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Chemical Stabilization of Expansive Esna Shale, Qena Region, Egypt

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Abstract— **This investigation deals with a chemical stabilization program of expansive Esna shale, which widely distributed on the surface at the arid region along both West and East of Qena governorate, Egypt. Chemical stabilization program was applied to improve the geotechnical properties of the studied Esna expansive shale using both lime and cement kiln dust (CKD, as by product). Optimum-lime and -cement kiln dust (CKD) contents were measured using pH test according to Eades and Grimm method. Geotechnical tests of both natural and treated Esna shale samples as free swelling, ultrasonic velocity (Vp), and unconfined compressive strength (qu) were carried out. The results showed that the geotechnical properties of the studied expansive Esna shale samples mixed with CKD and/or lime were improved and stabilized. Also the results indicated that curing time increase from 7 to 90 days having a great influence on the improvement of the geotechnical behavior of the studied expansive Esna shale samples.**

Keywords- **Chemical Stabilization, Lime, Cement Kiln Dust (CKD), Free Swelling, Unconfined Compressive Strength (qu), Ultrasonic Velocity (VP), Expansive Esna Shale.**

I. Introduction

Expansive shale has a wide distribution in Egypt especially in Upper Egypt. The arid region leads to form the expansive clay minerals. The expansive shale results in many geotechnical problems in engineering constructions like a heave. To prevent the heave of the expansive shale and its damage effect on the engineering constructions, several methods are applied. One of the most important treatment methods of the expansive shale is a chemical stabilization. Chemical stabilization for expansive shale is applied mostly by adding lime or cement or a mix of both. Other techniques are developed which use both flay ash and cement kiln dust (CKD), as by-products, because of their low cost. Flay ash results from the industry of coal, and cement kiln dust results from industry of cement. These techniques have another advantage; as flay-ash and CKD are toxic to environment, they should to be buried in sanitary landfills at a certain depth or storage in a certain containers, this processes are so expensive. So that reusing them in the chemical stabilization of expansive shale has environmental and economic benefits [1].

A. Location of the Study Area

The studied area exposed at El-Balas village, West of Qena City, at 26° 1' 3.8" N and 32° 43' 18.7" E, and at Gebel El-Quraya, East of Qena City, at 26° 24' 57.8" N and 33° 5' 20.7" E. The studied samples collected belong to Esna Formation exposed at the both El-Balas and El-Quraya sections (Figures 1 and 2).

B. Previous Studies

Several researches were carried out on the studied area including geological, structural, tectonic, stratigraphic studies, and etc. Few researches were carried out on the studied area including geotechnical and chemical stabilization like [1], [2], [3], and [4].

Figure1. Location of the study area

C. Scope of the Study

The present study deals with the effect of lime and cement kiln dust (CKD) on geotechnical properties like free swelling, unconfined compressive strength, and ultrasonic velocity of expansive shale of Esna Formation distributed at the studied area, West and East of Qena governorate. Evaluation of both bearing-capacity and swelling potentials of tested expansive Esna shale. This investigation also discusses the influence of long term curing of the treated Esna shale samples on their geotechnical properties and their microstructures.

