Stabilization of oily contaminated clay soils using new materials: Micro and macro structural investigation

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Abstract. Clay soils have a big potential to become contaminated with the oil derivatives because they cover a vast area of the earth. The oil derivatives diffusion in the soil lead to soil contamination and changes the physical and mechanical properties of the soil specially clay soils. Soil stabilization by using new material is very important for geotechnical engineers in order to improve the engineering properties of the soil. The main subjects of this research are a- to investigate the effect of the cement and epoxy resin mixtures on the stabilization and on the mechanical parameters as well as the microstructural properties of clay soils contaminated with gasoline and kerosene, b- study on the phenomenon of clay concrete development. Practical engineering indexes such as Unconfined Compressive Strength (UCS), elastic modulus, toughness, elastic and plastic strains are all obtained during the course of experiments and are used to determine the optimum amount of additives (cement and epoxy resin) to reach a practical stabilization method. Microstructural tests were also conducted on the specimens to study the changes in the nature and texture of the soil. Results obtained indicated that by adding epoxy resin to the contaminated soil specimens, the strength and deformational properties are increased from 100 to 1500 times as that of original soils. Further, the UCS of some stabilized specimens reached 40 MPa which exceeded the strength of normal concrete. It is interesting to note that, in contrast to the normal concrete, the strength and deformational properties of such stabilized specimens (including UCS, toughness and strain at failure) are simultaneously increased which further indicate on suitability and applicability of the current stabilization method. It was also observed that increasing cement additive to the soil has negligible effect on the contaminated soils stabilized by epoxy resin. In addition, the epoxy resin showed a very good and satisfactory workability for the weakest and the most sensitive soils contaminated with oil derivatives.

Keywords: oily contaminant; epoxy resin; kaolinite; mechanical properties; XRD; SEM

1. Introduction

Leakage and pouring of the oil derivatives such as gasoline and kerosene from old and rusty storage tanks, pipeline transport, process facilities and oil derivatives transportation equipment, contaminates the surrounding soils. Low compressive and shear strength, high compressibility, excessive settlements, high swelling and plasticity, erodibility and sensitivity to the environmental condition are some behavior and strength problems of contaminated clayey soil (Meegoda and Ratnaweera 1994, Khamehchiyan et al. 2007, Nazir 2011, Khosravi et al. 2013, Obeta and Eze-uzomaka 2013, Akinwumi et al. 2014). Problematic soils such as soft clay soil and soil contaminated with the oil derivatives which are used generally for civil constructions, need improvement and amelioration to improve their behavior and strength properties (Vichan and Rachan 2013, Yi et al. 2015, Yi et al. 2016, Sukpunya and Jotisankasa 2016). Global concern about enhancement and protection of environmental

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conditions and especially the modification of contaminated soils, has led to improvement and development of geoenvironmental aspects. Various suggestions have been offered by different researchers for the improvement and treatment of the soil contaminated with the oil derivatives. These suggestions include using the soil in the base layer of the roads or as floor covering layer in the car parks after mixing with the stabilizer. The other methods like biological burning, soil washing, absorption, extraction and vacuum separation by centrifuge methods have been advised, accordingly (Ebuehi et al. 2005, Ayotamuno et al. 2006, Greenberg et al. 2007, Burgess 2013). In this research, for the first time, the Deep Mixing Method (DMM) was used for the improvement and treatment of the soil contaminated with oil derivatives and for the clay based concrete formation. This method (DMM) is one of the amelioration methods of soil with high compressibility which is adopted by the geotechnical engineers. This method is carried by developing a column of stiff material based on mixing the binders like cement, lime, fly ash, plaster and other additives of soil through a mixer in order to improve and ameliorate the strength parameters such as to increase the soil bearing capacity (Porbaha 1998, 2002, Sukontasukkul and Jamsawang 2012, Voottipruex and Jamsawang 2014, Hamidi and Marandi 2018). Also, this method stabilizes the deep excavations or high embankments, slopes stabilization and decreases the

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settlement of soft compressive soils (Rubright and Bandimere 2004, Kogbara and Al-Tabbaa 2011, Anagnostopoulos 2015). Among the traditional stabilizer, cement has the most application in the geo-environmental projects because of the low cost, significant improvement of engineering properties of contaminated soil and its easy storage process (Bergado et al. 1999). However, despite these advantages, researchers indicated that the cement (as an additive of the above mentioned method) in the laboratory and site was not able to increase the strength features and the stiffness of the stabilized clay soil, significantly (Kamruzzaman et al. 2000, Horpibiulsuk et al. 2002, Ahnberg et al. 2003, Puppala et al. 2003, Tabbaa 2003, Petchgate et al. 2003, 2004, Wu et al. 2005, Impe and Flores 2006, Lorenzo et al. 2006, Horpibulsk et al. 2011, Pakbaz and Alipour 2012, Khemissa and Mahamedi 2014, Anagnostopoulos 2015). There are also some other factors such as high amount of water or organic compound (Saride et al. 2013), PH level (Yang et al. 2013), the type of clay minerals in the clay soil samples (Murugesan and Rajagopal 2007) and sensitivity of clay soil as the effective factors on the strength of the deep cement soil mixing (Anagnostopoulos 2015), which have to be considered for stabilizing the contaminated clay soil with the cement. Therefore, the concept of solely cement mixing appear to be impractical in many geotechnical engineering projects. It is necessary, therefore, to investigate on the stabilization of oily contaminated clay soils by other materials (complementary additives along with cement) such as chemical additives, resins, or polymer emulsions to overcome the shortcomings resulted from use of solely cement and to effectively improve the strength and stressstrain behavior of such contaminated soils. Some other researchers, in fact, have made comments and suggestions on the use of these materials, but, comprehensive studies are of few (Anagnostopoulos et al. 2003, Al-Khanbashi and Abdalla 2006, Estabragh et al. 2011). The behavior of clay soil contaminated with the oil derivatives is influenced by the type of oil contaminants and their effects on the clay minerals in the soil. Unfortunately, no comprehensive study has been carried on the effect of type of oil contaminant on the properties of contaminated clay soil containing epoxy resin. The type of oil derivatives and the type of mineral clay has great influence on diffuse double layer. The polymerization reaction was affected by cations in diffuse double layer and the epoxy resin structural components. Therefore, the performance of improvement and treatment of contaminated clay soil properties is related to the type of oil derivatives and the type of clay mineral in soil. So, in this study the cement and epoxy resin additives were used to stabilize the clay soil contaminated with the different oil materials. Epoxy resin consists of two parts: Epoxy (E) and Hardener (H). Epoxy was based on Diglycidyl Ether of Bisphenol. Hardener is a kind of Amine which can form a 3D molecular structure after being mixed with a definite proportion of epoxy. By mixing the epoxy with the optimum amount of hardener, a chemical reaction is performed and the liquid starts hardening till converting to a solid body, completely. However, by using incorrect proportion of epoxy to hardener, some parts of resin or

