

Microstructural Characterisation and Quantitative Enhancement in Strength Properties of Stabilised Soil

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Abstract: Due to rapid rise in the social standards results in the production of by product wastes which leads to unmanageable proportions causing pollution and disposal issues. Finding a sustainable uses of the wastes products in engineering field, is one of the alternative solution for the effective management. In this work Phosphogypsum as a byproduct from fertilizer industry having more SiO₂ and CaO is used to alter the characteristics of soil, which in turn improve the strength requirements. The chemical composition of Phosphogypsum consists of majorly the constituents of SiO₂, CaO, SO₃, MgO, Fe₂O₃ and Al₂O₃ and the presence of these compounds imparts the strength on soil samples by its addition. Microstructural analyses were studied by Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS) on untreated soil and Phosphogypsum to identify the possibility of using it, to modify the characteristics of soil. The improvement in strength properties were determined by the Unconfined Compressive Strength (UCS) tests on clayey soils in untreated and stabilized condition with varying content of Phosphogypsum as 2, 4, 6% at 7,14 and 28 days of the curing period. UCS results showed an increase in strength values in the order of 1.16 and 1.75 times at the 28th days of curing by the addition of 6% of PG in soil samples.

As a result of treatment, the minerals which confirm the pozzalonic reactions were examined by X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) spectrometer. Elements like Si, Al, Ca, Mg, and S in treated and untreated soils were analyzed to identify the variations in strength characteristics with the influence of the Phosphogypsum. In addition, the Free swell properties which are important in construction work related to the surface characteristics of soil were also carried out on treated and untreated soil samples, indicates a reduction of values of 50% and 60% at the 28th day. This reduction in swell value reduce the vulnerability of construction activity in the surface of soil upto 2 to 3m or upto the influence depth from the ground surface. This method of stabilization allows an improvement in characteristics of clayey soil by making it suitable for construction activities. It has been observed that the effect of Phosphogypsum in the soil as stabilizer results in increase the strength behavior by the treatment and makes a cost effective resource in construction activities.

Keywords: Phosphogypsum, SEM, XRD, XRF, Unconfined compressive Strength, Free Swell.

1. INTRODUCTION

The available sites with weak soft clays create increasing problems related to construction activities, because of that ground improvement techniques becomes necessary to strengthen the soil. Addition of any cementing materials can improves the resistance to compressible nature of soils and which can improve the strength characteristics. Many successful works has carried out using the materials such as fly-ash [1], lime [2, 3], cement

[4, 5] etc to change the basic volume changing characteristics of soil. The use of available resources for ground improvement, results a sustainable solution in engineering field to meet the engineering requirements in soils. Among that the usage of wastes products in soil stabilization as stabilizer proved innovative technique. There are several studied have been done in the utilization of different types of waste [6, 7, 8] and recycled materials [9, 10] as a pozzolonic agent to improving the strength characteristics of soil. Usually stabilizers rich in calcium and aluminium content have high impact on weak soils improvement. This paper showed the fine-structure of the soil and stabilizer providing an estimate of the cementitious compounds formed during hydration process and distinguishing the mineral components as reaction products by X- ray diffraction and X-Ray Fluorescence techniques. In this way an observation were also conducted by microscopic images that supports the precise changes of the fabric of stabilized soil by SEM analysis with EDS.

