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THE EFFECT OF USING FLUID CATALYTIC CRACKING CATALYST RESIDUE (FC3R) "AS A CEMENT REPLACEMENT IN SOFT SOIL STABILISATION"

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ABSTRACT

Construction sector suffer from many problems due to presence of the soft soil in many worlds' parts and to solve these problem the soft soil should be stabilised by either mechanically or chemically; the mechanical ways can be achieved by replacing with stronger materials or using special machines to increase the soil stability result which considered high cost, researchers try to find another method with alternative materials like cement, lime and pozzolanic materials to qualify the soft soil on the civil engineering project. The aim of this study is to evaluate the soft soil properties that cured with 9% binders of various mixtures of binary blended produced from Ordinary Portland cement (OPC) and Fluid catalytic cracking catalyst residue (FC3R), which is a by-produced material from petroleum sector. Geotechnical tests like (compaction, un-confined compressive strength (UCS) test and Scanning electron microscopy (SEM)) were used to investigate the optimum binary mixture. Results show that the use of FC3R as a cement replacement developed the strength of soft soil after 28 days result in a higher strength comparison to using OPC alone in soil stabilisation. SEM proved presence of OPC hydration products during different curing ages.

Keywords: FC3R, physical properties, SEM, soft soil stabilisation, un-confined compressive strength.

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1. INTRODUCTION

Soil stabilisation is a process of improving the soft soil properties (low shear strength, highly compressible and low permeability) and making it suitable for construction. Soil stabilisation could be applied by replacing the soft soil with stronger materials or use special machines to increase the stability, these ways are called mechanical ways which need time and money to achieve the stability, (Kumari, et.al., 2016).

Also, soil stability could be achieved by chemical stabilisation which is an effective technique applied by mixing the soft soil with cementitious materials such as Ordinary Portland Cement (OPC) and lime (traditional materials) to mitigate the soft soil problems by the chemical reaction between stabilisers and soil minerals in water presence condition and the using of traditional materials were proven its effectiveness for tens of years (Makusa, 2012). However, later on many issues appear in the environment due to using the traditional materials in soil stabilisation such as consumption the raw materials and rise CO₂ emission in atmosphere that present almost 9% of all human activities (Jafer et al, 2018). Cement manufacturing consider the biggest consumer for energy and resource. Moreover, this industry play a big role in global warming and climate change because of high CO₂ and other greenhouse gases emission. Also, cement manufacturing produce CO₂, CO, NO₂, SO₂, fluorides, Toxic metals and dust effect badly on human species and it could lead to serious problems in breathing system because of burning, crushers and grinders the clinker of cement (Hurford et.al, 2002 and K. N. Kadhim and Ghufuran 2016).

Nowadays, landfills are considered one of the biggest problems, and the dangerous of landfills will increase in the Industrial wastes case. The Industrial wastes that generated from various industries become a considerable problem and the process of disposal an even bigger issue. Industrial wastes are polluting any environmental where found such as (air, water, soil and groundwater) specially non-biodegradable waste which effect on human and bio-diversity health because of the bad effect of chemical components. Also, landfills considered one of the side effect of industries which effect on the project economy due to high taxes levied by many countries on landfills. So, landfills should reduction to lower amount and that could be achieved by reusing the waste materials in many applications to use economical-alternatives and an eco-friendly resource that produce less waste and reduction its problems (Sen and Mishra, 2010).

The construction materials and all cementitious materials are continuously developed and the researchers try to find alternative materials (supplementary cementitious materials (SCMs)) to reduce the depletion of raw materials as well as reduce the environmental effect of present materials manufacturing. SCMs are many alternative materials like silica fume (SF), palm oil fly ash (POFA), rice husk ash (RHA), ground granulated blast furnace slag (GGBS), pulverized fuel ash (PFA), etc. There are many waste materials used as cementitious materials because these materials have a high contain of calcium or aluminate and silicate; these components in presence of water react to make strong chain like calcium-aluminate hydrated (C-A-H) or calcium-silicate hydrated (C-S-H) which are called the cementitious gel. The gel is main responsible about the soil strength after stabilisation process (Jafar et al., 2015; Modarres and Nosoudy, 2015).

Moreover, in petrol refineries the petrochemical industries generated huge waste amount about (400 thousand tons) as a result of fluidized bed catalytic cracking (FCC) process, this waste called fluid catalytic cracking catalyst residue (FC3R). FC3R was about (200 thousand tons) in 2005, but this amount increased due to increase the petrol using as a fuel. And increasing (FC3R) production will lead to many environmental problems for that reason the scientists around the world try to reuse (FC3R) in many field to reduce the (FC3R) landfills (Antonovič et al., 2010). Many researches show that the different waste materials could be used as cement additive or cement replacement materials in many applications like concrete and soft soil stabilisation (Baghdadi 1990).

