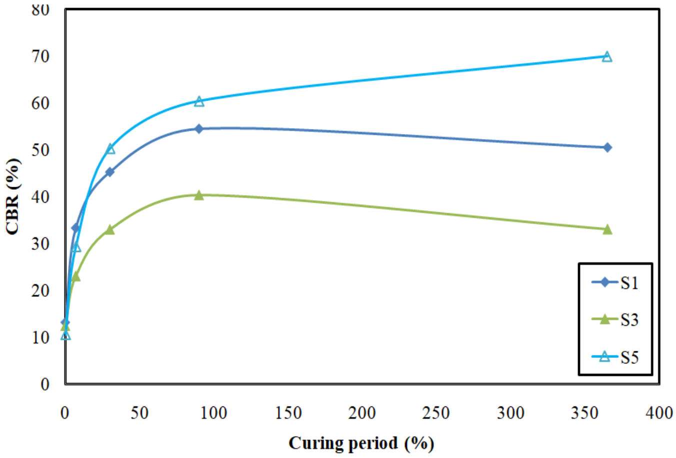

Figure 8 Variation in CBR values of samples treated with OPC and additives on curing

It depicts that S5 (Clay + 6% OPC + 4% Na_2SO_4 + $Ba(OH)_2$) has attained the highest CBR value of 70% at the end of curing, despite having a low initial strength. It was also found that there was a notable reduction in the CBR values of S3 (Clay + 6% OPC + 4% Na_2SO_4) and S1 (Clay + 6% OPC) after 90 days of curing. The reduction in strength was more pronounced in soil S3 than in soil S1. This can be attributed to the higher sulfate content in S3 that led to the formation of the swelling clay minerals. A considerable decline of 35% in CBR value was observed for S3 at the end of 270 days of curing when compared to S1.

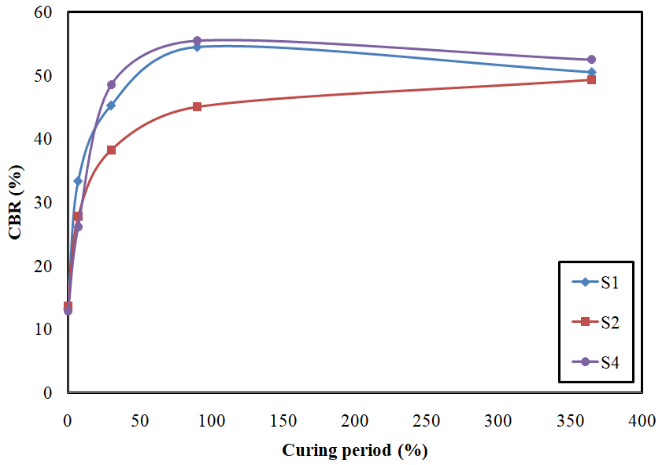

Figure 9 Variation in CBR values of samples treated with SRC and additives on curing

Figure 9 presents the CBR values of S1, S2 and S4 with an increase in curing period. S2 (Clay + 6% SRC) seemed to display an increasing trend in strength gain when compared to S1 and S4 (Clay + 6% OPC + 4% Na_2SO_4). It signifies that the effect of sulfates can be counteracted by sulphate resisting cement when the clays have low sulfate content. However, sulphate resisting cement does not retard the formation of ettringite when there is a higher percentage of sulfate present in clays. The decrease in strength of soil S4 is in agreement with this finding. At the same time, S4 sustained a good CBR value of 53% after 270 days of curing, as illustrated in Figure 10. Further studies on prolonged curing periods are strongly advised for clays treated with sulphate resisting cement.

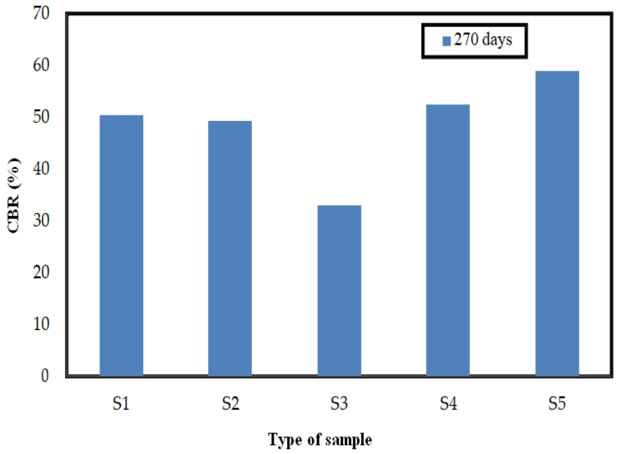

Figure 10 CBR of treated clay samples at 270 days of curing

3.4 Effect of Curing Period on Liquid Limit of Cement Treated Clay

The liquid limit was determined for all the treated clay specimens at different curing periods of 0, 7, 30, 60 and 90 days as per the guidelines in ASTM (2010) D 4318.