2. MATERIALS AND CHARACTERISTICS

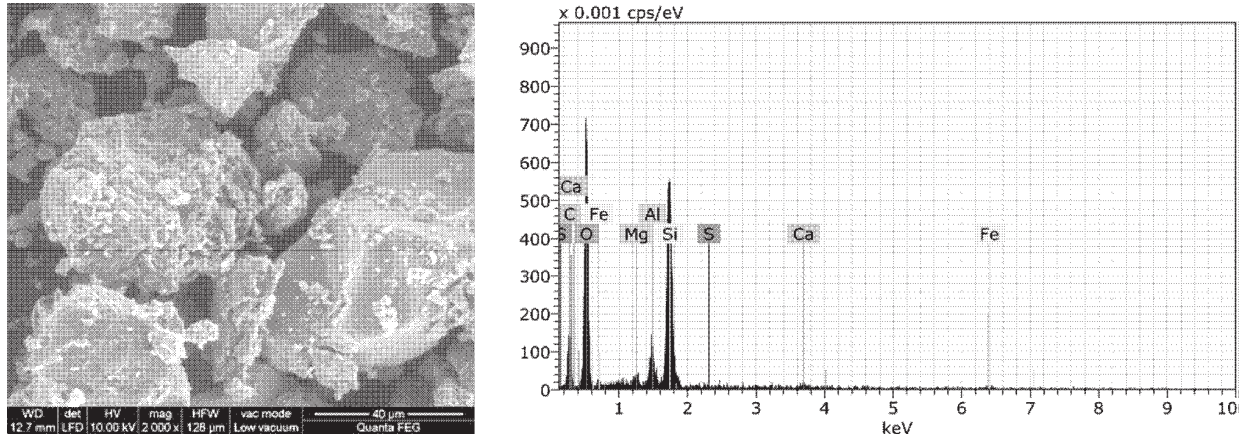
Two clayey soils (designated D1 and D2) were used and their properties are given in Table 1 in terms of Atterberg’s limits and particle size distribution. The geotechnical properties showed the soil samples as clay of high compressibility as per IS: 1498-1970[11]. The stabilizer used to strengthen the soil is the Phospho Gypsum (PG), which is one of the industrial waste products obtained from fertilizer industry. Chemical analysis shows the different major constituents present in the soils and the Phosphogypsum are listed in Table 2. The morphological changes of the untreated samples were studied by Scanning Electron Microscopy images and its relative element compositions of the samples were analyzed using Energy Dispersive X-ray analyzer (EDS) are shown in Figure 1.

Table 1
Engineering Properties of Soils

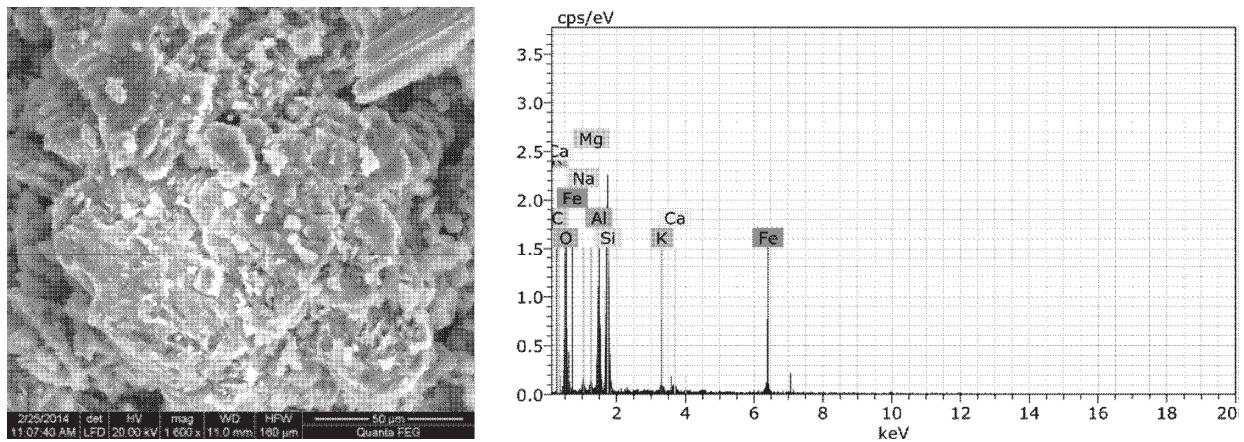
Description	Particle size distribution			Liquid limit (%)	Plastic limit (%)	Shrinkage limit (%)	Plasticity index (%)	Specific Gravity
	Clay (%)	Silt(%)	Sand(%)					
D1	70	28	2	51.5	20	7.71	31.5	2.23
D2	66	32	2	69	21.81	9.94	47.19	2.37