The chemical analysis of Fluid catalytic cracking catalyst (FC3R) proven the presence of high amount of Silica and Alumina (same meta-kaolin chemical composition) which play an important role in the pozzolanic reaction during water presence condition to produce same products as cement and lime paste (Payá et al., 2003) [a,b]. And SEM test indicate that the chemical reactions of FC3R produce (calcium-alumina-silicate hydrate) CSH and CAH which are the same products produce from cement hydration with water due to presence the same components as cement ((SiO₂, Al₂O₃ and CH). In low lime pastes experiments FC3R prove higher pozzolanic reactivity than meta-kaolin at early ages.

Dulaimi et al. (2017) indicated that FC3R has very low crystalline peaks of an amorphous nature meaning that it will illustrate high reactivity during the hydration process and can be used as an activator. Also, Dulaimi et al. (2016a) used FC3R to develop a new cold asphalt binder course bituminous emulsion mixture. They indicated that FC3R can activate high calcium fly ash and produce a binary blended cementitious material.

In this research, (FC3R) is used for the first time in stabilisation process of a soft silty-clayey soil. Compaction parameters, unconfined compressive strength and SEM test results were used to evaluate the improvement in the physical and geotechnical properties of the soft soil.

2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Soil sample

The banks of river Alt in High town to the north of Liverpool is the main source of soil which was used in this study. At a depth around (0.3- 0.5m) below the ground surface the soft soil sample was collected according to BS EN ISO 17892-1:2014 (European Committee for Standardization, 2014). After collecting, the soil samples were stored in plastic bag with a capacity of 20 - 25kg. The process of preparing the specimens was started instantly after laboratory arrival to determine the natural moisture content (NMC% = 37.5). Then the remaining soil was dried in the oven at 110°C to prepare the soil for experiments. The particle size distribution testing was performed accordance to BS EN ISO 17892-4:2014. And then other physical and geotechnical properties of the virgin soft soil (untreated soil) such as compaction parameters and unconfined compressive strength tests (UCS) were performed. Moreover, the chemical properties of the soft soil like pH was specified by conducting a chemical analysis. Table 1 identifies the effective physical and chemical properties of soft soil.

Table 1 The geotechnical properties of the virgin soil used in the study

| Property | Value |
|-----------------------------------|-------|
| Liquid Limit, LL % | 39.2 |
| Plasticity Index, PI | 18.35 |
| Sand % | 12.07 |
| Silt % | 75.03 |
| Clay % | 12.9 |
| Specific Gravity (Gs) | 2.61 |
| γ_{dmax} g/cm ³ | 1.63 |
| Optimum moisture content, OMC % | 20 |
| PH | 7.78 |
| Organic matter content % | 7.95 |

2.1.2. Binder materials

The binder materials that used in this research were Ordinary Portland cement (OPC) and Fluid Catalytic-Cracking Catalyst Residue (FC3R). The experiments were performed by using the commercial OPC type CEM-II/A/LL 32.5-N that supplied by CEMEX Company in Warwickshire, UK. While (FC3R) an industrial waste material producing in filtering processes-of petrol in the refinery was used as a SCM to produce binary blending materials. FC3R is considered as a pozzolanic residue, which is a type of inorganic zeolite consists of silica-alumina as a main compound. Figure 1 shows the SEM image of the FC3R used in this study while the XRF analysis results illustrated in Table 2 show the chemical analysis for FC3R and OPC. XRF analysis results indicated that FC3R contains a sufficient amount of Alumina-silicate with other components.