hardener may not participate in the reaction and remain without any visible changes, which reduce the strength and technical quality of production. High compressive and tensile strength of epoxy resin enable it to perfectly resist against the mechanical stress absorption due to its molecular structure. Some other special properties of epoxy resin are high fatigue strength, high resistance against chemical and corrosive materials (acids, bases, and lipids), being an electrical insulator and having excellent lifetime. The results of mixing the contaminated clay with cement and epoxy resin revealed a significant increase as about 30-300 times in the unconfined compressive strength (UCS) of the stabilized soil. In some specimens, the UCS reached up to 40 MPa which is 1.5 times the compressive strength of a normal concrete. More importantly, it has also been observed that increase in UCS caused a significant increase in ductility of the specimens including the parameters of failure strain and ultimate strain and material toughness. Also, in order to investigate into the microstructural studies and the chemical reactions during stabilization process, X-Ray Diffraction (XRD) analysis and Scanning Electron Microscope (SEM) tests were performed.

2. Materials and methods

2.1 Materials and experiments

In general, a series of tests were conducted on the clay soil specimens contaminated with oil derivatives such as gasoline and kerosene. The clay soil specimens were completely saturated with the contaminants. Then various amount of cement (0%, 5%, 10% and 20%) proportional to total dry mass was added to the contaminated clay soil. The water was subsequently replaced with the epoxy resin solution and then the mixture was compacted with a fixed energy. The stabilized specimens of contaminated soil were tested after 7 and 28 days of curing. In this regard, 700 specimens of contaminated soil stabilized with epoxy resin and cement were tested and the results obtained were analyzed and discussed in this study. The clay soil used in

Table 1 Geotechnical and geo-environmental properties of clay soil

5							
Parameter	Measured quantity						
Sand (%)	4						
Silt (%)	38						
Clay (%)	58						
Gs	2.75						
Organic materials (%)	1 8.79						
PH							
Liquid limit (%)	36						
Plastic limit(%)	20						
Plasticity index(%)	16						
Classification (USCS)	CL						
XRD analysis	Kaolinite, quartz, carbonate, calcite						

Table 2 Chemical composition of the type II cement

Constituents	(%)
SiO ₂	22.2
Al_2O_3	5.13
Fe ₂ O ₃	3.90
CaO	61.39
MgO	1.65
K ₂ O	0.81
Na ₂ O	0.65
Total alkaline	0.97
Free Lime	0.82
SO ₃	2.41
LOI	1.15
Insoluble residue	0.43

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ANALYSIS	LIMIT
Density @ 15°C	0.7 gr/cm^3
10% Evaporated(max)	65 °c
50% Evaporated(max)	115 °c
90% Evaporated(max)	180 °c
Metallic lead(max)	0.013 gr/Lit
Mercaptan content(max)	5 ppm
Sulphur Total (max)	1% wt%
Color	Red

Table 4 Physical and chemical properties of kerosene

ANALYSIS	LIMIT						
Density @ 15 °c	0.81 gr/cm ³						
Flash point (min)	41°C						
Auto-ignition temperature	220°C						
Smoke point (min)	25 mm						
final boiling point	275°C						
Sulphur Total (max)	2% wt%						
Color	Colorless, lacking any disagreeable						

the experiments called super zenouz kaolinite was taken from a large area in the west-north of Iran. Some geotechnical parameters of the clay soil such as Specific gravity, particle-size distribution, Atterberg limits and water content were obtained in experiments based on the ASTM method and results are summarized in Table 1 (ASTM 1994). Type II cement, was adopted for experiments and its chemical properties are reported in Table 2. Physical and chemical properties of gasoline and kerosene were taken from National Iranian Oil Refining and Distribution Company (NIODC) in city of Abadan which are presented in Tables 3-4. XRD analysis was also performed according to the suggested method by Ouhadi and Young (2003). Philips X-Ray diffraction model PW1370 was used in the course of experiments with the features listed in Table 5. SEM analysis instrument in this research was conducted by Table 5 X-ray diffraction instrument features

Start Position	4.15(°2Th)						
End Position	80(°2Th)						
Step Size	0.05(°2Th)						
Scan Step Time	1(s)						
Scan Type	Pre-set time						
Offset	0(°2Th)						
Divergence Slit Type	Fixed						
Divergence Slit Size	2(°)						
Specimen Length	10(mm)						
Receiving Slit Size	0.1(mm)						
Measurement Temperature	25(°c)						
Anode Material	Cu						
K-Alpha1	1.5406(Å)						
Generator Settings	0 mA, 0 kV						
Goniometer Radius	173(mm)						
Dist. Focus Diverge. Slit	91(mm)						