Table 2
Chemical Components of Soils and Admixture

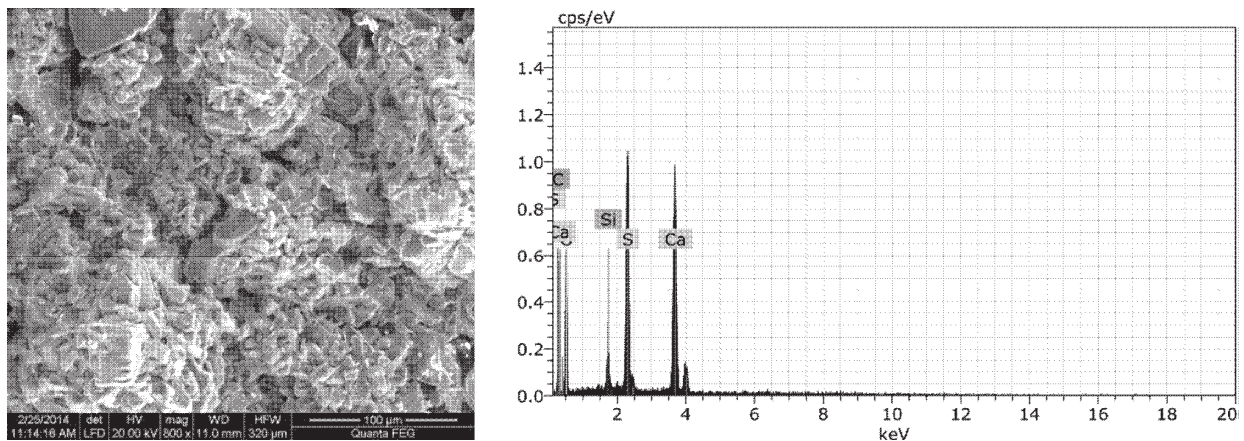
Chemical constituents	Chemical composition, %		
	Soil D1	Soil D2	Phosphogypsum
SiO ₂	53.4	56.8	3.8
CaO	3.5	1.1	32.3
SO ₃	0.1	0.2	44.6
MgO	0.7	0.6	---
Fe ₂ O ₃	7.8	7	---
Al ₂ O ₃	14.8	14.9	---



(a) SEM and EDS image of Soil D1



(b) SEM and EDS image of Soil D2



(c) SEM and EDS image of Phosphogypsum

Figure 1: SEM and EDS Results of Untreated Soils and Phosphogypsum

3. PREPARATION OF SPECIMENS

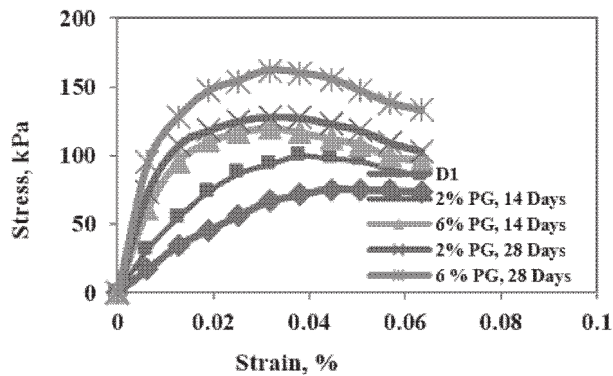
The treated specimens were prepared with Phosphogypsum of different percentages of 2, 4, and 6%. The samples for strength test of unconfined compressive strength were prepared with the compaction characteristics[12] of

virgin soils. Specimens after preparation has cured for specified periods by sealed inside the plastic bags for the particular curing ages and tested in UCC testing machine to determine the strength characteristics. After the strength test has conducted, specimens from untreated and treated conditions were dried in oven at 60°C until the constant weight and broken with fractured surface is taken for scanning electronic microscope (SEM) and in powdered form for XRD tests. The powdered soil specimens were again using to determine the free-swell index by saturated in water.