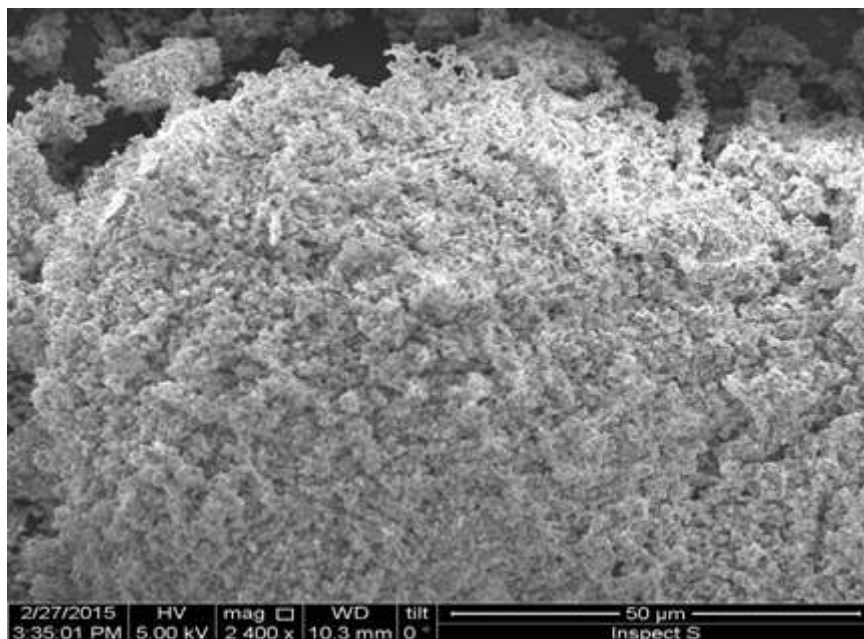


Figure 1 The SEM image for FC3R powder.

Table 2 Chemical analysis of the cement and FC3R

| Material | CaO | SiO ₂ | Al ₂ O ₃ | MgO | Fe ₂ O ₃ | SO ₃ | K ₂ O | TiO ₂ | Na ₂ O |
|----------|-------|------------------|--------------------------------|------|--------------------------------|-----------------|------------------|------------------|-------------------|
| FC3R | 0.05 | 35.45 | 44.17 | 0.69 | 0.37 | 0 | 0.05 | 0 | 0 |
| OPC | 62.34 | 26.64 | 2.44 | 1.57 | 1.75 | 2.59 | 0.72 | 0.39 | 1.53 |

2.2. Methods

2.2.1. Mixing proportions

The soil tests were performed by using binary mixtures (BM) (five of them) with different OPC and FC3R ratios along with untreated soil (virgin soil) and Reference samples (treated with 9% OPC which equivalent to 100% binder); Table 3 shows the proportions of binder mixture.

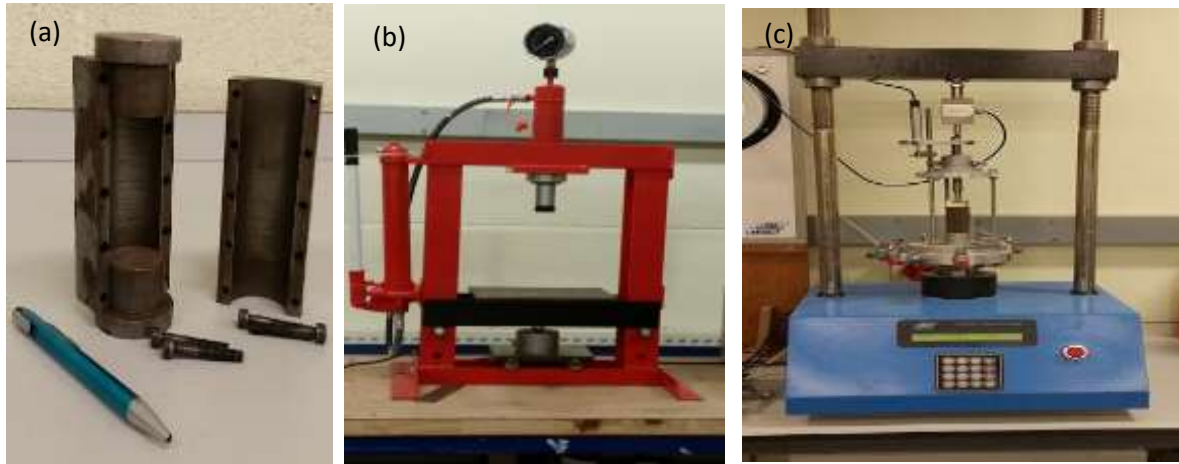
Table 3 Binder mixing proportions used in this study

| Mix ID | OPC % | FC3R % |
|--------|-------|--------|
| VS | 0 | 0 |
| RF | 100 | 0 |
| BM1 | 90 | 10 |
| BM2 | 80 | 20 |
| BM3 | 70 | 30 |
| BM4 | 60 | 40 |
| BM5 | 50 | 50 |