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Abbreviation for identification of the specimen	Contamination material	Sample content	Stabilizing material
CCG	Gasoline	Clay-gasoline	-
CCK	Kerosene	Clay-kerosene	-
SC	-	Clay	Stabilized by cement(5,10 and 20% cement)
SE	-	Clay	Stabilized by Epoxy resin(0% cement)
SCE	-	Clay	Stabilized by cement and Epoxy resin
CCG-SC	Gasoline	Clay-gasoline	Stabilized by cement(5,10 and 20% cement)
CCG-SE	Gasoline	Clay-gasoline	Stabilized by Epoxy resin(0% cement)
CCG-SCE	Gasoline	Clay-gasoline	Stabilized by cement and Epoxy resin
CCK-SC	Kerosene	Clay-kerosene	Stabilized by cement(5,10 and 20% cement)
CCK-SE	Kerosene	Clay-kerosene	Stabilized by Epoxy resin(0% cement)
CCK-SCE	Kerosene	Clay-kerosene	Stabilized by cement and Epoxy resin

SEM-Joel-JSM 840 A. The epoxy resin used here was a commercial product widely produced in National Iranian Petrochemical Company (NIPC). According to the advice of manufacturer, the optimum mixture proportion based on the weight of E and H was: E:H=2:1. The most important risk related to the epoxy resin usage, was the probability of developing allergy to the hardener which might cause an

allergic reaction during the course of investigations (Anagnostopoulos 2015). Therefore, preventive measures should be taken in order to avoid the consequences of allergies. It is to be mentioned, however, that the epoxy resin is completely safe after full hardening.

2.2 Specimen preparation

To contaminate the soil, a sample with 200% soil dry weight were combined with either gasoline or kerosene and reached full saturation. The contaminated soil samples were kept in a closed container for one month. The container was shaken every 24 hours to make the mixture homogenous and uniform. One month duration was considered in this research simply for the perfect mixing of clay soil and oil derivatives. After one month, the contaminated soil samples were put in the oven (40 oC temperature) to lose its moisture, and to dry out completely. Then the samples were mixed with the different percentages of cement in dry condition to reach a homogeneous mixture. In the contaminated clay-cement-epoxy resin mixture, the water was replaced by the epoxy resin. Therefore, the optimum amount of epoxy resin for each soil samples was equal to the amount of water added to the dry mixture of cementcontaminated clay. In this regard, the amount of epoxy resin corresponding to the maximum amount of UCS (after 7 and 28 days of curing) was assumed as optimum amount. In this study, epoxy and hardener (E/H=2) was combined with an electric mixer for 4 min to form a homogeneous white compound. Then the optimum amount of epoxy resin was added to each dry sample of contaminated clay-cement and was mixed in accordance with ASTM C 938-16 for 6 min (ASTM, 2016). A three-edge blades mixer with the specifications advised in ASTM C 938-97 was used (ASTM 1997). Ultimately, mixed materials were poured in the cylindrical mold and were compacted with a fixed energy. The proportion of cement contents to total dry weight was considered as 0, 5, 10 and 20%. With regard to the undeniable effect of sample preparation method on the test results and also in order to investigate the effect of epoxy resin on the stabilized soil compaction, all specimens were prepared similarly and were compacted with the fixed energy method equal to the energy suggested by the standard Proctor test (0.055 Kg.m/cm3). The uniaxial test was carried with the universal equipment model ZWICK available at Shahid Bahonar University of Kerman (Iran). The universal equipment is a heavy loading machine which can record the displacement with the accuracy of 0.01 mmwhich is equal to 0.00014 axial strains. Such accuracy in recording displacements appeared to be acceptable for most of engineering applications especially for calculation of strength properties and evaluation of stress-strain behavior of specimens (such as elastic modulus and materials toughness). Since the variety and number of specimens tested in this study are relatively large, they are each attributed an identification code and their name are abbreviated by capital letters corresponding to their content and stabilization methods. These are all summarized in Table 6.

2.3 Parametric study

In most of the recent studies, the efficiency of stabilization method has been evaluated only with reference to UCS (the Unconfined Compressive Strength). A comprehensive study on stabilization efficiency, however, needs to investigate on the effect of the stabilization method and stabilizing material on some more parameters such as elastic modulus, failure strain and material toughness which have been all taken into consideration in this study. The elastic modulus is an indicator to describe the stiffness of solids which is equal to the slope of stress-strain curve in the elastic deformation area. The toughness is the amount of mechanical energy absorbed in the loaded solid before failure point which is equal to the area under the stressstrain curve. Also, in this study, the strain related to the UCS is called as failure strain (\mathcal{E}_f). Moreover, due to the significance of microstructural reactions and their effects on soil strength and deformation properties, the microstructural studies were conducted by XRD analyses and SEM images.