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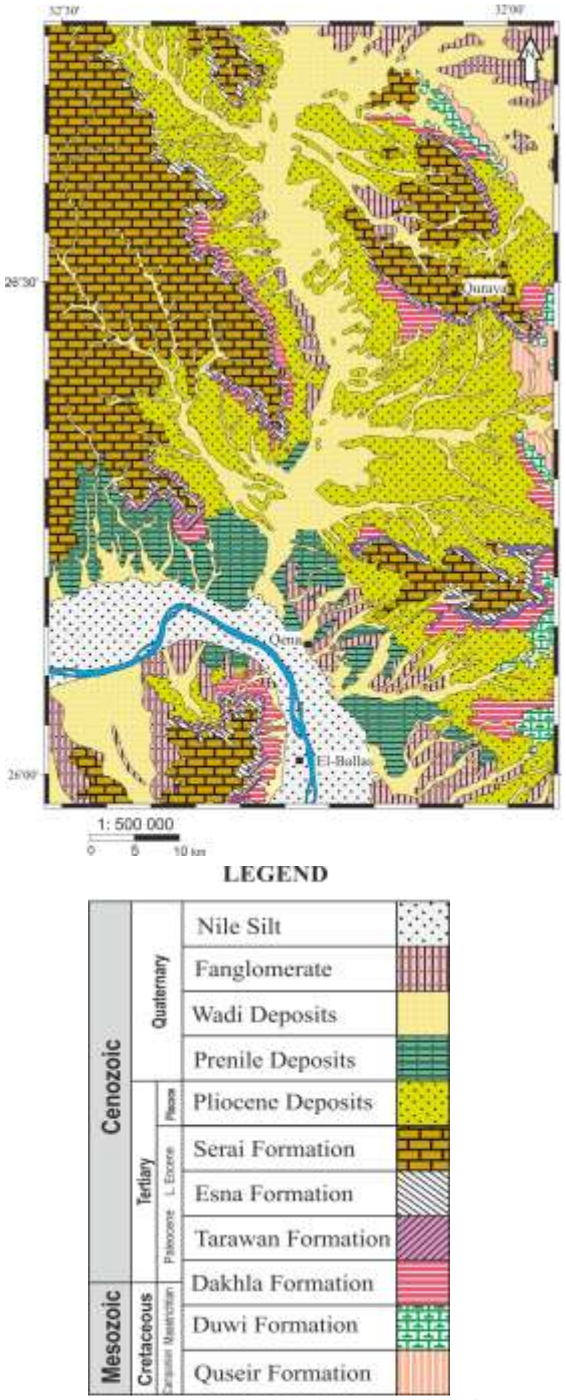

Figure2. Geological map of the study area, modified after [5]

II. Materials and Methods

A. Materials

• Esna Formation

Esna Formation deposited during Paleocene to Eocene age. It is composed of green shale and marle enclosing carbonate intercalations (Figures 3 and 4). It overlies the Tarawan Formation and underlies the Thebes group. Esna Formation has a wide distribution in the central and southern parts of both Eastern and Western Deserts of Egypt and along the Red Sea [6]. Their index properties and engineering classification are showed in Table 1.

TABLE 1 INDEX PROPERTIES AND CLASSIFICATION OF STUDIED ESNA SHALE

Sample	LL %	PL %	PI	Wn (%)	On (%)	δd	MDD (kg/m³)	OMC (%)	Classification	
									USCS	AASHTO
EB	55.20	38.13	17.07	4.93	4.62	1.667	1.45	24.50	MH	A-7-5
EQ	53.60	37.33	16.27	3.13	3.65	1.73	1.455	24.20	MH	A-7-5

LL= Liquid Limit, PL= Plastic Limit, PI= Plasticity Index, Wn= Natural Water Content, On= Natural Organic Content, δd= Natural Density, MDD= Maximum Dry Density, OMC= Optimum Moisture Content, EB= Esna Shale of El-Balas Section, EQ= Esna Shale of El-Quraya Section, USCS= Unified Soil Classification System, AASHTO= American Association of State Highway and Transportation Officials.

Figure3. Esna shale at the study area (El-Balas section)

Figure4. . Esna shale at the study area (El-Quraya section)

• Cement Kiln Dust (CKD)

CKD is a fine material, 75-80% passing 200meshes, and its appearance similar to Portland cement [7]. It is produced as a waste material from Portland cement manufacturing. The studied CKD was obtained from Qena cement factory, Qift, Qena, Egypt. CKD can be reused in many purposes as stabilizing landfills, soil stabilization, agricultural applications, in asphalt pavement, environmental remediation, and as fill material for embankments structures.

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- ***Lime***

The lime used in the present study is hydrated lime $Ca(OH)_2$ (Calcium hydroxide), which resulted from reaction between quicklime (CaO) (Calcium oxide) and water. Addition of lime has two stages of reactions; flocculation and pozzolanic reactions. Lime can react with all clay minerals; however it has a more significant effect on montmorillonites than kaolinites, as montmorillonites have a higher plasticity index (PI) [8]. Firstly; flocculation processes occur immediately after adding lime to the soil with sufficient amounts of water, so that it is referred to as short term reaction. Flocculation results from cation exchange processes; as the Sodium and other cations on the surfaces of clay minerals exchanged by Calcium cations of lime, causing the fine grains of the soil to flocculate forming aggregates. These process resulting in increasing the grain size to a sand size [9]. Secondly; pozzolanic reactions take place after flocculation, when there is sufficient lime and water content.