2.2.2. Sample Preparation

After preparing the soil, the soil specimen was grind to break any chunks and sieved to be ready for testing. Experiment was performed instantly after mixing the soil with binder and water according to standard procedure for each test. Soil samples in each test contain 9% binder for different proportions of (OPC+FC3R) by the dry weight of soil. The FC3R percentage range (10-50) % by the dry weight of the total binder. The UCS samples casted by specific mould dimensions (D = 38mm and H = 76mm) as shown in Figure 2-a. The UCS samples consisted of soil, binder and water; and the weight of soil/water depending on the maximum dry density (MDD) and optimum moisture content (OMC) for each binder ratio which had been gotten from compaction test results Firstly, the mixture prepared manually and then formed under pressure in the mould to produce the UCS specimens (Figure 2-b). The specimens of treated soil stored under the lab condition for different curing period (7 and 28) days. The UCS tests were performed on a computerised tri-axial machine by applying vertical load only without a horizontal stress in the tri-axial cell, $\sigma_3 = 0$ as shown in Figure 2-c. The (SEM) test was performed on the sample that record optimum binary ratio during Unconfined compressive strength test (UCS), Reference sample (RF) (soil with 9% OPC) and paste of the optimum binary ratio that consist of (70%OPC+30%FC3R). SEM samples were produced by using same procedure for preparing sample as UCS test with the same curing periods and method. On the other hand, the paste was casting in cylindrical shape by using plastic disposal cup, which stay at the cup for 1 day and then transport to water for curing (all these steps should happen in room temperature). SEM specimens took from centre of the crushed specimens to discover the material particles shape without any changing on samples by either touching or by the curing plastic. And then dry the pieces of the sample, and fix it on the aluminium stubs by using carbon disks which is double-sided glued to be ready for

application on vacuum. A palladium coating was then applied on the pieces, before taking the SEM images, using an auto fine sputter coater to make the images very accurate and clear.



Figures 2 Fixed volume mould, Compression rig, tri-axial machine.

2.3. Laboratory Tests

Test's experiments were performed as following:

2.3.1. *Standard Proctor compaction test*

According to BS 1377-4:1990 (British Standard, 1990a) the test was carry out on 2000g of dry soil to find the maximum dry density (MDD) and optimum moisture content (OMC) for each binder ratio. The electrical compaction machine used to provide 25 blows with a 2.5kg rammer for each layer until complete three layers in a stander mould.

2.3.2. *Un-confined Compressive Strength test (UCS)*

According to BS 1377-7:1990 (British Standard, 1990b) this test had been carried out on four specimens for each binder ratio in two curing periods (7 and 28 days) by covering the sample by cling film and storied at $20 \pm 2^\circ\text{C}$. Each sample prepared by using soil, binder and water, which compacted in the mould under hydraulic load after mixing all materials together.

2.3.3. *Scanning Electron Microscopy Test (SEM)*

This test was carried out to acquire information about the specimens that include extrinsic morphology (texture) and interior microstructure such as crystalline structure and the chemical composition (hydration products). Dulaimi et al. (2017b) indicated that SEM is a technique for high-resolution picture of surfaces. It is used to test the morphology of the object. SEM is a type of electron microscope that depicts the sample's surface by scanning it with a high-energy beam of electrons across the sample's surface. SEM images were collected for the specimens at different ages with the aid of Inspect scanning electron microscope. The sample has around 1mm width, and the magnification ability of the device is about (20X to 30,000X) to access the specimen structure until (10 to 20 nm). The device consists of high vacuum and test voltage ranging from 5 kV to 25 kV as shown in Figure 3. The study carried out by EDX Oxford Inca x-act detector, FEI SEM model Inspect S.



Figure 3 The SEM device with the screen to show the SEM images

3. RESULTS AND DISCUSSION

3.1. Standard Proctor compaction test

Table 4 demonstrates compaction test results, which has clearly changed in the MDD and OMC before and after adding the binder to the soil. The experiments indicate that MDD decreased from 1.58 Mg/m³ to 1.534Mg/m³, while OMC increased from 20% to less than 23% for FC3R (0-50) % from binder weight. The reduction in MDD may participate in reduction in the compressive strength of the sample with decrease of the workability as a result of increasing OMC. Furthermore, the decrement occurred in the dry density may be related to the fineness of FC3R particles which raise the molecules' surface area and increasing the interaction's area.

