



Synthesis of geopolymer & its efficacy as a stabilizer of expansive soil

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Abstract: Expansive soil is one among the problematic soil that has high potential of swelling and shrinkage. Also it has considerable strength in dry state, but strength goes on reducing on absorption of water. The most important aspect for construction purpose is soil stabilization which is widely used in foundation and road pavement construction. In the present study expansive soil collected from Zari (Navsari, Gujarat) which has FSI 76 % and belongs to CH classification as per IS:1498-1970 was stabilized using three different types of geopolymers. Geopolymers are alumino-silicate binders that have received more attention as a sustainable alternative to conventional chemical additives. Geopolymers have high compressive strengths and can be synthesized at room temperatures from aqueous solutions by utilizing waste materials which have high alumina and silica content. Three geopolymer used in studies are namely Metakaolin based, Crushed waste ceramic based and Sodium bentonite based. These materials are activated by alkali activator (NaOH, KOH, Na₂SiO₃) for synthesis of geopolymer in laboratory. 8%, 12% & 15% geopolymer added by weight of dry soil for treatment of expansive soil. The parameters such as unconfined compressive strength, swelling & shrinkage property of three geopolymer treated expansive soil was studied under controlled condition after stabilization at various curing period based on soil-geopolymer-water interaction. The test result indicates that geopolymers have significantly improved strength and volume change properties of expansive soil. Both pre and post chemical treatment was analyzed using EDAX. Increased in geopolymer quantity and curing period have resulted in further property enhancement. Swell and shrink studies also indicated reduction in the strain when compared to natural soil. Geopolymers are apparently found to be quite efficient in stabilizing expansive soil.

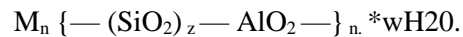
Key Words: Geopolymer, Alkaline activator, UCS, black cotton soil, Swelling-shrinkage, Stabilization.

1. Introduction

In India, about 20% of the total land area is covered by expansive soil and is also referred as a black cotton soil [1]. Expansive soil is a problematic soil that has a high potential for shrinking or swelling due to changes in water content. The main problem that arises with regard to expansive soils is that deformations are considerably greater than elastic and plastic deformations [2]. Differential settlement and movement is usually in an uneven pattern and of such a magnitude to cause extensive damage to the structures resting on them. Over the last several decades, several types of stabilization techniques – including physical, mechanical, and chemical methods have been developed to treat expansive soils [3]. Of these methods, chemical stabilization, particularly using calcium-based stabilizers, proved to be more reliable and capable of stabilizing soils effectively [4]. Generally, cement and lime are common materials used to improve these soils by reducing its plasticity, swelling characteristics, and increasing their strength. However, the production process of these traditional stabilizers is

energy intensive and it also serves as a major source of greenhouse gas emissions, leading to severe problems like global warming [5]. Therefore, there is a need for such type of soil stabilizers which are sustainable and durable as well as capable to significantly improving engineering properties of expansive soils. Geopolymer are a new generation alternative binding material for conventional cement [6]. There are so much information is available on synthesis of geopolymer for concrete, ceramic & resin industry but very little study have investigated their viability as a soil stabilizer.

Geopolymers can be synthesized from industrial byproducts and are known to have high compressive strength, low shrinkage, as well as heat and fire-resistant properties. Geopolymers have a lower carbon footprint than lime and OPC [6] and are therefore being considered as an alternative to conventional soil stabilizers. Geopolymers can be synthesized from raw materials which have high Al-Si content; these raw materials are activated by alkaline activators [6]. Alumino-silicate rich materials are industrial waste such as slag; fly ash; red mud; ceramic dust, quarry dust, sodium bentonite, and metakaolin etc. One common formula for all types of geopolymers is given as follows [7,8]



Where, M is an alkali metal cation such as Na, K or Ca; 'n' is degree of polymerization; 'z' is Si/Al ratio (generally 1,2,3) & 'w' is molar water content [8]. For the synthesis of geopolymer 4 main component required alumino-silicate precursor, alkaline activator, additional silica (if required) & water [7]. Molarities of AAS used for synthesis of GP varying from 8M to 18M [8]. Steps involved for formation of geopolymer (figure 1.0) [7].