B. ***Methods***

Chemical stabilization laboratory program using cement kiln dust and cement kiln dust with lime was conducted including three main steps. The first step was a preparation of the sample; sample was dried in the air and then it was put into the oven at 50˚C for 24 hours. The dried sample was crushed in crushing-machine. The second step was a determination of an optimum cement kiln dust and an optimum cement kiln dust with lime contents to stabilize the studied sample using pH-test [10]. The third step was a preparation of the cement kiln dust-stabilized samples compacted at a maximum dry density and an optimum water content and a preparation of the cement kiln dust with lime-stabilized samples compacted at a maximum dry density and an optimum water content. There are two methods to evaluate the geotechnical properties of the stabilized samples. The first method is destructive and including a measurement of several geotechnical parameters like unconfined compres- sive strength. The second method is nondestructive and including a measurement of ultrasonic velocity of the studied sample before and after the chemical stabilization [2]. Studied Esna shale samples classified using grain size analysis tests according to [11]. Optimum-lime and -CKD were detected using pH method as pozzolanic reactions are most likely to occur in 12.4 pH environment [8], while optimum water content for mixtures were determined using standard proctor test according to [1**2**]. The chemical stabilization program was carried out using lime, CKD, and mixture of lime and CKD. The treated Esna shale samples were cured in a water bath (under about 98% moisture environment and under chamber temperature) for 7, 28, and 90 days to investigate the influence of curing time on enhancing geotechnical properties of the treated Esna shale samples. Free swelling test of natural and treated Esna shale samples was carried out according to [13]. Geotechnical properties of natural and treated samples were measured using destructive method using unconfined compressive strength test according to [14]. Also they tested using nondestructive method using ultrasonic velocity (longitudinal velocities) [**15**] using JAMES instrument.

TABLE 2 pH-RESULTS

Sample	Lime (L) %	CKD %	Mixture L/CKD	
			L %	CKD %
EB	4	10	2	6
EQ	4	10	2	6

X-ray diffraction (XRD) and X-ray fluorescence (XRF) were applied to determine mineral and chemical compositions of the investigated samples. Scanning electron microscope (SEM) was applied to understand the changing in the microstructures and the development of cement materials with increasing curing time.

III. Results

A. ***pH-Test Results***

pH value of 12.4 is the most suitable environment for proceeding hydration reactions [16]. The results showed that the optimum percent of lime content is 4% for both Esna shale samples collected from El-Balas section (EB) and for Esna shale samples collected from El-Quraya section (EQ). The optimum CKD content of both EB shale sample and of EQ shale samples is 10%. The optimum lime plus CKD content of both EB shale sample and of EQ shale sample is 2% L+6% CKD (Table 2).

B. ***Compaction Test Results***

Optimum water content (OWC) and maximum dry density (MDD) were determined using standard proctor test. The results illustrated in Figure (5). The results proved that the addition of lime, CKD, and mixture of them can decrease MDD and increase OMC. MDD values of EB samples decreased from 1.43g/cm^3 for the natural compacted sample to 1.35, 1.36, and 1.33g/cm^3 for lime, CKD, and the mixture (L plus CKD), respectively. While OWC values increased from 24.50% for the natural sample to 31.50, 32.20, and 33.00% for lime, CKD, and the mixture, respectively. However, for EQ samples the MDD values decreased from 1.455g/cm^3 for the natural compacted sample to 1.360, 1.365, and 1.340g/cm^3 for lime, CKD, and the mixture, respectively. While OWC values increased from 24.20% for the natural sample to 31.70, 32.50, and 33.31% for lime, CKD, and the mixture, respectively.