Table 4 Dry density-moisture content for the soil samples

| Mix ID | MDD(Mg/m ³) | OMC (Mg/m ³) |
|--------|-------------------------|--------------------------|
| VS | 1.63 | 20 |
| RF | 1.58 | 21 |
| BM1 | 1.575 | 21.5 |
| BM2 | 1.565 | 22 |
| BM3 | 1.56 | 22.5 |
| BM4 | 1.55 | 22.5 |
| BM5 | 1.535 | 23 |

3.2. Unconfined Compressive Strength Tests (UCS)

Figure 4 demonstrates the maximum stresses for UCS results in two curing periods (7 and 28 days) for all sample IDs. At 7 days curing, the samples BM1, BM2 and BM3 show slight increments in the compressive strengths when compared with the compressive strength of the reference soil samples (treated with 100% OPC) and the highest increasing was obtained from sample BM3. While the 28 days curing shows a significant increasing in compressive strength values when comparing it with untreated soil. Figure5 shows the changes in the strength of the soft soil after replacing different cement ratios by FC3R. It can be seen that 30%FC3R is

the optimum percentage for replacing OPC in the total binder. A noticeable reduction in the UCS was observed when the FC3R used with a percentage higher than 30% of the total binder.

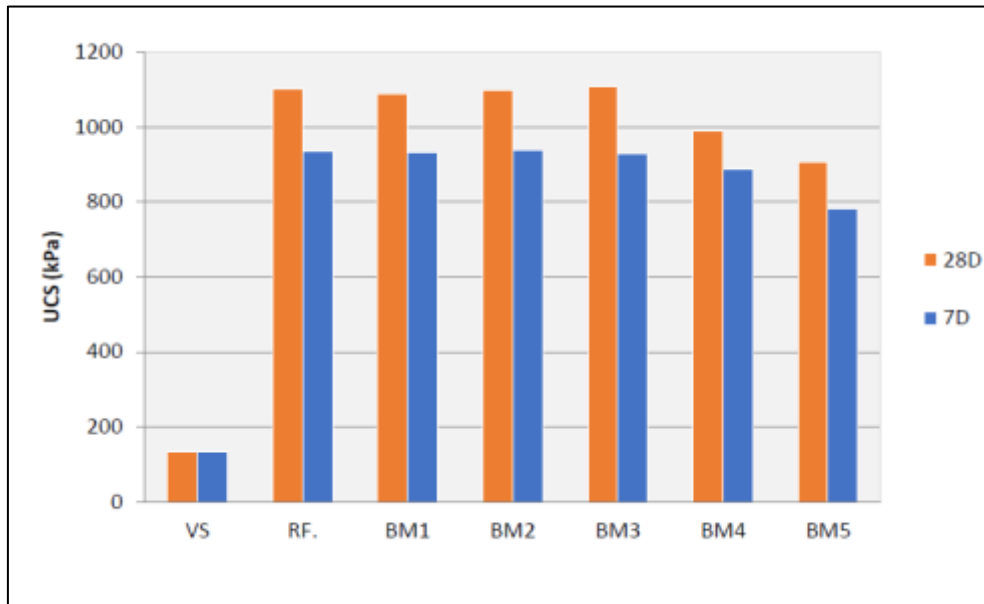


Figure 4 UCS results of the soil samples during different curing period.

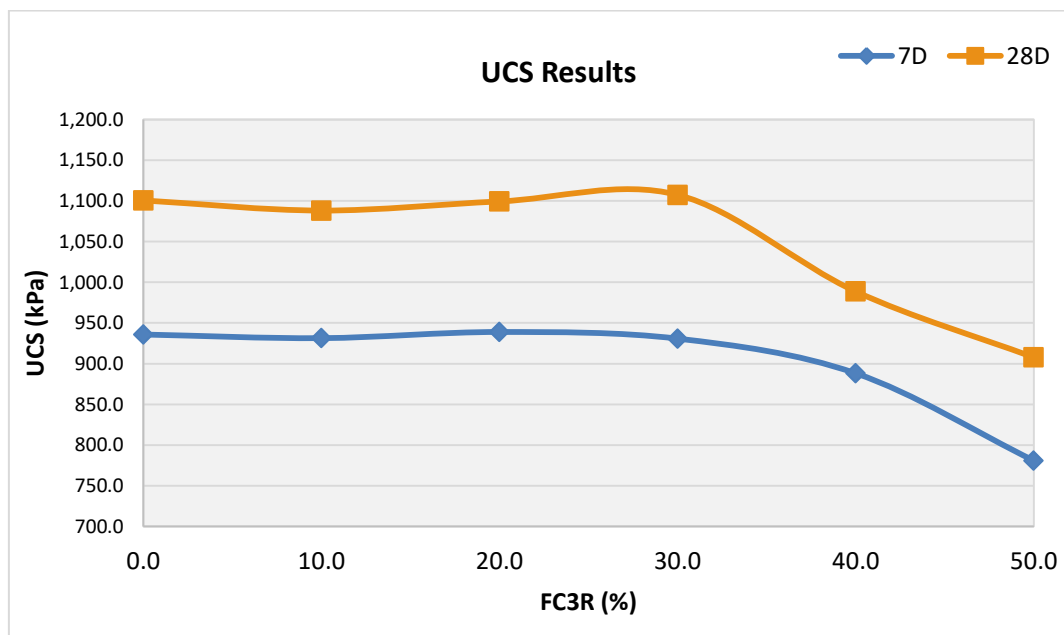


Figure 5 Development of UCS result after replacement OPC by FC3R in soil stabilisation.