3. Results and discussion

3.1 Uniaxial test results

3.1.1 Uniaxial test on CCG sample (clay soil specimens contaminated with gasoline)

The strain-stress curves for CCG, CCG-SC, CCG-SE, CCG-SCE after 7 days and 28 days of curing are illustrated in Figs. 1(a)-(d). The stress-strain curves of CCG-SC after 7 and 28 days of curing are shown in Fig. 1(a) and 1(b) and those related to the CCG-SCE mixed specimens were illustrated in Fig. 1(c) and 1(d), respectively. As observed in Fig. 1(c) and 1(d), CCG-SCE mixed specimens are able to sustain more than 20% axial strain without any prefailure cracks. From Fig.1(c) and 1(d), it is also found that the UCS and the failure strain have increased from 20 to 390 times and 5 to 25 times compared to the CCG-SC, respectively. The most important point to note is that the minimum amount of UCS (31.8 MPa) for CCG-SCE mixed specimens reached more than the compressive strength of a normal concrete. It is also noticeable that the UCS of some specimens reached to 39.9 MPa which is comparable with the high strength concrete. Furthermore, the failure strain for various specimens was evaluated 10 to 80 times more than failure strain of the concrete. Also, the dry mass density (0.9 gr/cm^3) and wet mass density (1.5 gr/cm^3) of the CCG-SCE mixed specimens are 35% and 65% lower than the bulk density of fresh concrete, respectively. Therefore, the CCG-SCE mixed specimens are lighter, more ductile and stronger than the normal concretes. Therefore, the mixture results in specimens that are both higher in strength and more cost effective.

The increase in cement percentage for the CCG-SCE mixed specimens had no noticeable effect on ductility but it reduced the strength properties of these specimens. Probably by replacing water with epoxy resin to the CCG-SCE mixed specimens, the pozzolanic reaction could not proceed completely. Also, it was found that the effects of adding epoxy resin in the presence of cement was not successful for CCG-SCE mixed specimens. Therefore, the optimum percent of cement for the CCG-SCE mixed



Fig. 1 The stress-strain curves of the contaminated specimens stabilized with cement and epoxy resin additives obtained from uniaxial tests; (a) 7-day cement-stabilized specimens, (b) 28-day cement-stabilized specimens, (c) 7-day cement-epoxy resin stabilized specimens and (d) 28-day cement-epoxy resin stabilized specimens



Fig. 2 (a) The UCS and (b) The failure strain; results in the contaminated specimens stabilized with cement and epoxy resin additives

specimens was estimated at 0%. Also, the failure strain for the CCG-SCE mixed specimens increased without developing any cracks in the absence of cement.

The UCS and strain value at the ultimate stress in the absence or presence of epoxy resin with various percentage of cement was demonstrated in Fig. 2(a) and 2(b). As can be observed, increasing cement percentage in the CCG-SC, causes the UCS value to increase. As illustrated in Fig. 2(a), the UCS values obtained at 7 days for the CCG-SC with 5, 10 and 20% cement percentage were 1.9, 4.8 and 10.1 timeshigher than those observed for the CCG, respectively.

These results for the same specimens at 28 days revealed an increase of 2.6, 6.1 and 15.5 times, respectively. The UCS of the CCG-SC containing 10% cement after 7 and 28 days of curing were about 2.5 times higher than those of the CCG-SC with 5% cement content. Therefore, the optimum amount of cement for stabilizing the CCG was 10%. The growth rate of UCS after 7 days of curing in the CCG-SC with 5, 10 and 20% cement content were about 73%, 75% and 65%, respectively in comparison with UCS of those specimens cured for of 28 days which showed advancement pozzolanic reaction in the primary days.



Fig. 3 (a) The toughness and (b) The elastic modulus; contaminated specimens stabilized with cement and epoxy resin additives



Fig. 4 The stress-strain curves of the contaminated specimens stabilized with cement and epoxy resin additives obtained from uniaxial tests; (a) 7-day cement-stabilized specimens, (b) 28-day cement-stabilized specimens, (c) 7-day cement-epoxy resin stabilized specimens and (d) 28-day cement-epoxy resin stabilized specimens

By replacing water with epoxy resin, UCS of the CCG-SCE mixed specimens (0, 5, 10 and 20% cement) were about 320, 171, 70 and 30 times higher than those CCG-SC having the same cement percent after 7 days of curing, respectively. Also, for the same specimens after 28 days of curing, it was observed an increase of about 383, 138, 57 and 21 times, respectively. Additionally, it was noticed that by increasing the cement content (5, 10, 20%) to the CCG-SCE, the UCS values observed after 7 days was 1.05, 1.09 and 0.99 times and after 28 days was 0.94, 0.9 and 0.84 times as those of the CCG-SE, respectively. However, considering the long term effect of mechanical properties of

soils on the design and analysis of geotechnical problems (such as soils' bearing capacity), one may rationally come to the conclusion that the optimum curing time for the specimens (mentioned above) could be 28 day having 0% cement. The UCS of all CCG-SCE was more than 30 MPa after 7 and 28 days which is more than the compressive strength of a normal concrete (25 MPa). The maximum amount of UCS was 39.9 MPa after 28 days which was related to the CCG-SE. Therefore, it can be called clay concrete mixture (Hamidi and Marandi 2017, 2018). As mentioned earlier, to carry a comprehensive study, the effect of other factors such as toughness, elastic strain and elastic