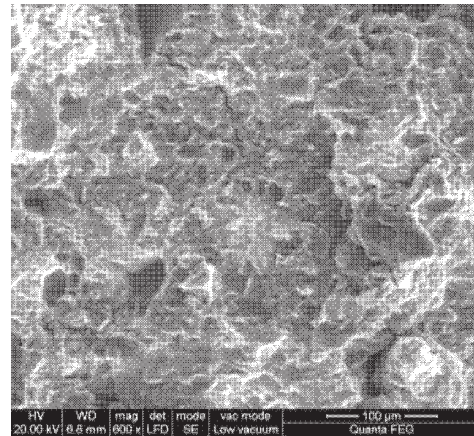
4. CHARACTERISATION OF UNTREATED AND TREATED MATERIALS

4.1. Strength and Microstructure Characteristics

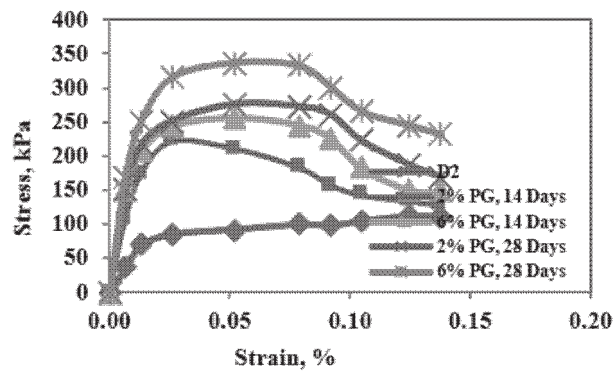
The development in strength characteristics are done by conducting the unconfined compressive strength test [13] on untreated and treated samples. Figures 2 shows the development of the unconfined compressive strength and microstructural changes of treated soils, in relation to curing time for soils D1 and D2. It was obtained that considerable higher compressive strengths are obtained for both the soils due to the effect of Phospho Gypsum at various proportions with the soils. It is inferred from the chemical composition that the effect of Phospho Gypsum on strength is due to the lime content which promotes the pozzolanic reactions.



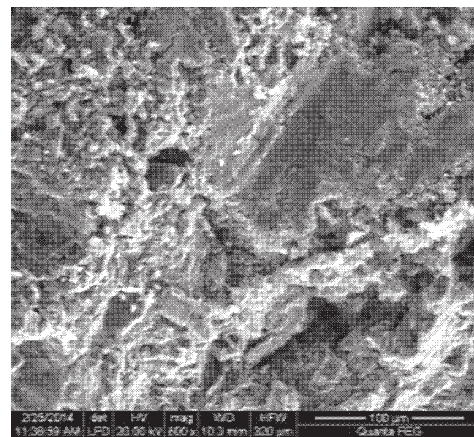
(a) Stress-strain variation for soil D1 mixed with different percentages of PG



(b) SEM image of soil D1 treated with 6% of PG at 28th day



(c) Stress-strain variation for soil D2 mixed with different percentages of PG



(d) SEM image of soil D2 treated with 6% of PG at 28th day

Figure 2: Strength results and SEM images of treated soils

The peak values obtained for each percentages of admixture for the soil samples at specified curing periods of 7, 14 and 28 days are shown in Table 3. Due to the treatment of soil with PG, the development in strength characteristics are achieved by the formation of granular structure in soil due to the cementing products with the soil and admixture minerals such as C-S-H and C-A-H between the particles.