3.3. Scanning Electronic Microscopy Test (SEM):

Figure 6 shows the SEM image after 7 days of curing for sample consists of 70% OPC +30% FC3R which shows the availability of considerable amounts of cement hydrates that were produced at the early stage of curing due to reaction of cement and the pozzalanic materials with water. While Figure 7 demonstrates the SEM image after 7 and 28 days of curing for the sample consists of (100%OPC by binder weight + Soil) which shows the formation of cement hydration products such as Ettringite, CH and C-S-H at 7 days curing and the transformation of most of the Ettringite to C-S-H at 28 curing days. Figure 8 illustrates the SEM image after

7 and 28 days of curing for sample consists of (70%OPC+ 30%FC3R (BM3) + Soil) which shows the presence of the same cementitious products for the reference sample.

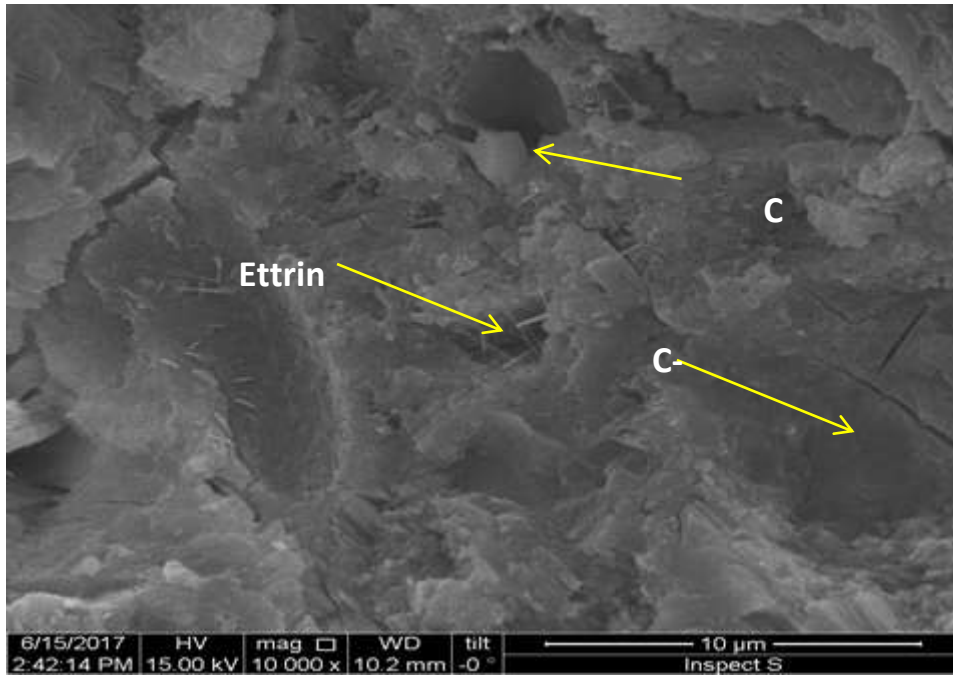
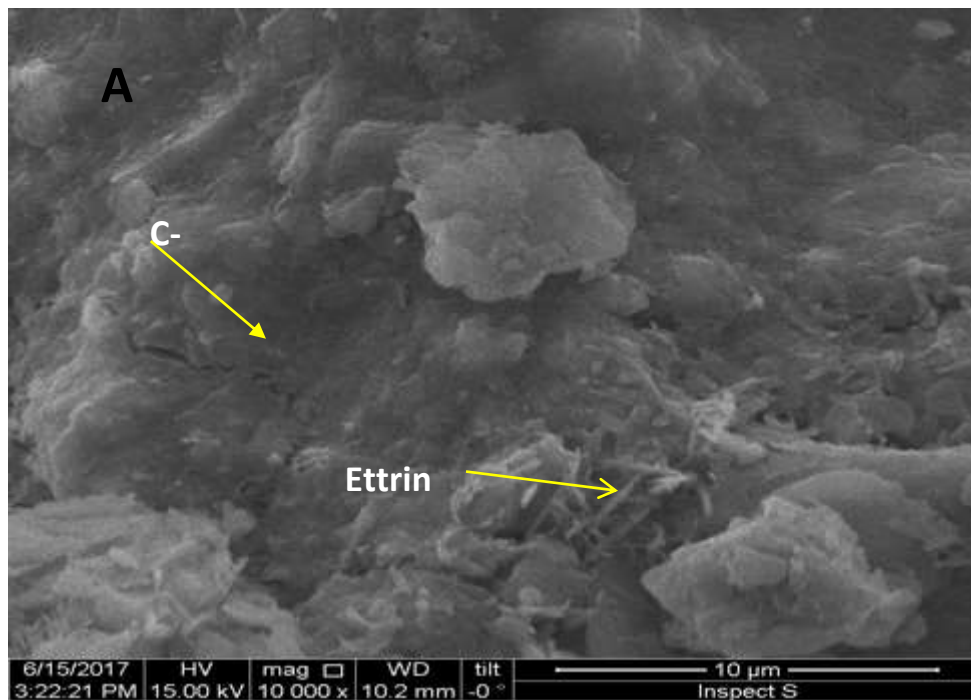