modulus on the strength of the stabilized specimens were analyzed. Fig. 2(b) illustrated the elastic strain for different values of percentage of cement. It can be noted that, a decrease in the elastic strain of the CCG was observed. Increasing cement content in the CCG-SC, leads to the failure strain to decrease. The highest reduction observed was about 45% which was related to specimens having 20% cement content. Also, by replacing water with epoxy resin, a significant increase was observed in the failure strain. The failure strain for different content of cement (0, 5, 10, and 20% cement), respectively. Results for the same specimens cured for 28 days revealed an increase of about 0.92, 1.3 and 1.15 times, respectively. Therefore, maximum toughness ratios observed were about 1.2 and 1.3 times for 7 and 28 days of curing respectively and were related all to the specimens having 10% cement in the mixture. The variation of the elastic modulus for different percentage of cement was demonstrated in Fig. 3(b). Generally, by replacing water with epoxy resin, the elastic modulus in the CCG-SCE mixed specimens was increased and it was decreased by increasing the cement content. However, this reduction was not observed for the CCG-SCE mixed specimens with 10% cement. The elastic modulus obtained at 7 days of curing for the CCG-SCE mixed specimens were 420.2, 140.5, 36.8 and 6.1 times higher than those observed for the CCG-SC having the same cement percent as those mentioned above (0, 5, 10 and 20%), respectively. These results for the same specimens cured for 28 days showed an increase of 706.9, 160.3, 39 and 17 times, respectively. The highest increase in the ratio of the elastic modulus in CCG-SCE mixed specimens was related to the CCG-SE (0% cement) and were about 420 and 700 times for 7 and 28 days of curing, respectively. However, maximum amount of the elastic modulus observed was 1549 MPa for 28 days of curing and was related to the CCG-SCE mixed specimens having 10% cement. The elastic modulus in the CCG-SCE mixed specimens (having 5, 10 and 20% cement) after of 7 days of curing were about 32, 42, and 57% less than those of the CCG-SE (0% cement), respectively. Also, these results for the CCG-SCE (having 5 and 20% cement) after 28 days of curing showed a reduction of 23% and 29% and an increase of 9% for the CCG-SCE (having10% cement), respectively. Therefore, it can be inferred (from the discussion on the results given above) that by progress in the pozzolanic and polymerization reactions during the time, the elastic modulus increased and by increasing the cement content (except at 10%), it was decreases. Thus, with regard to the results obtained for the elastic modulus, the optimum percentage of cement is considered as approximately as 10%.

3.1.2 Uniaxial test results on CCK sample (clay soil specimens contaminated with kerosene)

In Fig. 4(a)-4(d), the results of uniaxial tests performed on the stabilized specimens contaminated by kerosene were shown. The stress-strain curves for CCK-SC at 7 days and 28 days were illustrated in Fig. 4(a) and 4(b). Stress-strain curves for the CCK-SCE mixed specimens after 7 and 28 days of curing were also demonstrated in Fig. 4(c) and 4(d). A brittle failure behavior was observed in the CCK-SCE mixed specimens. The failure strain was less than 8% and a sharp drop has occurred in the stress-strain curves after the peak point of the normal stress. Furthermore, the UCS and failure strain of CCK-SCE mixed specimens have both increased from 19 to 338 times and 3 to 5 times compared to the CCK-SC, respectively. Based on the results presented and the discussion give above, it can be inferred that the minimum strength (UCS) observed for CCK-SCE was higher than those of a normal concrete. Furthermore, the failure strain for different specimens, mentioned above, was evaluated about 5 to 15 times higher than those for normal concrete. Also, the dry mass density (1 gr/cm^3) and the wet mass density (1.6 gr/cm^3) of the CCK-SCE mixed specimens were approximately 40% and 65% less than the bulk density of fresh concrete, respectively.

In Fig. 5(a), variations of UCS for various cement content in the CCK-SC and the CCK-SCE mixed specimens were illustrated. An increase in UCS of the CCK-SC was observed by the increase in cement percentage. By increasing the cement percent (5, 10 and 20%), the UCS for the CCK-SC after 7 days of curing were 2.2, 3.8 and 11 times higher than those of the CCK samples, respectively. Also, the results for same specimens after 28 days of curing showed an increase of 3.6, 6 and 14.9 times, respectively. Furthermore, the UCS of the CCK-SC containing 20% cement, after 7 and 28 days of curing, were roughly 2.9 and 2.5 times higher than those of the CCK-SC having 10% cement, respectively. Therefore, the optimum percentage of cement for the stabilization of the CCK by cement was considered as 20% cement. Investigation on the growth rate of UCS in the CCK-SC (having 5, 10 and 20% cement) cured for 7 days showed 61, 63 and 73%, growth rates respectively compared with those specimens cured for 28 days (having the same cement percent) which showed the significance of the proceeding pozzolanic reaction in the primary days. Therefore, the maximum growth rate of UCS observed was related to the specimens having 20 % cement. Also, by replacing water with epoxy resin, the UCS obtained for the CCK-SCE mixed specimens (0, 5, 10 and 20% cement) after 7 days of curing were about 338, 156, 94 and 30 times higher than those for the CCK-SC having the same cement percent, respectively. These results for same specimens cured for 28 days revealed an increase of about 326, 90, 58 and 19 times, respectively. Therefore, the UCS for CCK-SCE mixed specimens (except in 0% cement content) were 20 to 150 times higher than the UCS for CCK-SC. Furthermore, the UCS of CCK-SE was about 330 times higher than the UCS for CCK samples.

As can be inferred in Fig. 5(a), despite progress in the polymerization reactions in epoxy resin and pozzolanic reactions in the cement during the time, an important point could be noticed as the reduction in the UCS of CCK-SCE mixed specimens after 28 days of curing compared to the UCS of the same specimens after 7 days of curing. The UCS of CCK-SCE mixed specimens (0, 5, 10, 20% cement) after 28 days of curing compared to 7 days of curing was defined as 96, 95, 97 and 85%. Probably, the presence of kerosene and completion of the pozzolanic reactions in cement prevented further advancement of the polymerization reactions in epoxy resin which is necessary



Fig. 5 (a) The UCS and (b) The failure strain; results in the contaminated specimens stabilized with cement and epoxy resin additives



Fig. 6 (a) The Toughness and (b) The elastic modulus; contaminated specimens stabilized with cement and epoxy resin additives

for solidification process of the contaminated soil with cement. Also, the UCS of the CCK-SCE mixed specimens for less than 10% cement content was increased. It was noticeable that the influential factors, leading to decrease in the strength in presence of cement, were evaluated by the microstructural investigations such as X-ray diffraction analyses and SEM Images.