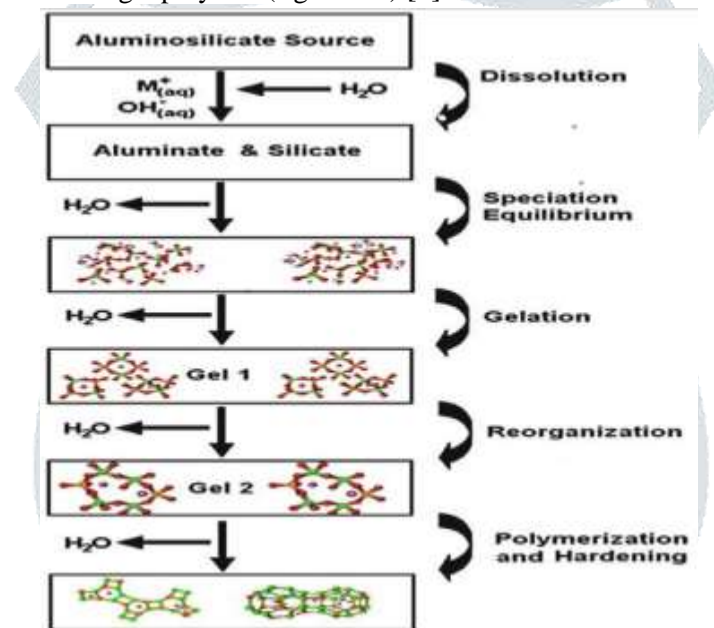


Figure: 1 Synthesis of geopolymer process

Figure 1 shows that five stages of geopolymerization. The dissolution process starts when an alumino-silicate resource is mixed with AAS, and a breakdown of Si–O–Si and Al–O–Si covalent bonds occurs. Accumulations of destroyed product occur, which interact among them to form a coagulated structure, leading in a third phase to the generation of a condensed structure and crystallization. From the second stage there is no need for water for the process, but water is released from the gel porous during the process. Rearrangement and reorganization of the gel system continues. Gelation occurs. Further curing result in hardening of geopolymer gel [7]. In present paper discusses the synthesis of geopolymer and swell-strength properties of three different geopolymer-treated expansive soils, and utilization of locally available material for synthesis of geopolymer. The main objective of the research is to stabilize expansive soil using geopolymers (CWC-based, Metakaolin based & Sodium bentonite based). Potassium hydroxide (KOH), Sodium silicate (Na₂SiO₃) & Sodium hydroxide (NaOH) with 12M concentration is used as an alkaline activator.

2. Materials and Method

2.1 Expansive soil

Expansive soil was collected from natural deposit near, Zari, Gujarat India. The soil was collected 0.6 m depth by undisturbed sampling. The geotechnical test (Such as sieve analysis, specific gravity, Atterberg limit test, hydrometer,

standard proctor test, free swell index, swelling pressure test) has been carried out as per the reported protocol of Indian Standard Code. The geotechnical properties of a soil sample are presented in Table 1.

2.2 Raw material selection

Any type of raw material which has high alumina & silica content can be used as GP resources. In this paper, total five GP resources were used and Energy-dispersive X-ray spectroscopy (EDAX) tests were also performed on these raw materials to find about chemical component present (Mainly Al, Si, Fe) content. Crushed waste ceramic powder was collected from ceramic tiles factory (Navsari). Quarry dust (<75) was collected from rock crusher factory (Chikhli), Metakaolin, Sodium bentonite & Micro-silica were collected from traders (India-mart). All these materials were selected based on literature review of standard research paper.

2.3 Alkaline activator

Sodium hydroxide (NaOH) & Potassium hydroxide (KOH) with 99% purity, Sodium silicate (Na_2SiO_3) with 90% purity were collected from SDFCL (Mumbai) & all three chemicals were analytical grade. These three alkaline activators are most commonly used for the synthesis of geopolymer and selected based on literature review and some standard book of geopolymer.

Table: 1
Properties

TEST/ PERAMETER	VALUE	IS CODE
Hydrometer	Clay – 51 %, Silt - 23%	IS 2720 PART IV- 1985
Specific gravity (G)	2.66	IS: 2720 – Part III, 1980
Liquid limit % (WL)	69.10%	IS: 2720 – Part V, 1985
Plastic limit % (WP)	35.26%	IS: 2720 – Part V, 1985
Shrinkage limit % (WS)	12.05%	IS: 2720 – Part VI, 1972
Free swell index % FSI	66%	IS : 2720 Part – XL , 1977
Optimum moisture content % OMC	28.86%	IS: 2720 – Part VII, 1980
Maximum dry density g/cc MDD	1.49 g/cc	IS: 2720 – Part VII, 1980
Soil type	CH	IS 1498 – 1970