Figure 6 SEM picture for (70%OPC+30%FC3R) paste.



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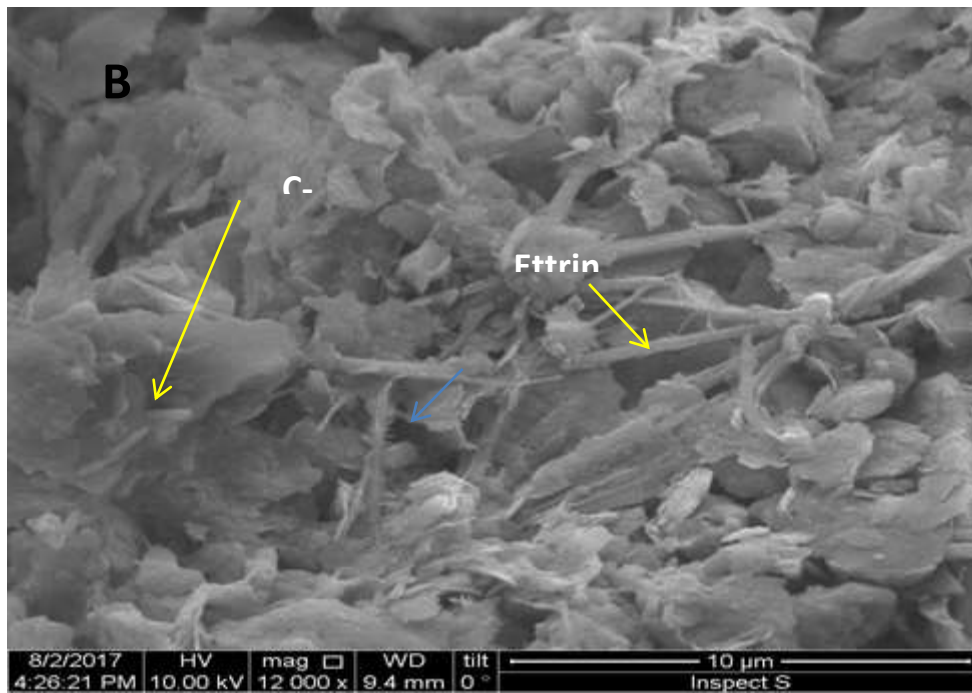
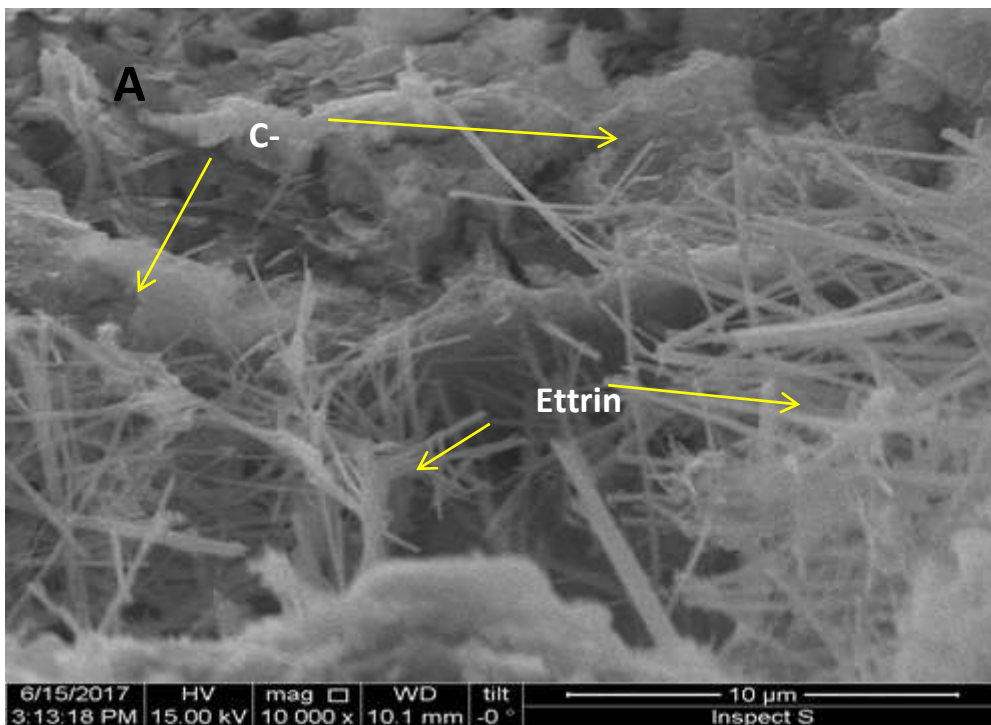