As demonstrated in Fig. 5(a), the UCS of CCK-SCE mixed specimens (5, 10 and 20% cement) after 7 days of curing were 1.02, 1.05 and 0.97 times higher than those observed for the CCK-SE (0% cement). The results for the same specimens after 28 days of curing showed an increase of 1.005, 1.05 and 0.85 times, respectively. Therefore, the optimum amount of cement in CCK-SCE mixed specimens was 10%. Also, the UCS of CCK-SCE mixed specimens content 10% cement after 7 and 28 days of curing were more than 36 and 35 MPa, respectively and was more than the compressive strength of a normal concrete. In Fig. 5(b), the variation elastic strain against the cement percentage were shown. As can be deduced, increasing cement content in the CCK-SC, causes the elastic strain to reduce and that the largest reduction (about 65%) is related to the CCK-SC with 20% cement. Elastic strain of CCK-SCE mixed specimens (5, 10 and 20%) cured for 7 days were 2.1, 3.07 and 2.77 times higher than those of the CCK-SC with the same amount of cement, respectively. Result for the same

specimens after 28 days of curing revealed an increase of 1.24, 2.55 and 4.26 times, respectively.

In Fig. 6(a), the toughness variations were illustrated for the different percent of cement. By replacing water with epoxy resin, the toughness obtained after 28 days of curing for the CCK-SCE mixed specimens (contained different cement percent) was decreased compared with those specimens cured for 7 days which showed a similar trend in the changes in the UCS curves. Therefore, it can be inferred that the completion of polymerization reactions in the epoxy resin for solidification process were likely prevented by the pozzolanic reactions of cement. It can be first deduced that the optimum curing time for CCK-SCE mixed specimens cloud be considered as 7 days having best cement content as 10%. As it was discussed for UCS (considering long term effect of mechanical properties), the optimum percent of cement content for the specimens (mentioned above) was noted to be 5% despite the fact that the peak toughness for these specimens observed at 10% for 7 days curing. A significant increase was observed in the toughness of CCK-SCE mixed specimens compared with the CCK-SC specimens with the same amount of cement (0, 5, 10 and)20%). The toughness values of both group of mixed specimens (compared to each other) were 794.2, 408.6, 397.6 and 70.8 times after 7 days and 740.95, 145.56, 132.42 and 38.34 times after 28 days. Therefore, by



(a) Soil contaminated with gasoline(CCG)





(b) Soil contaminated with gasoline+10% Cement(CCG-SC)



(c) Soil contaminated with gasoline + ER(CCG-SE)
(d) Soil contaminated with gasoline+10%Cement+ER(CCG-SCE)
Fig. 7 SEM images taken from various soil samples contaminated with gasoline studied in the research

replacing water with epoxy resin, the toughness of CCK-SCE mixed specimens after 7 days of curing was increased by 70 up to 750 and after 28 days of curing by 30 up to 700 times. Also, the toughness of CCK-SCE mixed specimens after 28 days compared to 7 days was decreased by 8, 34, 50 and 6%, respectively. The toughness values of the CCK-SE (0% cement) ratio to CCK-SCE mixed specimens (5, 10 and 20% cement) were 0.68, 0.61 and 1.2 times after 7 days and 0.96, 1.14 and 1.23 times after 28 days. The results shown in toughness curves revealed that by adding epoxy resin to the CCK samples contained different amount of cement, the toughness of specimens increased from 2.4 to 5.16 MPa.

In Fig. 6(b), the variations of elastic modulus against cement contents for different percent of cement were shown. An increase in the elastic modulus of the CCK-SC was observed due to increase of the cement percent. Also, the elastic modulus of CCK-SCE mixed specimens compared to the CCK-SC with the same amount of cement (0, 5, 10 and 20%) was 580.7, 189.5, 28.1 and 25.6 times after 7 days and 635.5, 239.5, 42.97 and 14.68 times after 28 days, respectively. The most increase ratio in elastic modulus was about 580 times after 7 days of curing and 680 times after 28 days of curing. The elastic modulus of the CCK-SCE mixed specimens with 10% cement, after 28 days reached to the maximum amount of 1903.7 MPa. After 7 days of curing, the elastic modulus of the CCK-SCE mixed specimens (5 and 10% cement), compared to the CCK-SE (0% cement) decreased by 7% and 42%, respectively but it was increased about 27% for specimens contained 20% cements. After 28 days of curing, the elastic modulus of specimens contained 5 and 10% cements increased by 24% and 49%, respectively whereas it has decreased to approximately 32% for specimens contained 20% cements. So, by developing the pozzolanic reactions in cement and polymerization reactions in epoxy resin during the time, an increase in elastic modulus was observed. Also, for specimens contained 20% cements, the solidification process of polymerization reactions in epoxy resin is prevented by the pozzolanic reactions in cement. This process leads to increase the ductility and the failure strain, as a result the elastic modulus decreased. Therefore, the optimum amount of cement was considered 10%.

3.2 Microstructural studying

3.2.1 SEM images

The interaction process between the contaminated specimens and additives formed a new compound by affecting on the forces between clay flakes and by changing in some microstructural properties of the contaminated clay soil. Therefore, X-ray analysis and SEM images were used to specify the impact of cement and epoxy resin additives and to perform a microstructural studies on CCG-SCE and CCK-SCE mixed specimens. Investigation into the pozzolanic and polymerization reactions on the specimens was also of interest in the current study. In Figs. 7 and 8, SEM images of the CCG and CCK samples are shown.