C. ***Free Swelling Test Results***

Table 3 showed the free swelling percent values of the studied expansive shale before and after the treatment. The results illustrated that the free swelling percent value of the natural shale (EB) was 98.00% and it described as moderately expansive according to [13]. After 90 days curing, the addition of lime, cement kiln dust and cement kiln dust plus lime led to a reduction of the free swelling percent from 98.00% to 41.67.00, 25.00, and 07.00%, respectively. In case of shale samples (EQ), its free swelling value was 86% and it described as moderately expansive according to [13]. The addition of lime, cement kiln dust and cement kiln dust plus lime led to a reduction of the free swelling percent to 06.67, 00.00, and 00.00%, respectively.

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TABLE 3 FREE SWELLING AND ULTRASONIC TESTS RESULTS

Sample	Additives (%)	Curing Time (day)	Vp-Velocity (m/sec)	Free Swelling (%)
EB	Natural compacted sample	0	0190.0	098.33
	4 lime	7	0400.0	056.00
		28	0485.0	010.5
		90	0781.0	041.67
	10 CKD	7	1238.0	012.33
		28	1545.0	021.67
		90	1730.0	025.00
	2L + 6CKD	7	0840.0	030.67
		28	1156.0	040.00
		90	1471.0	007.00
EQ	Natural compacted sample	0	0167.0	086.66
	4 lime	7	0570.0	040.00
		28	0850.0	010.00
		90	0578.0	006.67
	10 CKD	7	1508.0	010.00
		28	1510.0	034.33
		90	1519.0	000.00
	2L + 6CKD	7	1124.0	031.67
		28	1598.0	000.00
		90	1529.0	000.00

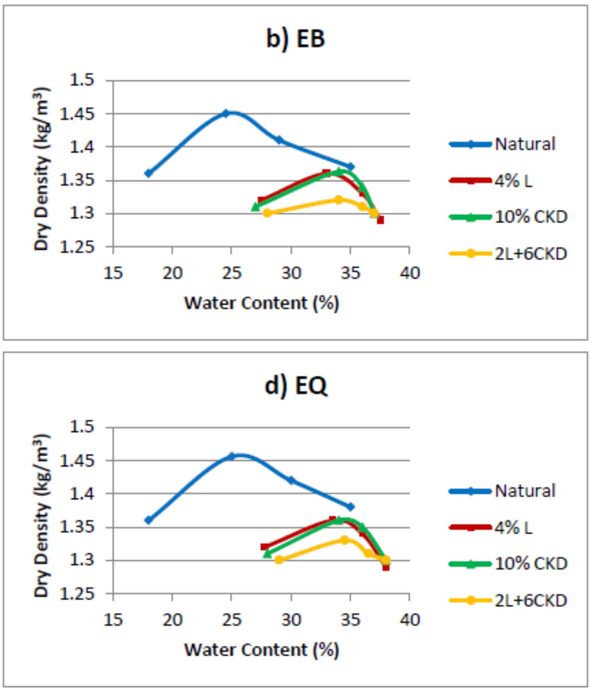

Figure5. Compaction curves of studied Esna shale samples

D. *Ultrasonic Velocity (Vp) Test Results*

Table 3 showed the ultrasonic velocities values (Vp) of the studied expansive Esna shale before and after the treatment. The results showed that the velocity value of the natural Esna shale (EB) was 190m/sec. After 90 days curing, the addition of lime, cement kiln dust and cement kiln dust plus lime led to an increase of the value from 190m/sec to 781, 1730, and 1471m/sec, respectively. In case of natural Esna shale samples (EQ), its Vp value was 167m/sec and the addition of lime, cement kiln dust and cement kiln dust plus lime led to an increase of the value from 167m/sec to 578, 1519, and 1529m/sec, respectively. Figure 6 illustrates the used ultrasonic velocity instrument. By measuring the travel time (ta) of the waves transmitted through the specimen, it is possible to calculate the P-wave velocity (Vp) as follow: $Vp = x/ta$ Where (x) is the length of the prepared sample, it is usually equal to 0.12 m [15].