Figure 7 SEM image for RF sample: A) 7 days curing, B) 28 days curing



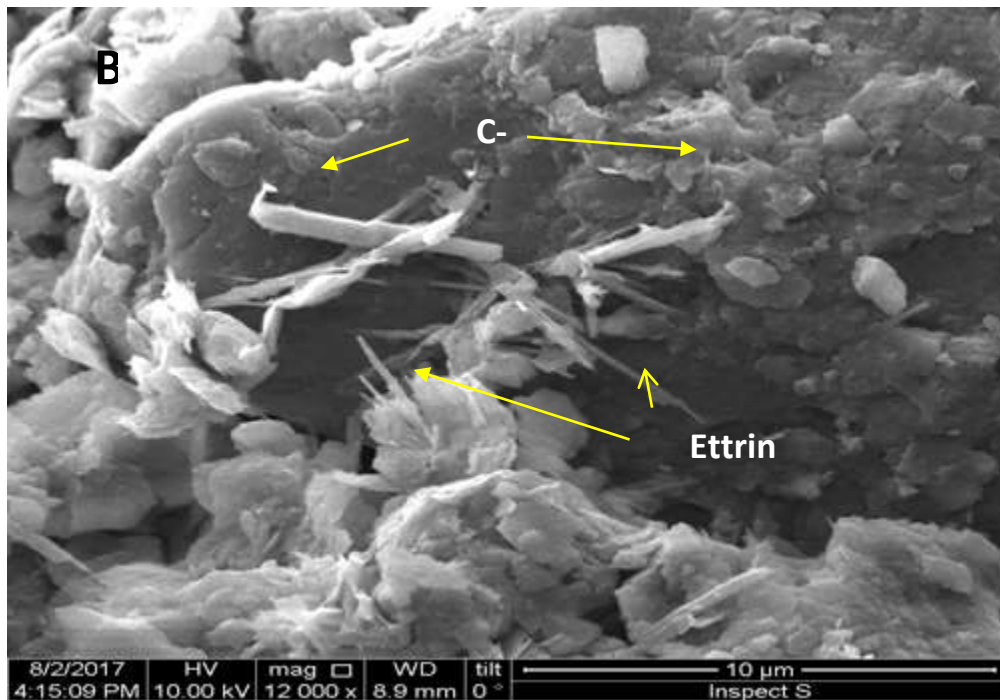


Figure 8 SEM image for Z3 sample: A) 7 days curing, B) 28 days curing.

4. CONCLUSION

To sum up, according to the research results, they led to develop a new binary blended cementitious binder.

In the binary blended system, the mix of (FC3R with OPC) has an effect on the geotechnical properties of soft soil due to pozzolanic reaction for both (FC3R+OPC) and the influence of this mix was determined in this research. This will provide a novel approach in the use of waste materials and develop new cementitious materials for soft soil stabilisation.

In compaction test, the using of FC3R was led to a considerable decrease in the maximum dry density, but it increased the water content. Also, adding FC3R improves the unconfined compressive strength for soil; UCS results indicated a significant increase in soil strength from 134.2kPa for untreated sample to 1107kPa for sample treated with BM3 (70% OPC + 30% FC3R) after 28 days of curing. The improvement percentage for the sample BM3 was eight times higher than VS sample results. UCS results showed that soil samples treated with 9% of BM3 indicated compressive strengths higher than that RF samples. Finally, SEM images for sample contains (30%FC3R from the total binder) showed presence of the same cementitious products that found in sample contain OPC alone as a binder.

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