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3. Methodology & Sample preparation

3.1 Basic Soil Characterization Tests

The results of index properties & engineering properties of natural soil are summarized in table 1. The grain size distribution tests were conducted on natural soil as per the IS code 2720 part-4 to classify the soil according to USCS (Unified Soil Classification System). The natural soil was classified as highly cohesive (CH) clay. Atterberg limits were determined as per IS code. Density and moisture content relationship of natural soils were determined by standard proctor test as per IS code guideline. For identification of expansive nature of soil free swell index test also performed on natural soil which give the value of FSI was 66 %. From the free swell index (FSI) soil identified as expansive in nature.

3.2 Geopolymer synthesis & treatment

The geopolymers used in the study were metakaolin (MK) based, crushed waste ceramic (CWC) based & sodium bentonite (SB) based. Raw material used with some standard quantity given below. Solid to Liquid ratio 2:3 remain constant for all three geopolymer formations. The concentration of alkaline activator solution (AAS) was fixed 12M (molar). Quantity of material used in formation of geopolymer was:

Metakaolin based: For the synthesis of geopolymer 40% metakaolin mixed with 60% KOH (12M) solution. For the homogeneity geopolymer, mixed it with magnetic stirrer with 300 rpm. The percentage of geopolymer was based on past research work.

Crushed waste ceramic based: For the formation of AAS, Na_2SiO_3 & NaOH ratio 1.5:1 was used. Crushed ceramic 17% + Quarry dust 17% + Micro-silica 6% + 60% AAS (12M) were mixed in mixture at 300 rpm.

Sodium bentonite based: For the formation of AAS, Na_2SiO_3 & NaOH ratio 1.5:1 was used. Sodium bentonite 32% + Micro-silica 8% + AAS (12M) 60% + 10% water were mixed in mixture at 300 rpm. Synthesis of the geopolymer is shown in figure 2.

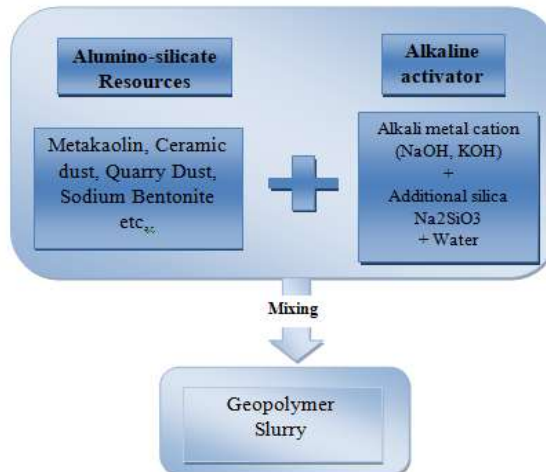


Figure: 2 Synthesis of geopolymer

When alumino-silicate resources are activated by a strong alkaline activator, a new gel-type material formed is termed as a geopolymer. In this study, three geopolymers were synthesized & compared the results of three different polymer-treated soil and find best geopolymer with optimum dosage. 8%, 12% & 15% geopolymer content by dry weight of soil was used in the study. All the above value selected from standard research paper & doctoral thesis.

3.3 Swelling-Shrinkage Test

Constant volume swelling pressure tests were conducted in accordance with IS 2720 part 41 (1977). In this method, standard proctor mould used and the sample was prepared at optimum moisture content and maximum dry density [10]. The soil specimen is kept between two porous stones saturated in boiling water, providing a filter paper between the soil specimen and the porous stone. The brass perforated plate is placed in the center of the porous stone. Soil samples are always kept submerged in water, so that soil samples will be saturated through both ways. Initially, a 5 kPa load is applied as the seating load. To keep the sample at a constant volume, the plate will be adjusted so that the dial gauge always shows the initial reading (Max. vertical swells permit up to 0.1 mm). The difference between the final dial gauge reading and the initial dial gauge reading of the proving ring gives total load in terms of division which, multiplied by the calibration factor, gives the total load. The value when divided by c/s area of soil sample gives the swell pressure. Setup for swelling pressure test is shown in figure.2. Total 10 numbers of 1-D swelling pressure tests were performed (1 natural soil, 9 GP treated soil). Tests were performed after 3 hours of GP mixed soil. Linear shrinkage bar tests were conducted in accordance with IS 2720 part 20 (1966) using soil passing through a 425 micron sieve. The soil sample was prepared at liquid limit or soil start flow itself. The shrinkage bar was clean and greased. The soil was then poured into the bar, gently tapping the bar so air bubbles could escape and making the surface flat with a spatula. Then the bar was placed for oven dried about 24 hours at 105 to 115 °C. After 24 hours, the length of the soil bar was measured to calculate the linear shrinkage percentage. Linear shrinkage bar shown in figure 3. Total 10 numbers of linear shrinkage bar tests were performed (1 natural soil, 9 GP treated soil).