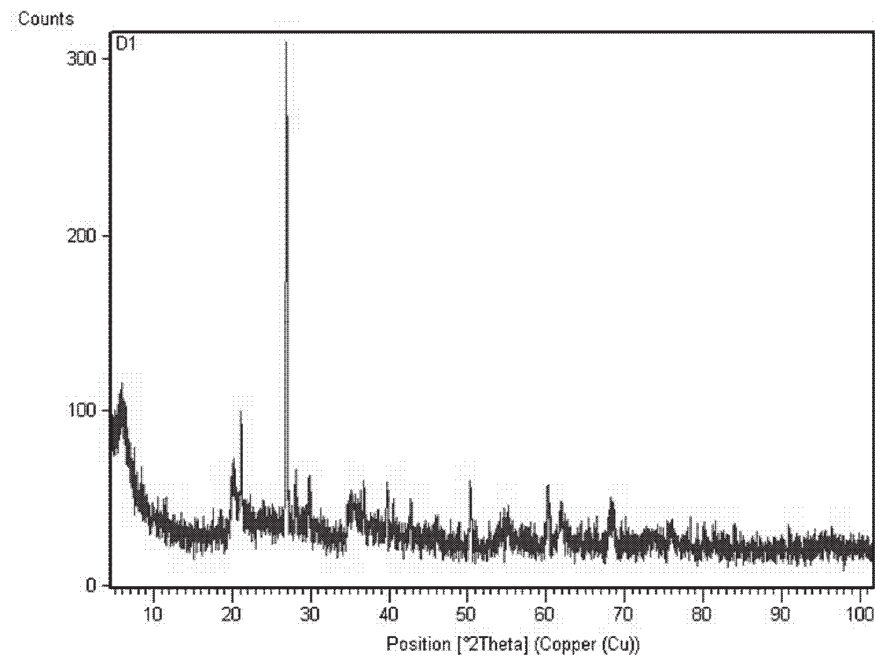
It is clearly observed that the higher compressive strength is observed with more dosages of stabilizer combinations in soil samples. The results showed that the UCS peak stresses are increased to 1.16 and 1.75 times at the 28th days of curing by the addition of 6% of PG in soil samples. Stabilized soil micrographs shows that, each clusters in soil are overlapped and joined together due to hydration with the mineral grains present in soil and stabilizer [14, 15]. The Phospho Gypsum particles accelerate pozzalonic reactions due to their high reactive capacity. Similarly the increase in curing period and percentages of stabilizer also results in the improvement in microstructure with a proper bridging with soil and stabilizer.

Table 3
Unconfined Compressive Strength Values for Untreated and Treated Samples at Different Curing Periods

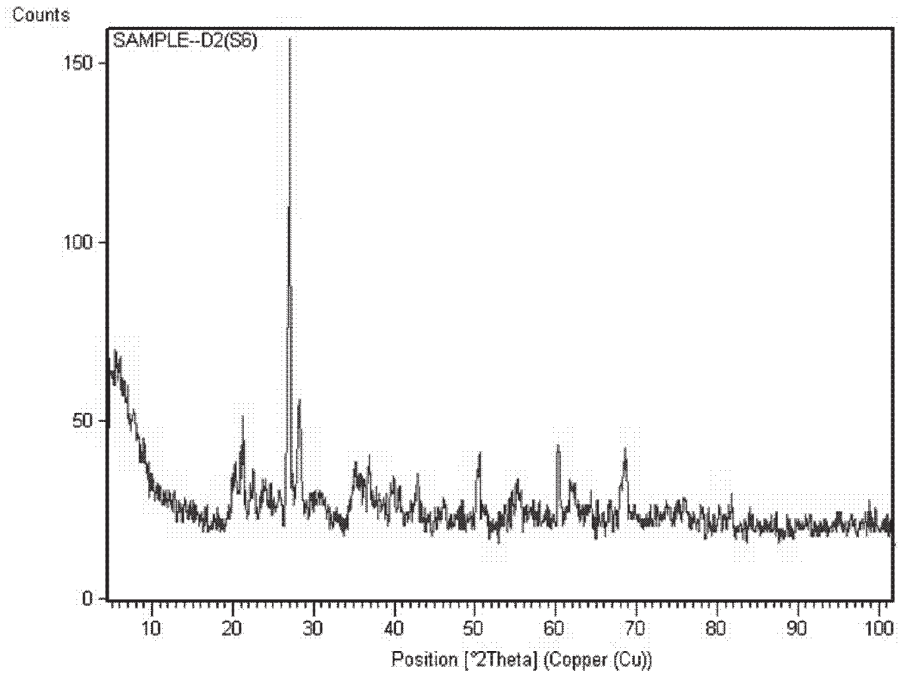
Soil sample	Unconfined compressive strength (kPa)										
	0 days		7 days			14 days			28 days		
	0%	2%	4%	6%	2%	4%	6%	2%	4%	6%	
D1	75	82	88	95	100	112	119	128	143	162	
D2	122	134	176	208	221	224	257	276	292	336	