Figure6. Ultrasonic velocity instrument

E. *Unconfined Compressive Strength (qu) Test Results*

Figure 7 showed the unconfined compressive strength values (qu) of the studied expansive Esna shale before and after the treatment. The results illustrated that the strength value of the natural shale (EB) was 127.07Kpa. After 90 days curing, the addition of lime, cement kiln dust and cement kiln dust plus lime led to an increase of the value from 127.07Kpa to 447.93, 1847.47, and 1927.05Kpa, respectively. In case of Esna shale samples (EQ), its qu value was 276.67Kpa and the addition of lime, cement kiln dust and cement kiln dust plus lime led to an increase of the

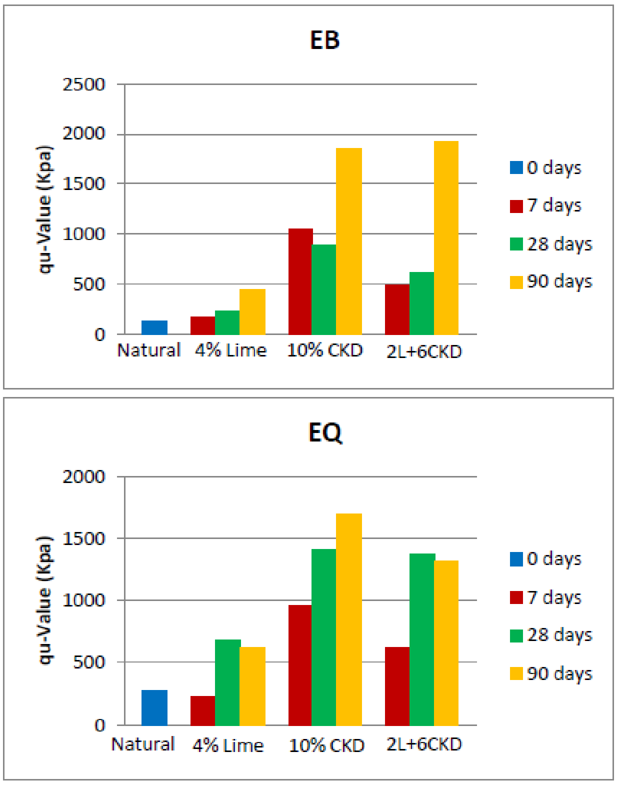

Figure7. Unconfined compressive strength values of the studied samples

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value from 276.67Kpa to 624.01, 1696.97, and 1315.77Kpa, respectively. Figure 8 showed the development of behavior of the studied Esna shale samples from ductile to brittle due to the treatment process.

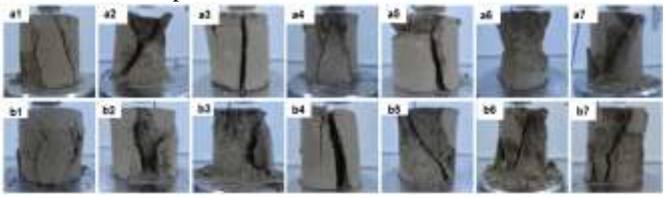

Figure8. The development of the studied samples behavior from ductile (for untreated compacted samples) to brittle (for stabilized samples); a) EB; a1) Natural Compacted EB; a2) EB+CKD 7days; a3) EB+CKD 28days; a4) EB+CKD 90days; a5) EB+L/CKD 7days; a6) EB+L/CKD 28days; a7) EB+L/CKD 90days; b) EQ; d1) Natural Compacted EQ; b2) EQ +CKD 7days; b3) EQ +CKD 28days; b4) EQ +CKD 90days; b5) EQ +L/CK 7days; b6) EQ +L/CKD 28days; b7) EQ +L/CKD 90days

F. *XRD and XRF Analysis*

Studied Esna shale samples were analyzed using XRF to detect their chemical composition (Table 4), and using XRD to determine their mineral composition. The used CKD is mostly similar in its composition to Portland cement containing a high percent of CaO (67.7%) and a low percent of SO_3 (<2%) [1]. Sulfide oxides are considered harmful for hydration reactions as they result in forming ettringite. The results showed that the percent of both SiO_2 and Al_2O_3 of the tested Esna shale samples is ranging from 28-38%. XRD results illustrated that all tested Esna shale samples consist of 15-23% quartz, while they contain abundant of clay minerals ranging from 30 to 34% including both montmorillonite and kaolinite. Calcite also present in a high percent equal to 42%. Anhydrite is less abundant than calcite, it found in a percent equal to 11%.