Figure: 2

Figure: 3

Figure: 4

Figure: (2) Swelling Pressure Setup (3) Linear Shrinkage Bar (4) UCS sample

3.4 Unconfined Compression Test

UCS tests were carried out for the determination of the compressive strength of the sample to know the variation in soil strength before and after treatment. The compressive strength characteristics of the specimens were studied by conventional laboratory testing method based on IS Code 2720 part 10. The specimens were placed in a load frame driven at a constant strain of 1.25 mm/min until failure occurred. UCS tests were performed on natural soil sample & polymer-treated sample with diameter 34 mm and height 75 mm (L/D ration at least minimum 2) [11]. The samples were tested for three curing periods of 3, 7, and days. 3 numbers of specimens were prepared for each test, and the average result was taken. The UCS results of natural soil and three different polymer-treated soils were compared to find improvement in strength & optimum dosage of geopolymer. The UCS sample is shown in figure 4.

4 Results and Discussion

The results of basic tests on natural soil are shown in table 1. Standard proctor test, Swelling pressure test, Linear shrinkage bar test & UCS tests results are discussed in this section

4.1 Geopolymer paste

Total three geopolymers were synthesized for treatment of expansive soil. The following three figures show the 3 geopolymer pastes namely (a) Metakaolin GP (b) Crushed waste ceramic GP (C) Sodium bentonite GP



Figure 3: (a) MK paste

(b) CWC paste

(c) SB paste

4.2 Effect of geopolymer on compaction characteristics

The compaction behavior of the geopolymer-treated soils was observed to improve in terms of optimum moisture content and dry density. There was consistent increase in MDD and associated decrease in OMC with addition of geopolymer content. Among all three geopolymers, MK gave the best result compared to other GP. It was possible that the formation of new chemical compounds has occurred which has led to an increase in MDD with the addition of geopolymers [15]. This behavior may also be due to cation-exchange reaction, flocculation, poly-condensation and filling the voids within the soil matrix, thereby improve the porosity as well in addition, the flocculation and agglomeration of the clay particles due to polarization, release and exchange of ions[15]. The increased in percentage of GP content decrement of OMC was observed that may also be due to cation exchange that caused flocculation of soil particles. The results of Standard proctor test in table 2.

Test	Natural Soil	Metakaolin			CWC			Sodium bentonite		
		8%	12%	15%	8%	12%	15%	8%	12%	15%
MDD (g/cc)	1.49	1.56	1.61	1.62	1.54	1.55	1.58	1.52	1.57	1.6
OMC (%)	28.86	24.53	22.75	22.86	24.98	24.32	24.12	25.17	23.71	22.69

Table: 2 Standard proctor test result

4.3 Effect of geopolymer on shrinkage properties

The shrinkage properties of geopolymer-treated soils were improved as compared to natural soil. There was a consistent decrease in shrinkage percentage as geopolymer content increased. The reduction in shrinkage percentage of treated soil may be due to alumino-silicate gel formation or hardening of the gel [13]. Among the three GPs, MK geopolymer gave the best results compared to the others. From the experiment, it was observed that only MK-based geopolymers show very low shrinkage with zero cracks. CWC & SB-based GP shows slight improvement in shrinkage properties but is not capable of reducing soil cracks. For MK GP at 8, 12 and 15 (%) content, it decreased 54, 62 & 75 (%) respectively compared to natural soil. For CWC GP at 8, 12 and 15 (%) content, it decreased 33, 46, and 58 (%) respectively compared to natural soil. And for SB GP at 8, 12 and 15 (%) content, it decreased by 27, 37 & 46 (%) respectively compared to natural soil. The results of the linear shrinkage test are in Figure 6.

