International Journal of Advances Engineering and Civil Research

Print ISSN 2974-4385

VOLUME 2, ISSUE 1, 2022, 72 - 90.

Online ISSN 2974-4393

Spectroscopic study of collapsing soil microstructure to explore the physical properties afterward incorporating textile wastes

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Abstract

Most countries now have difficulty disposing of industrial waste safely. With the fast growth of textile waste all around the world, it has become critical to discover cost-effective ways to decrease and utilize sludge. Mixing sewage with collapsible soil to improve mechanical qualities is one of the most cost-effective methods in civil applications. The major goal of this research is to highlight the importance of microstructural studies in evaluating the behavior of collapsible soil before and after treatment with textile sludge. The microstructure of three samples was examined using four techniques: collapsible soil, textile sludge, and soil mixed with 24% textile sludge. SEM (scanning electron microscope), EDAX (energy-dispersive X-ray spectroscopy), FTIR (Fourier transform infrared spectroscopy), and X-ray diffraction (XRD) are examples of these methods. According to the findings, the basic cause of soil collapsing is that the structure of soil is kept together by calcium carbonate connections. Stress and saturation cause these connections to weaken and break. Furthermore, the soil's structural surface has many big bracket pores, making the soil more prone to collapse. The findings also indicated the significance of textile sludge in lowering soil collapse potential owing to the aggregate shape of the sludge, the high specific surface area, and the large amount of pozzolanic elements, all of which led to the formation of strong cement connections between soil grains. Upon soil treatment, pores are filled with textile sludge particles, and the soil is transformed from loess to a dense structure.

Keywords: textile sludge; collapse potential; XRD; EDAX; SEM; FTIR

Highlights

Collapsing soil was treated with different percentages of textile sludge
Collapse potential reduced dramatically with the addition of sludge.
Microstructure of treated and untreated soil was investigated
The sludge represents economical stabilizer for collapsing soil

Introduction

At low moisture content, collapsible soil may withstand relatively high loads with tiny settlements. However, following soaking, it will show a decrease in volume and accompanying settling, with no increase in applied stress. Construction on this soil had a catastrophic impact on many structures, such as roads, railways, pavements, channels, buildings, and airports (Schwartz, 1985). As a result, an accurate geotechnical investigation must be carried out before construction to identify the degree of collapsibility, either through field tests or in the laboratory. Furthermore, it is required to investigate the soil microstructure, which is a novel way to predict soil collapsibility based on four factors: grain shape, pore size, grain contact, and bonding materials (Li et al., 2015). Each factor's impact on soil stability is described below:

the soil's particle structure, which might take the form of primary grains or aggregates. Because primary grains predominate and have an open structure, primary grain soil is more susceptible to collapse than soil with aggregates (Li et al., 2015). Second, there is the pore form, which might be mosaic or bracket shaped. Because they allow for particle mobility, bracket pores are more prone to collapse. However, mosaic pores are smaller than the surrounding particles, between interlocked particles, and less prone to collapsing (Xie et al., 2018). The third aspect is the relationship between the particles' points of contact, or faces, depending on the situation. In contrast to the face-cementation relation, which is more permanent and occurs in aggregate-dominant soils, the point-contact connection is more likely to exist in primary grain-dominant soils (Li et al., 2015). The last element is bonding materials. The primary bonding components in collapsible soil are known to be calcium carbonate and clay. Bonds were broken following load and saturation, resulting in soil collapse. According to Liu et al. (2016) and Xie et al. (2018), clay buttresses and bridges were connected by clay bonding. On the other hand, calcium carbonate cementations are challenging to see because of their unique form. However, using XRD, it may be recognized by its mineralogical properties (Xie et al., 2018). Spectroscopic methods, such as X-ray diffraction (XRD) and scanning electron microscopy (SEM), might be used to investigate all of these elements. Additionally, FTIR spectroscopy is an essential device for the investigation of soil samples' mineral and organic contents. Additionally, FTIR spectroscopy may be used to resolve the complexity of low crystallization in XRD analysis (Margenot et al., 2016). The step of selecting the most effective form of soil treatment, which may be mechanical treatment, chemical treatment, or a mix of the two approaches, comes after evaluating the features of collapsible soil through geotechnical testing and microstructure analysis. The following are some of the most current studies that focus on the chemical repair of soil that collapses:

Nazir et al. (2020) evaluated the impact of adding water treatment residue on collapsible soil. They determined that by adding 10% WTR to the soil, the collapse potential was reduced by 24.7%. Nazir et al. (2020) also investigated the influence of marble dust on collapsible soil qualities. They discovered that combining soil with 30% marble dust reduces collapse potential by 64.32%. Abbas et al. (2021) investigated the effect of textile sludge on the geotechnical parameters of collapsible soil. When soil was combined with 24% textile sludge, the collapse potential was reduced by 76.36%. In addition, raising the amount of textile sludge increased soil cohesiveness and liquid limit while decreasing friction angle.

Significance of the work

The goal of this study is to finish our prior work on Abbas et al. (2021) from a different angle. The authors of the published study want to identify a long-lasting and low-cost addition to enhance the mechanical qualities of collapsing soil. The mechanical qualities of the chosen textile wastes are greatly improved. The novel spectroscopic analysis provided in this article demonstrates the causes connected to the microscopic characteristics of the soil and the textile sludge that caused the improvement of soil qualities.