TABLE 4 CHEMICAL COMPOSITION OF THE STUDY ESNA SHALE

Compound Formula	Concentration (wt %)	
	EQ	EB
Na_2O	00.706	01.171
MgO	02.049	02.699
Al_2O_3	06.225	08.699
SiO_2	21.984	29.858
P_2O_3	00.416	00.824
SO_3	01.308	00.350
K_2O	00.544	00.847
CaO	32.737	22.274
TiO_2	00.462	00.691
Cr_2O_7	00.032	00.047
MnO	00.105	00.041
$Fe_2O_3^{tot}$	04.764	05.712
NiO	00.035	00.037
CuO	00.014	00.009
ZnO	00.052	00.036
SrO	00.160	00.132
ZrO_2	----	----
Cl	0.406	00.891
L.O.I	28.000	25.600

(L.O.I): loss of Ignition at 1000°C

G. *SEM Results*

Figures 9 (a & e) illustrated the micrographs of the natural expansive Esna shale of both El-Balas section, EB, (a) and El-Quraya section, EQ, (e) which showed laminated arrangement or sheet structure of clay particles. Figures 9 (b & f) showed the micrograph after 7 days curing of the treated sample (EB) with 6% CKD plus 2% L (b) and the micrograph of the treated sample (EQ) with 10% CKD (f). The micrographs illustrated crumbs of floccules with a porous nature and cementitious compounds, calcium aluminum hydrate (CAH) and calcium silicate hydrate (CSH), coating the relics of the silt particles and the flocs. The edges of the relics of the particles were attacked by cement kiln dust and their boundaries had a ragged-form. Additionally, the reaction of cement kiln dust with clay led to a formation of an aggregate of various sizes and that was responsible for the increase in the porosity. Similar microfabric structure was observed by [1, 2, 3, and 4]. Figure 9 (c & g) showed the micrograph after 28 days curing of the treated sample (EB) with 6% CKD plus 2% L (c) and the micrograph of the treated sample (EQ) with 10% CKD (g).

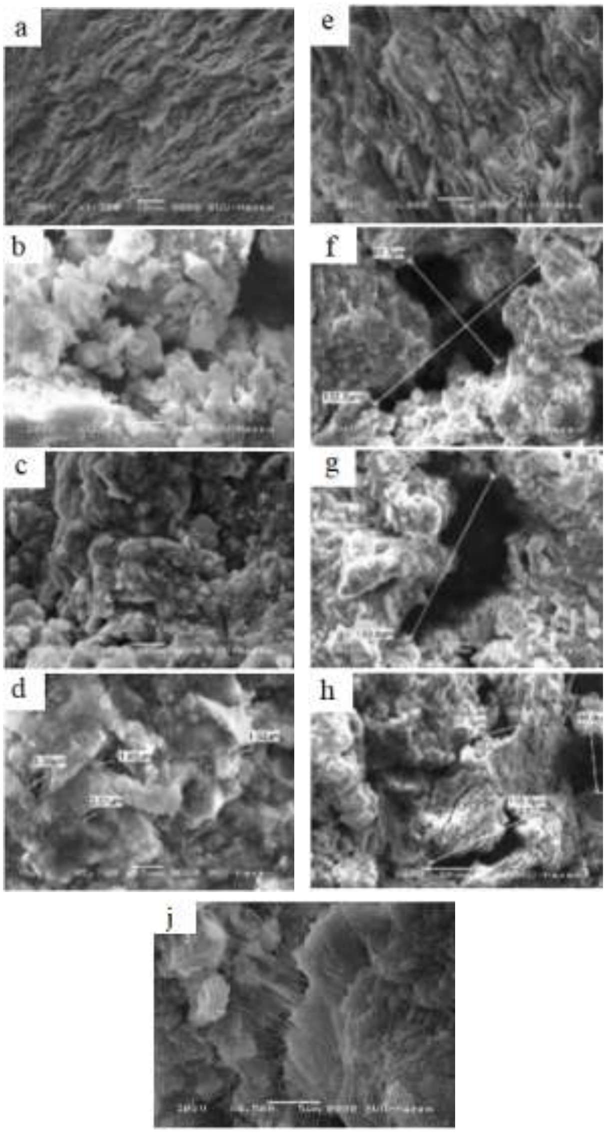

Figure9. The development of the microstructures of the treated Esna shale samples