4.2. X-Ray Diffraction

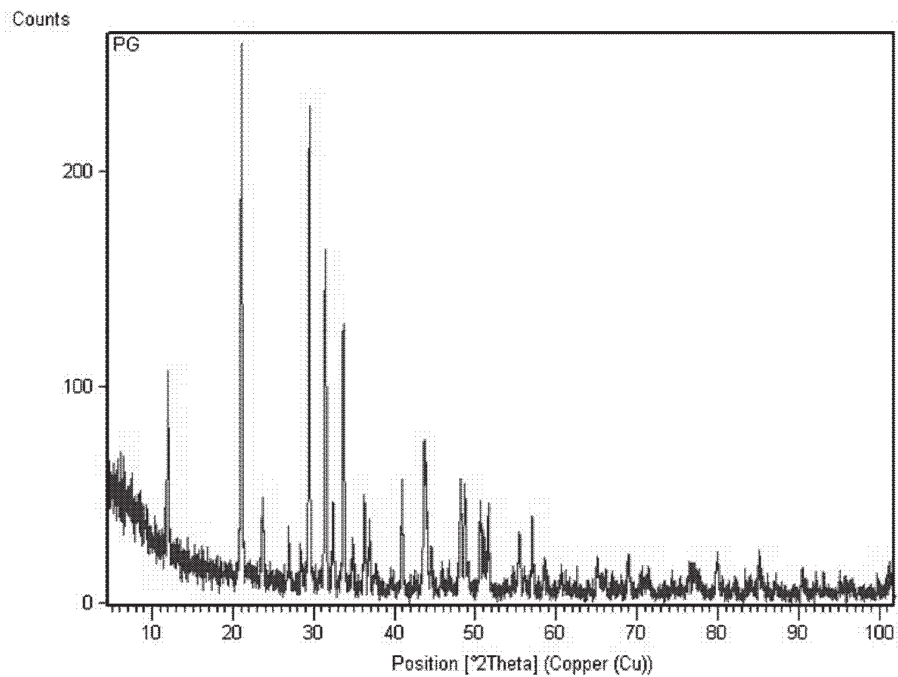
Energy dispersive spectroscopy is used to obtain the minerals in sample and it shows an X-ray mapping of each elemental composition. The XRD results of the textured samples of admixture, raw and treated soil were shown in Figure 3. The results of stabilizer and treated soil show the main mineralogical constituents of free CaO which