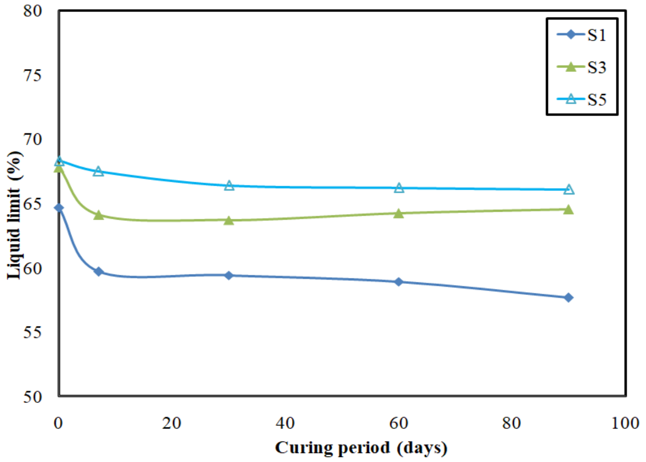

Figure 11 Variation of liquid limit of sulfate conta0ining clay specimens with OPC and additives on curing

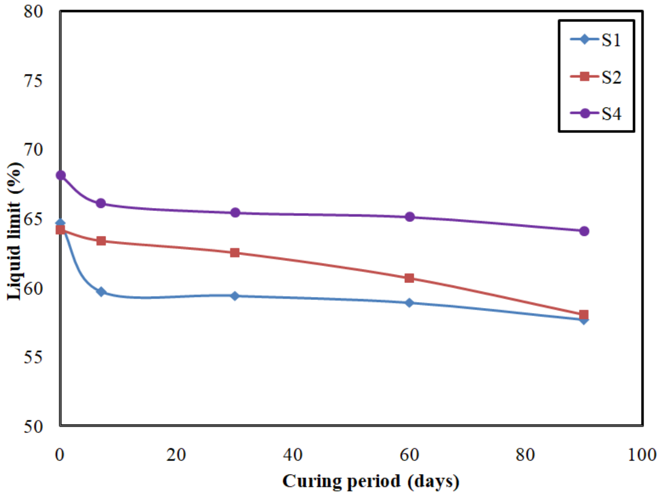

Figure 12 Variation of liquid limit of sulfate containing clay specimens treated with SRC on curing

The liquid limit values of S1, S3 and S5 with respect to increasing curing periods are compared, as shown in Figure 11. All of the treated specimens showed a declining trend as the curing time increased, with the exception of S3, which displayed a minor increase in the liquid limit towards the end of 3 months. The observed decrease in liquid limit values can be attributed to cationic exchange reactions (Leroueil and Le Bihan, 1996). Figure 12 illustrates the decreasing trend of liquid limit values for S1, S2 and S4 with an increase in curing period. The liquid limit of all the treated clay samples has also decreased significantly compared to the untreated clay samples.

3.5 Effect of Curing Period on Free Swell Index of Cement Treated Clay

The expansive nature and potential of clays are usually determined with free swell tests. High swelling in clays can pose problems for pavements. Therefore, it is important to conduct this test on all treated clay samples. This test was performed at specific curing periods, such as 0, 7, 30, 60, and 90 days, using the method proposed by Sridharan and Rao, 1985. The free swell index of untreated clay samples was also determined, and the value was obtained as 1.24 cm^3/gm.

Figure 13 presents the typical case values for the free swell index of all treated clay samples. It indicates that the free swell index value of all the treated clay specimens has increased beyond 1.24 cm^3/gm, which corresponds to the free swell index value of the untreated clay sample. At the end of the curing period, the free swelling of S3 increased to 86% when compared to S1, whereas S4 and S5 produced swelling that was nearly identical.