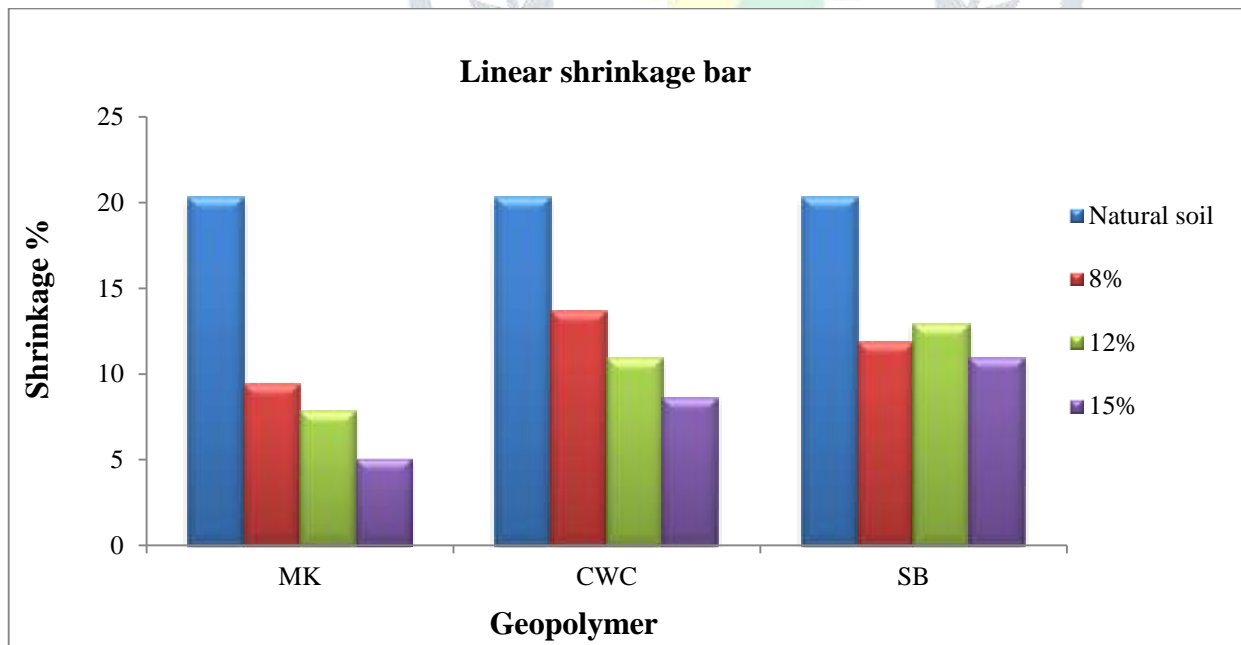


Figure 6: GP treated and untreated soils shrinkage percentage.

4.4 Effect of geopolymer on swelling pressure

The swelling pressure of GP treated soils was decreased by more than 50% compared to natural soil. All three geopolymers were effective for reducing swelling pressure. Among them, MK-based GP showed the best result, which was 90% less compared to natural soil. For MK GP at 8, 12 and 15% content, swelling pressure was reduced by about 60, 80 & 90% respectively compared to natural soil. For CWC GP at 8, 12 and 15 (%) content, it reduced 48, 55 & 75 (%) respectively compared to natural soil. And for SB GP at 8, 12 and 15% (%) content, it reduced 42, 60 & 72% respectively compared to natural soil. The results of the swelling pressure test in figure 7.