(a) XRD image of soil sample D1

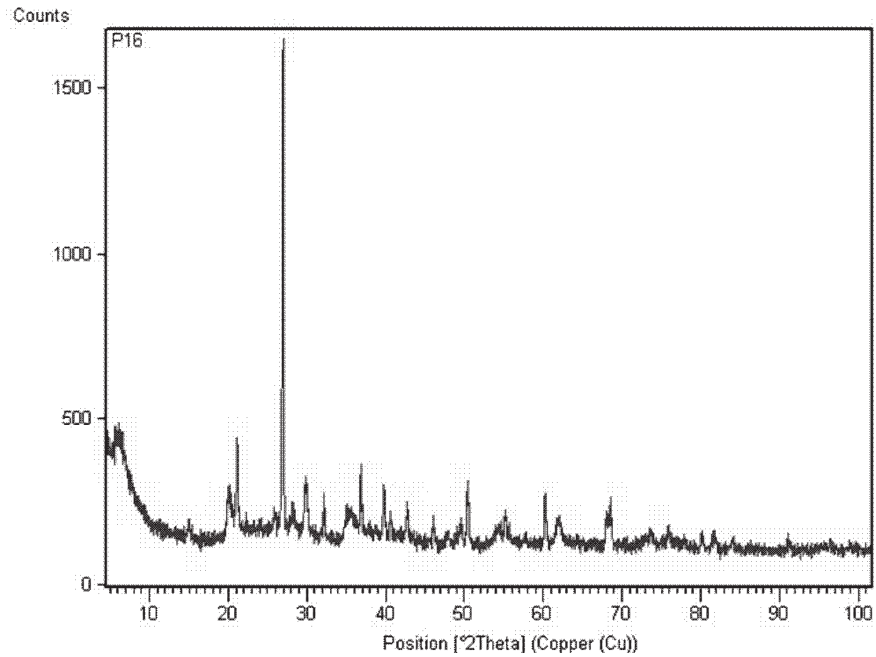


(b) XRD image of soil sample D2

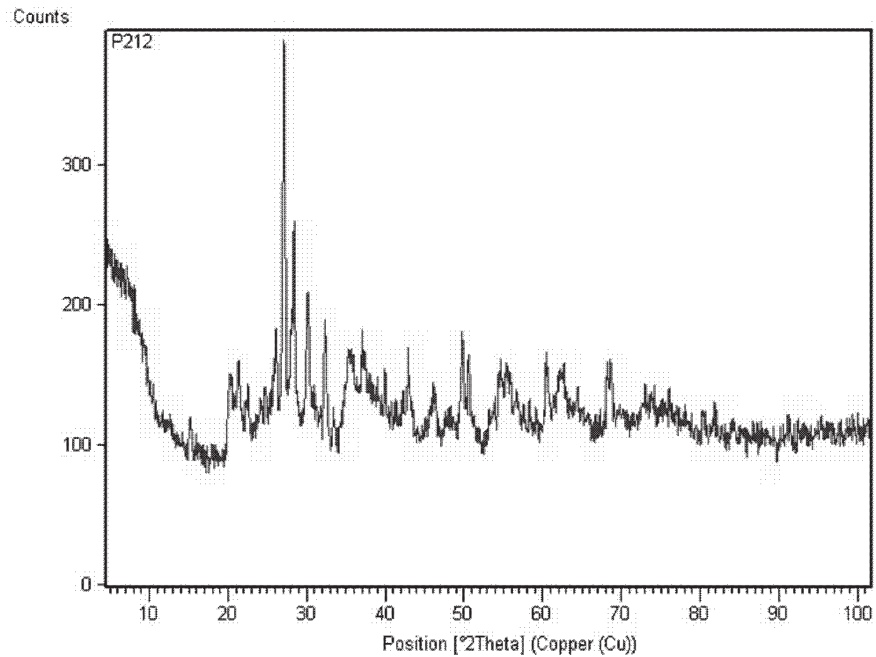


(c) XRD image of Phosphogypsum

leads to the development in strength of soil by the treatment process. The typical X-ray diagrams presented for 14 day old specimens stabilized with 6% PG for both the samples shows the possibility of compounds formed as hydration products resulted of the reaction mainly of the CaO and SiO₂ contained in clay with the Phosphogypsum. This is showing the effect of the addition of PG which has enhanced the strength attainment in clayey soil with different dosage of admixture. The free CaO changes the properties of clay through physiochemical modifications mainly by the pozzolanic reaction between the clayey soil and stabilizer [16, 17].



(d) XRD image of soil sample D1 treated with 6% of PG at 14th day



(e) XRD image of soil sample D2 treated with 6% of PG 14th day

Figure 3: X-ray diffraction data of Phosphogypsum, untreated and treated soils

4.3. X-Ray Fluorescence

Microstructural study was also conducted based on XRF analysis and its results showed an improvement in treated soil by the formation of cementitious compounds in stabilized form of soil samples. The XRF results indicates that the concentrations of elements has increased more in treated soils due to admixture addition and results showing the quantities of elements of the stabilizer, untreated and treated soil samples are presented in

Table 4. There was a large variation in the quantities of few elements Ca, Si and Al, which were determined in the treated soil. Mainly these elements contribute the compactibility of minerals to form pozzolonic compounds which has given the greater strength in stabilized soil.