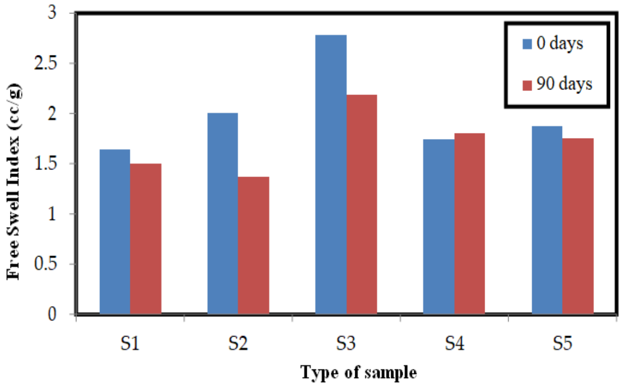

Figure 13 Variation in free swell index values of treated clay samples at 0th and 90th day

From the variation of free swell index values of treated clay samples at 90 days of curing, it was observed that the free swell index of S3 remains the highest when compared to all other treated samples. This can be attributed to the nucleation of the expansive mineral, ettringite, in the treated sample. The result also shows that the samples incorporated with barium hydroxide and sulphate resisting cement exhibit comparable free swell index values at both curing periods. All throughout the curing period, the clay specimens treated with an increased percentage of sulfate peaked in free swell index compared to other treated and untreated samples. Therefore, it can be inferred that the increase in concentration of sulfates can produce notable swelling in clays, and it becomes a very important factor to be considered when it comes to the construction of pavements over clayey subgrades.

3.6 Scanning Electron Microscope Analysis

To prove the visible presence of significant quantities of needle-shaped crystals, SEM experiments were carried out.

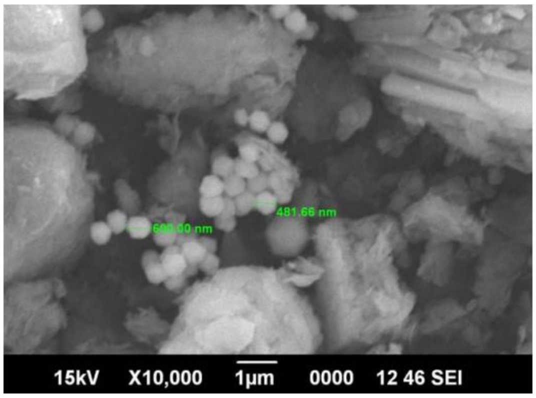

Figure 14 SEM images of ettringite in OPC treated clay (4% sulphate)

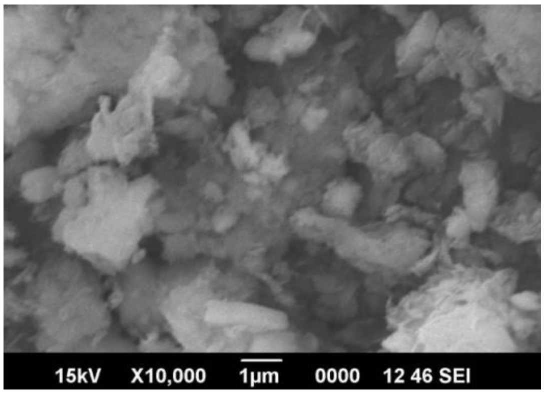

Figure 15 SEM images of OPC treated clay with 4% sulphate & $Ba(OH)_2$

Figure 14 indicates SEM images of ettringite in OPC treated clay bearing 4% sulfate after curing for 270 days. The presence of the well-known ettringite needle was observed in the SEM image. Shapeless gel particulates enveloped the ~0.6 micron-wide particles, which were not present in the sample treated with barium hydroxide (Figure 15).