The results discussed in sections 3.1.1 and 3.1.2 indicated that by replacing water with epoxy resin, the UCS



(a) Soil contaminated with kerosene(CCK)





(b) Soil contaminated with kerosene + 10% Cement(CCK-SC)



(c) Soil contaminated with kerosene + ER(CCK-SE)(d) Soil contaminated with kerosene+10% Cement +ER(CCK-SCE)Fig. 8 SEM images taken from various soil samples contaminated with kerosene studied in the research



Fig. 9 X-Ray diffraction observed for soil contaminated with gasoline stabilized with cement and epoxy resin additives

of CCG-SCE and CCK-SCE mixed specimens increased about 2 times in comparison with the UCS of normal concrete. An increase in ductility parameters was also observed, simultaneously.

Microstructural experiments were, therefore performed on CCG-SCE and CCK-SCE mixed specimens. The SEM images taken form CCG and CCK were illustrated in Figs. 7(a) and 8(a). In these images, flaking and discontinuous structure of contaminated clay soil with numerous cavities are observable. As also illustrated in Figs. 7(b) and 8(b), by adding 10% cements and stabilization of contaminated specimens, some changes occurred in the structure of contaminated soil due to pozzolanic reaction and



Fig. 10 X-Ray diffraction observed for soil contaminated with kerosene stabilized with cement and epoxy resin additives

cementation process. Therefore, significant changes in morphology were observed compared to the CCG and CCK samples such that the structure of soils has changed from mass particles to aggregated structure. Furthermore, the CCG-SE and CCK-SE were shown in Figs.7 (c) and 8(c). Epoxy resin caused some significant changes in contaminated clay structure. Therefore, flaking structure of contaminated clay particles were destroyed and a flocculated structure was formed. Then, a gelatinous bond was presumably formed due to the polymerization reaction. The gelatinous bonding increased the ductility of CCG-SE and CCK-SE. According to the uniaxial tests, the CCG-SE and CCK-SE were able to sustain axial strain more than

Table 7 Ratio of UCS, failure strain, toughness and elastic modulus of the stabilized Kaolinite contaminated with gasoline to the stabilized Kaolinite contaminated with kerosene containing same amount of cement and having the same curing age

Cement (%)	UCS (7)ER	UCS (28)ER	UCS (7)	UCS (28)	ε _f (7)ER	ε _f (28)El	$R\epsilon_{f}$ (7)	ε _f (28)	T (7)ER	T (28)ER	T (7)	T (28)	E (7)ER	E (28)ER	E (7)	E (28)
0	0.91	1.17	0.99	0.99	5.42	2.36	1.01	1.01	2.98	1.97	1.01	1.01	0.72	1.11	1	1
5	0.95	1.1	0.86	0.72	1.89	2.37	1.52	1.06	1.7	1.74	1.43	0.87	0.53	0.7	0.71	1.03
10	0.95	1.01	1.27	1.01	4.45	3.93	1.9	1.19	2.25	2.94	1.82	1.62	0.72	0.81	0.55	0.9
20	0.93	1.17	0.92	1.04	4.37	2.94	1.63	1.6	2.97	2.8	1.23	1.25	0.24	1.12	1.02	1.01

20% without developing any prefailure cracks. In Figs. 7(d) and 8(d), the effect of cement and pozzolanic reactions on polymerization reactions of CCG-SCE and CCK-SCE mixed specimens having 10% cements were conceptually illustrated. In most specimens, the optimum amount of cement was observed to be 10%. An enhancement in the stabilization efficiency of the CCG-SCE and CCK-SCE mixed specimens compared to the CCG-SE and CCK-SE (0% cement) was observed due to producing a more continues structure with less gelatinous coating bond. Also, by increasing the cement content up to 20%, pozzolanic reactions prevented the solidification process of contaminated specimens due to the polymerization reaction, destroyed stabilized soil structure and removed gelatinous bonds. Therefore, the strength and ductility parameters decreased significantly. These results were similarly observed in the analysis of the parameters obtained from stress-strain curves at uniaxial tests. The SEM imagery analysis were compatible with the uniaxial test results.

3.2.2 X-Ray diffraction analyses

The results of X-Ray diffraction experiments on the CCG and CCK samples, with and without additives, are shown in Figs. 9 and 10, respectively. Kaolinite soil consists of mainly kaolinite, calcite, quartz and carbonate minerals. The main peaks intensities related to the kaolinite contaminated with gasoline were kaolinite $(d_{100}=7.2\text{A}^{\circ})$ with 2232 CPS (Counts Per Second), carbonate $(d_{100}=3.57A^{\circ})$ with 2319 CPS and quarts $(d_{100}=4.25A^{\circ})$ and $d_{100} = 3.35 A^{\circ}$) with 2000 and 7190 CPS, respectively. By adding 10% cements to kaolinite contaminated with gasoline, the intensity of main peaks produced (due to the pozzolanic reactions) have decreased to 48% in kaolinite, 51% in carbonate and 19 and 12% in quartz, respectively. Also, by replacing water with epoxy resin leads to decrease in the intensity of main peaks about 62, 63, 7 and 18%, respectively. In this study, it was observed that intensity of main peaks have reduced about 71, 66, 30 and 28% by adding 10% cements and replacing water content with epoxy resin.

By adding 10% cement to kaolinite contaminated with kerosene, the intensity of main peaks of kaolinite $(d_{100}=7.2A^{\circ})$ with 2414 CPS, carbonate $(d_{100}=3.57A^{\circ})$ with 2097 CPS and quartz $(d_{100}=4.25A \text{ and } d_{100}=3.35A^{\circ})$ with 2161 and 7400 CPS have dropped to approximately 56, 43, 21 and 13%, respectively. Moreover, by replacing water with epoxy resin, the intensity of main peaks were decreased about 66, 59, 29 and 24%, respectively. The results revealed that the intensity of main peaks have

reduced to roughly 74, 70, 34 and 40% by adding 10% cements and replacing water with epoxy resin. Therefore, these significant reductions in main peaks intensity of contaminated kaolinite indicated that by adding cement and epoxy resin to the contaminated soil, an acceptable and effective stabilization method of the mixture is reached.