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The micrographs illustrated cementitious compounds (due to the beginning of pozzolanic reaction) coated and joined the shale and the cement kiln dust particles. The pores were partially filled with the cementitious compound and their diameter reduced relatively. Figure 9 (d & h) illustrated the micrograph after 90 days curing of the treated sample (EB) with 6% CKD plus 2% L (d) and the micrograph of the treated sample (EQ) with 10% CKD (h). The micrographs showed cementitious compounds (due to increase of the pozzolanic reaction) coated and joined the shale and the cement kiln dust particles. The pores were filled with the cementitious compound and their diameter reduced strongly. Figure 9 (j) showed the micrograph of the treated Esna shale sample (EB) with 6% CKD plus 2% L cured for 28 days. The microstructure illustrated ettringite mineral which have fibers crystals.

IV. Discussions and Conclusions

Esna Formation exposed at Qena region is expansive shale (its free swelling percent ranging from 86 to 98 % and described as moderately expansive shale according to [13]). Due to its occurrence in the arid region and containing high percent of expansive clay minerals is described as problematic shale and it needs to be treated. Depend on pH-test it was founded that the optimum contents of the chemical additives used for stabilization of Esna shale were as follow; 4% if using lime, 10% if using CKD, and 2% lime plus 6% CKD in the case of using a mixture of lime and CKD. The improvements of the engineering properties due to use of the chemical additives can be explained by two basic reactions: short-term reactions consisting cation exchange and flocculation and the long-term reaction named pozzolanic activity. During the first stage of the reaction between the chemical additives and the clay, excesses of calcium ions in lime or in cement kiln dust replace all other monovalent cations in the clay and change the electrical charge density around the clay particles. This results in an increase in the inter-particle attraction causing flocculation and aggregation and a consequent decrease in the plasticity of the soil [17].

Unconfined compressive strength values (qu) and ultrasonic velocities values (Vp) of the treated Esna shale samples revealed that using CKD alone can increase the strength and the velocity of the treated samples more than that using lime, or than using a mixture of lime and CKD. Also the compressive strength and the ultrasonic velocity increased with increasing curing time from 7 to 90 days. Increasing Vp-values with increasing curing time proves the decreasing of pore size in treated samples. Esna shale sample Vp- values reach their higher velocities when treated with 10% CKD and cured for 28 and 90 days. The use of ultrasonic p-wave velocity method is a simple and practical (non-destructive) tool to evaluate the stabilization process using lime, cement kiln dust, and lime/cement kiln dust and would need many studies to establish a guideline and standard specification. Ttreatment program of the Esna shale samples led to decrease the free swelling percent. Generally, the reduction of free swelling was increased with increasing the curing time from 7 to 90 days. But some treated Esna shale samples have relatively high percent of free swelling that may be due to the formation of ettringite mineral which considered as expansive mineral. Ettringite result may be from the occurrence of sulfate in an inhomogeneous distribution in the Esna shale. Its prismatic crystals (Figure 9j) leading to increase the volume of the treated samples. To avoid the problems of ettringite crystals, must be emphases the following: In case of, sulfate (Gypsum) bearing sample treated with lime and CKD requires to special technical methods in situ. National Lime Association [18] reported that these technical methods in situ include "two applications of lime (and/or CKD), the first before the first mixing and the second after the mellowing period. The moisture content of the soil is raised to 5% over the optimum during a multi-day mellowing period to soluble as many as sulfate as possible and to force ettringite to form before compaction. Once formed, ettringte is relatively stable and is unlikely to cause further problems. After the mellowing period, additional lime (and/or CKD) is added to the soil and construction proceeds normally.

Finally, CKD as by-product of Qena cement plant (at Qift industrial city) contains relatively small percent of sulfates (<2%) and can be utilized to treat and stabilize the expansive Esna shale as economical (cheaper) alternative to Portland cement and other (expansive) chemical stabilizers. The use of cement kiln dust for chemical stabilization applications is an environmental solution of the problems associated with its disposal process.

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