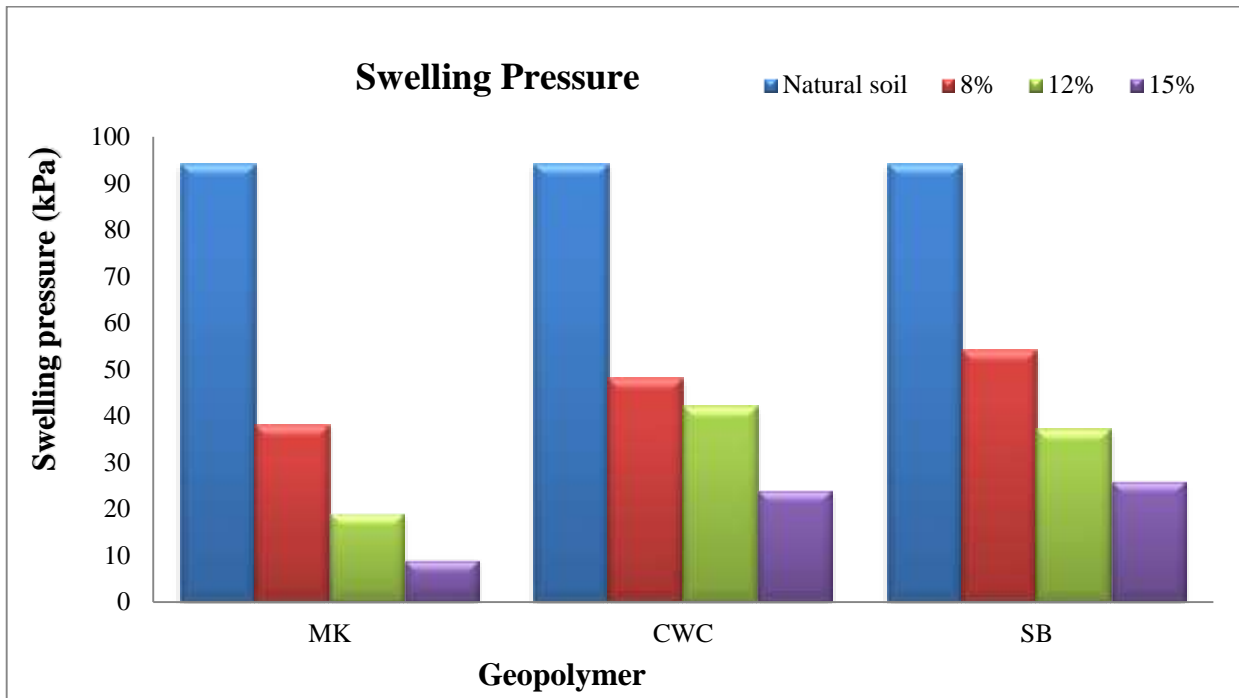


Figure: 7 swelling pressure test graph of treated & natural soil

4.5 Effect of geopolymer on UCS

From the analysis of the compressive strength tests, it was observed that compressive strength increased with an increase in curing period for each GP treated soil. The soils treated with the geopolymer maintained a consistent improvement, which showed that further addition of GP content will bring a further increase in the strength of the treated soil. CWC GP gave higher strength values compared to MK and SB GP. The addition of GP made uncemented soil samples denser by filling some voids in the soil sample structure [12]. The increase in GP concentration increased the interconnection between soil particles and produced compressible material. Therefore, the GP had a considerable effect on increasing the unconfined compressive strength of the uncemented test soils [12].

For MK based GP, it shows that as GP content & curing period increased, compressive strength increased significantly, and at 15% content & 28 day curing period, it gave the highest value of compressive strength, 3.5 times compared to natural soil. The graph plot of the MK-based GP is shown in figure 8. CWC-based GP shows comparatively less compressive strength than MK & SB. The compressive strength increased as the curing period and CWC content increased. The highest compressive strength value was 2.7 times higher than natural soil. The graph plot of the CWC-based GP is shown in figure 9. For SB-based GP, it shows that compressive strength increased with an increase in curing period but increased in GP content, compressive strength started decreasing. At 8% SB GP content and a 28 day curing period, the value of compressive strength is 3.35 times more compared to natural soil. The graph plot of the SB-based GP is shown in figure 10. UCS sample failure pattern shows in figure 11.