In addition, the C-S-H nanostructure peak intensity $(d_{100}=3.57A^{\circ})$ related to the kaolinite contaminated with gasoline was equal to 382 CPS. By adding 10% cements to these specimens the intensity increased almost 29% and reached to 491 CPS. Also, by replacing water with epoxy resin and adding 10% cements to the kaolinite soil contaminated with gasoline, the C-S-H nanostructure peak intensity increased about 23% and reached to 468 CPS. Moreover, adding 10% cements caused an increase about 46% in C-S-H nanostructure peak intensity (d₁₀₀=3.57Ű) related to the kaolinite contaminated with kerosene from 397 to 581 CPS. Also, in the CCK-SCE mixed specimens with 10% cement, the peak intensity of C-S-H has increased about 6% and reached to 421 CPS. Therefore, it was found that the peak intensity of C-S-H nanostructure of the CCG-SCE and CCK-SCE mixed specimens is decreased when compared with those of CCG-SC and CCK-SC. This is because the solidification process based on polymerization reactions in epoxy resin prevented the completion process of pozzolanic reactions in the cement and, hence, reduced the C-S-H nanostructure peak intensity. The reason is that the polymerization reactions in epoxy resin occurred at the initial hours of the stabilization, while pozzolanic reactions in the cement, prolonged to at least 28 days. Therefore, the high resistance of epoxy resin with hard structure prevented the completion of pozzolanic reaction of cement. The obtained results revealed that the strength parameters and stress-strain curves are in compliance with the SEM imagery and UCS results.

3.3 Influence of gasoline and kerosene contaminants in kaolinite clay mineral on the results

In Table 7, quantitative evaluation of the effect of contaminant type in the kaolinite soil on the stabilization performance and in presence of epoxy resin (assuming the same amount of cement and the curing age) is briefly illustrated. From Table 7, the ratio of UCS, failure strain, toughness, and elastic modulus related to the CCG samples to those of the same parameters in the CCK samples are given. The ER symbol stands for the specimens contained epoxy resin and the suffices 7 and 28 numbers denote 7 days and 28 days of curing. Furthermore, c, ε_f , T and E

represent failure strain, toughness and elastic modulus, respectively. According to the results shown in Table 7, in the specimens stabilized with various cement content, the ratio of UCS, ε_{f} , T and E of CCG samples to those of the same parameters in the CCK samples was in the range of 0.5 to 2. It was also observed that the effect of stabilization by cement on the CCG samples was more than the CCK samples and additionally, significant improvement in the strength parameters has occurred after 28 days of curing. By replacing water with epoxy resin, the ratio of UCS of CCG samples to those the same the CCK samples was in the range of 0.9 to 1.2, demonstrating the likely effect and formation of the clay based concrete (contaminated claycement-epoxy resin mixture). Also, the ratio of failure strain, toughness and elastic modulus of contaminated specimens was in the range of 2 to 5.5, 1.5 to 3 and 0.2 to 1.2, respectively. In addition to the significant improvement in strength, the ductility of the CCG samples has significantly increased in comparison with the CCK samples.

4. Conclusions

In this study, the improvement of clay contaminated with oil derivatives such as gasoline and kerosene stabilized by cement and epoxy resin additives was experimentally studied. Further, the impact of the stabilizers on the mechanical parameters and microstructural properties of soil was investigated. Accordingly, the following conclusions can be drawn from this study:

• In stabilization of the contaminated, poor and sensitive soils, the use of epoxy resin is advantageous since it is environmentally-friendly, workable and saves water.

• In CCK-SCE mixed specimens, the improvement in UCS is more efficient than in the ductility while in CCG-SCE mixed specimens, the UCS and ductility are both improved significantly.

• The problematic uncontaminated and contaminated clay soils such as expansive, dispersive and soft soils can be stabilized using epoxy resin.

• Polymerization reaction caused the strength and toughness of the CCG-SE specimens to increas from 400 to 1500 times in comparison with the CCG samples.

• The strength and ductility of the CCK-SCE mixed specimens (having 10% cement content) increased from 60 to 140 times in comparison with the CCK samples due to the polymerization and pozzolanic reactions.

• It is interesting to note that an overall of 200% to 300% increase in the UCS, toughness and elastic modulus of stabilized soils could be observed in comparison with those of a normal concrete. The rate of increase depends, in fact, to the type of contaminant and of the minerals forming the soil mass.

• Several main peaks representing the mineral structure of contaminated clay was detected in the XRD analysis of the samples (CCG and CCK). On the other hand, in the contaminated clay-cement-epoxy resin mixture (CCG-SCE and CCK-SCE), the reduction in the intensity of peaks related to main minerals of clay and the increase in the intensity of C-S-H nanostructure indicated the success of stabilization process.

• The SEM images illustrated a discontinued, flaky and highly porous structure of clay contaminated with oil derivatives. Also, the SEM images of the contaminated clay-cement-epoxy resin mixture (CCG-SCE and CCK-SCE) indicated more continues and condensed structure with less pores in which the soil particles were coated by a gelatinous layer.

• Using CCG-SCE and CCK-SCE mixed specimens (clay based concrete) for stabilization of contaminated clay soil is extremely desirable because the strength and ductility of stabilized specimens were more than the cement concrete and also were much lighter. It is advised to use clay based concrete through DDM instead of concrete pile for improvement and modification of the contaminated clay soils.

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