4. CONCLUSIONS

The influence of sulfates in cement treated marine clays and the measures adopted to inhibit its effect on soils were studied through unconfined compressive strength and CBR tests. The following conclusions were drawn based on the experimental studies:

- Drying enhanced the index properties such as liquid limit, plastic limit and shrinkage limit of marine clays. It reduced the liquid limit to 75% and the plastic limit to 31%. As a result, the plasticity index also improved, reaching a value of 44%. An increase from 23% to 26% was observed in the values of the shrinkage limit. Additionally, the strength and compressibility characteristics were improved. Unconfined compression strength of clay specimen increased from 13 kPa to 50 kPa upon drying.
- Prolonged curing periods significantly affected the unconfined compressive strength and CBR values of cement treated sulfate bearing marine clays. As the curing period progressed from 0 to 270 days, the effect of sulfate was predominant on artificially prepared samples of marine clay treated with sodium sulfate and OPC. The unconfined compressive strength of the treated clay specimen declined after 60 days of curing. After cement stabilization, clays bearing 4% sulfate content showed an adverse strength loss of about 90% towards the end of curing period. The soil samples treated with OPC and sodium sulfate also yielded lowest CBR value at the end of 270 days of curing. A considerable decline of 35% was observed for clay specimens prepared artificially with 4% sulfate & 6% OPC.
- In the case of sulfate bearing clayey soil treated with OPC alone, both unconfined compressive strength and CBR value decreased after 90 days of curing. After 90 days of curing, the cement-treated clays that naturally contain 0.5% sulphate had a substantially lower strength loss of 11% and a CBR value decline of 9%.
- Incorporation of sulphate resisting cement (SRC) in clays provided a steady increase in strength even though the early developed strength was low compared to OPC treated samples. After being treated with cement, the clays containing 0.5% sulphate only produced a reduction of 9%, while the CBR values consistently increased by roughly 10% at 270 days of curing. This suggests that sulphate resisting cement might be capable of preventing the formation of ettringite in low sulfate bearing clays treated with cement, at low water content.
- In artificially prepared clay specimens treated with sodium sulfate and sulphate resisting cement, there was reduction in the unconfined compressive strength after 180 days of curing. Despite showing a consistent increase in strength up to 90 days of curing, S4 gradually displayed a significant drop of 34% from the peak strength value it attained. Also, a slight decrease of almost 10% was observed in CBR value of the soil sample stabilized with sulphate resisting cement at the end of 270 days of curing, even though the rate of strength reduction was low as compared to artificially prepared clay treated with OPC. Therefore, it implies that treatment with sulphate resisting cement is not advisable for high sulfate containing clays treated with cement.
- In sulfate bearing clays stabilized with cement, treatment of barium hydroxide is a promising method for mitigating the sulfate induced heave. As the curing period increased from 0 to 270 days, a significant increase in strength gains up to 551 kPa was observed for the treated clay samples. It also yielded

the highest CBR value of 70% compared to all other treated clay samples. This can be attributed to the formation of barium sulfate and thereby arresting the nucleation of ettringite. Therefore, this method can be adopted for sulfatic clays containing high sulfate content.

- For all the additives adopted, the liquid limit of treated clayey soil seemed to decrease compared to the liquid limit of untreated clays. All treated clayey samples showed a declining trend over the course of the curing period.
- Free swell volume increased with the addition of both types of cements (OPC and SRC) and additives (sodium sulfate and barium hydroxide). The free swell index appeared to decrease with an increase in curing time. The free swelling of clay specimens treated with OPC and sodium sulfate increased to 86%, which corresponded to the highest free swell index value over the curing period, indicating that an increased level of sulfates in soils can cause considerable swelling in clays.

From the experimental results, it can be concluded that the prolonged curing periods considerably affects the unconfined compressive strength and CBR value of cement treated marine clays containing sulfates. Moreover, the amount of sulfate present in clayey soils dictates the effect of the swelling clay mineral and the type of pre-treatmentmethods to be adopted. The detailed study with incorporation of sulphate resisting cement and barium hydroxide at longer curing periods of 270 days has provided an indication of the effectiveness of methods advocated. Sulphate resisting cement is sufficient in mitigating the adverse effects of low percentages of sulfates present in clayey soils. But, in the case of high sulphatic soils treated with cement (OPC), barium hydroxide is found to be very effective in counteracting the formation of swelling clay mineral ettringite. Therefore, incorporation of barium compounds is a promising method for mitigating the 'Sulfate induced heave' in cements treated clays.

In order to protect pavements from sulphate damage, this approach can be used in sulfate-bearing subgrade soils after being treated with calcium based stabilizers.

Since the research was limited to only 270 days, as a recommended future study, the long-term performance of marine clays containing low sulfate concentration, when stabilized by sulphate resisting cement, should be investigated. The influence of different percentages of water should also be researched because the amount of water content also becomes a crucial factor that controls the formation of ettringite.

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