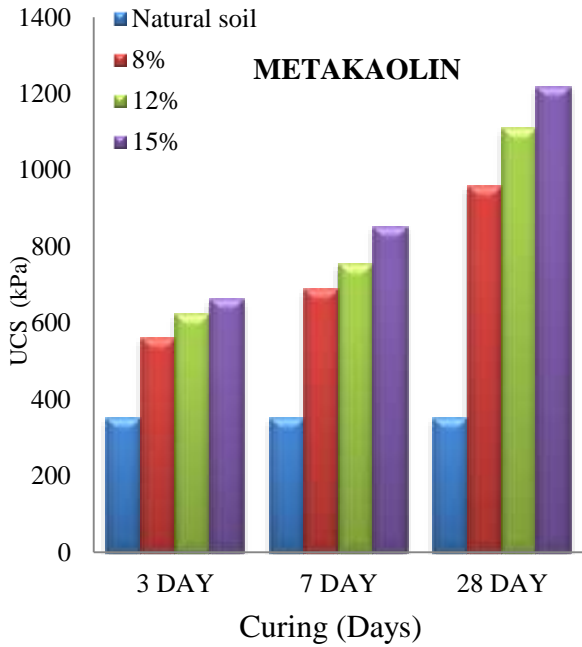


Figure: 8

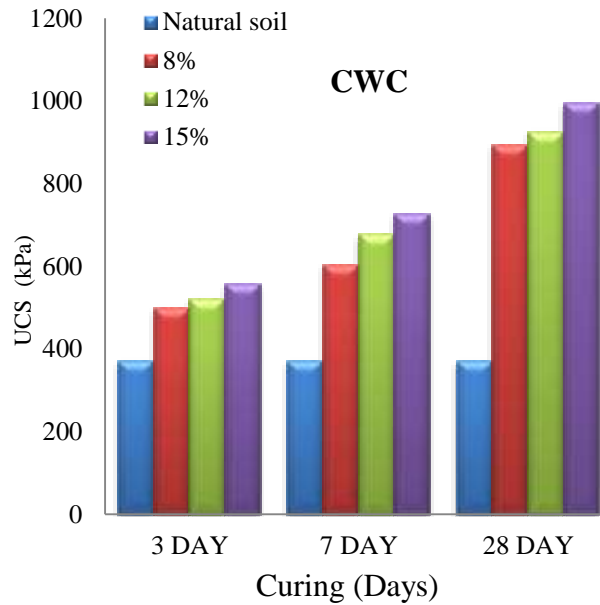


Figure: 9

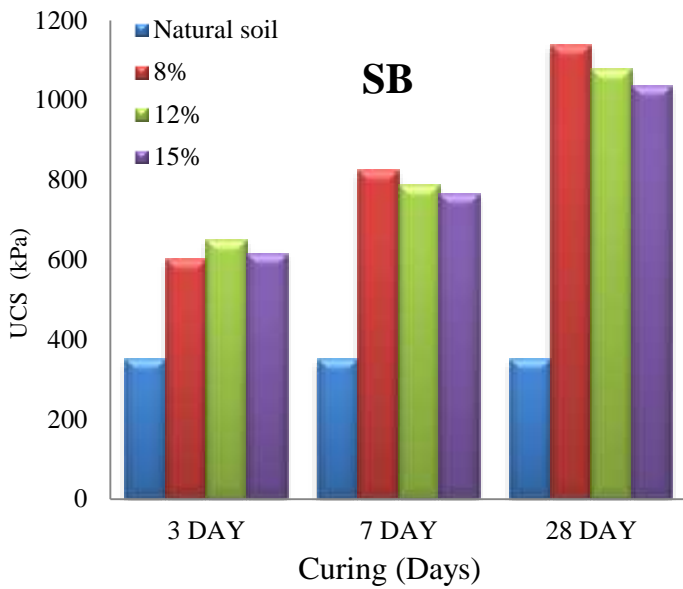


Figure: 10



Figure: 11

5 Conclusion

From the analysis of test results it is observed that all the geopolymers are proven effective in stabilization of expansive soil. Compaction characteristics such as OMC and MDD of geopolymer treated soil are significantly improved compared to natural soil. Linear shrinkage percentage of geopolymer treated soil are reduced as the quantity of geopolymer increase, MK geopolymer exhibit better performance in reducing shrinkage percentage and soil cracks where as SB and CWC found quite efficient reducing shrinkage percentage but not efficiently control the soil crack. Swelling pressure of geopolymer treated soil decrease with increasing in geopolymer quantity, among the three geopolymer MK geopolymer reduced 90% swelling pressure compared to natural soil.

UCS result it is observed that as geopolymer content increase compressive strength of treated soil increased except SB geopolymer and as the curing period of geopolymer treated soil increase significant improvement in strength is observed. From the EDAX results, it is observed that the amount of alumina (Al), Silica (Si) and Iron (Fe) increases so amount of this ion increases in soil system indicate geopolymer chain reaction present in the treated soil which leads to decrease in

swelling- shrinkage percentage and because of strong bonding with soil compressive strength of soil is increasing comparing to natural soil. When comparing all the properties of geopolymers treated soil with their different proportions, it is concluded that 15% MK geopolymer is proven efficient in increasing maximum dry density, unconfined compressive strength and reducing swelling pressure, Linear shrinkage percentage as well as efficient in reducing soil cracks (No any crack observed). So the MK geopolymer is proven efficient with 15% optimum dosage and can be used as an efficient soil stabilizer.

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