Table 4
XRF Test Results of Phosphogypsum, Untreated and Treated Soils

Compound concentration	Phospho Gypsum	Soil D1	Soil D2	D1 + 6% PG, 14 Days	D2 + 6% PG, 14 Days
Na	38.4	35.8	30.33	33.0	33.3
Mg	7.2	10.3	7.326	11.4	6.7
Al	48.8	376.9	426.404	373.5	500.0
Si	460.1	3816.9	3729.226	3797.2	3447.4
S	19046.2	95.8	311.961	2703.0	2889.4
K	20.6	366.1	1466.03	391.5	1929.1
Ca	38089.9	5854.9	3376.007	9696.2	6971.3
Fe	432.1	29379.6	27031.86	25826.2	26610.1

4.4. Free Swell Index tests

The Figure 4 showing the effect of Phosphogypsum in soil D1 and it shows a reduction in swelling level compared with the untreated specimen. The stabilization process changes the soil specimen characteristics and it leads to the decrease in the swelling potential of soils with the higher dosages of stabilizer. The Free Swell Index values determined from the series of tests showed a decreasing trend with an increase of Phosphogypsum quantity added to the soil and with an increase in curing time. The variation of Free Swell values at different PG dosages at increasing curing periods of 7, 14, 28 days are shown in Table 5.

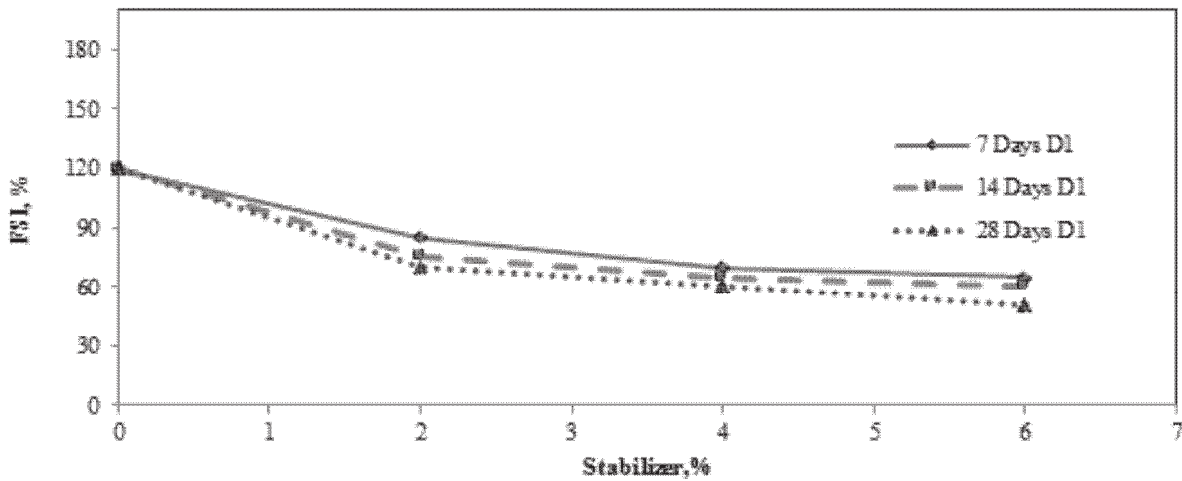


Figure 4: Free swell index variation in untreated and treated soil D1

5. CONCLUSIONS

The following are the conclusions drawn based on the experimental investigation carried out on the soil stabilised with Phosphogypsum.

The SEM micrographs clearly illustrate the improvement in stabilized clay due to the formation of cementing products with the addition of Phospho Gypsum.

Table 5
Variation of Free Swell Index of PG Stabilized Soils

Sample	Free Swell Index (%)					
	Soil D1			Soil D2		
	7 days	14 days	28 days	7 days	14 days	28 days
Soil + 0% PG		120			109	
Soil + 2% PG	85	75	70	90	85	80
Soil + 4% PG	70	65	60	80	75	65
Soil + 6% PG	65	60	50	70	60	55

From the material characterization by XRD and XRF estimates the amount and possibility of cementing compounds which formed during stabilization process, which has enhanced the strength attainment in clayey soil with different dosages of admixture.

The compressive strength results showed an increase in strength values in the order of 1.16 and 1.75 times at the 28th days of curing by the addition of 6% of PG in soils the clayey soils.

Free swell index values on treated and untreated soil samples, indicates a reduction in values of 50% and 55% at the 28th day for both soil samples.

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