2. Materials

2.1 Collapsible soil

According to Abbas et al. (2021), the soil used in this study comes from the Borg el-Arab region in northern Egypt, which is roughly 62 kilometres west of Alexandria. As indicated in Fig. 1, collapsible soil appears to be a yellow or brown silty sand interlocked with a crack of limestone. Soil samples were oven-dried before being processed through sieve #40. The physical properties of the soil were then calculated, as shown in Table 1.

Fig. 1 Collapsible soil

International Journal of Advances Engineering and Civil Research

Print ISSN 2974-4385

VOLUME 2, ISSUE 1, 2022, 72 - 90.

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Table 1. Physical properties of soil (Abbas et al., 2021)

USCS	SM
Color	yellow/brown
G_s	2.7
L.L	30%
PI	NP
P.L	NP
OMC	12.27%
MDD	1.9 g/cm^3
Water content	4.35%

2.2 Textile sludge

According to Abbas et al. (2021), The textile sludge (T.S) was taken from a textile Plant in Tanta, Egypt. As illustrated by Fig. 2, the sludge was oven dried before being crushed and passed through sieve #40. Table 2 shows the chemical characteristics of textile sludge measured using ICP (inductivity coupled plasma).

Fig. 2 Textile sludge: (a) before crushing; (b) after crushing (Abbas et al., 2021)

Table 2. Chemical properties of textile sludge (Abbas et al., 2021)

Chemical component	Concentration (mg/Kg)
Al	55620.01
Ca	38763.93
Fe	26376.04
Mg	20178.59
K	2812.20
Pb	100.48
Cd	2.19
Cr	500.66
Cu	163.23
Ni	64.5
Zn	515.58
Hg	7.89

3. Experimental work

3.1 Geotechnical tests

Abbas et al. (2021) investigated the effect of textile sludge on the geotechnical properties of collapsible soil, which include Atterberg limits, collapse potential, CBR value, compaction parameters, and shear parameters.

3.2 Chemical and microstructure analysis

Microstructure study was performed on three samples: collapsible soil, textile sludge, and soil mixed with 24% textile sludge. The following is an explanation of the different techniques that have been applied.

XRD (X-ray Diffraction)

The mineral composition of soil, textile sludge, and soil with 24% textile sludge was determined by X-ray diffraction (GNR X-ray Diffractometer, model APA 2000PRO). The radiation was performed with a Cu anode, λ= 1.54 A°) at 35kV and 30 mA. The XRD measurement collected was 2θ data in the range of 15° to 75° with a scanning rate of 0.05°/2s.

FTIR (Fourier Transform Infrared Spectroscopy)

The functional groups of soil, textile sludge, and soil with 24% textile sludge were identified by Fourier transform infrared spectroscopy (tensor-27 FTIR spectrometer), the spectrum was recorded at wave length in the range of 5000–400 cm^{-1}.

SEM (Scanning Electron Microscopy)

The microstructure and morphology of collapsible soil and soil treated with textile sludge were investigated by (JEOL- JSM-6510LV scanning electron microscope). The specimens were coated with thin film of gold and test was conducted at accelerating voltages of 30kV.

EDAX (Energy-dispersive X-ray spectroscopy)

Elemental and chemical compositions of collapsible soil and soil treated with textile sludge were monitored by an Energy-dispersive X-ray spectroscopy (Oxford X-Max 20).

4. Results and discussions

4.1 Geotechnical results

Effect of textile sludge on collapse potential

Collapse potential was significantly decreased by adding 24 percent textile sludge to collapsible soil, reaching a value of 76.36%, as revealed by the decline in volumetric strain at 200 KN/m2 in Fig. 3 (Abbas et al., 2021).

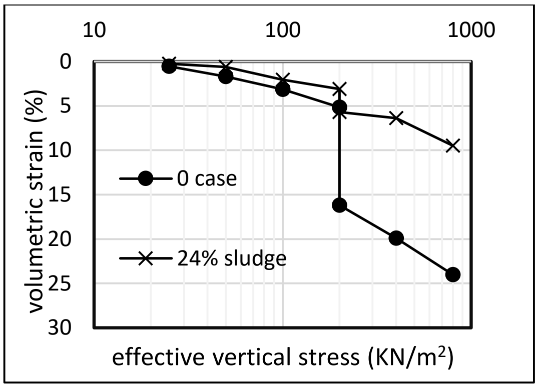

Fig. 3 Oedometer results for soil and soil mixed with 24% of textile sludge (Abbas et al., 2021)

Effect of textile sludge on cohesion

On both dry and wet samples, Abbas et al. (2021) performed a direct shear test. The cohesiveness value of collapsible soil increased when 24% of textile sludge was added. Cohesion rose from 29.3 KN/m2 to 41 KN/m2 for dry samples. While in the case of wet samples, as seen in Fig. 4, the cohesiveness value rose from 32.1 KN/m2 to 41.9 KN/m2.

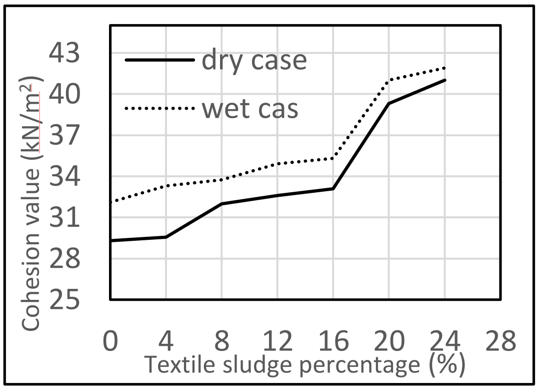

Fig. 4 Effect of textile sludge on cohesion value (Abbas et al., 2021)

Effect of textile sludge on liquid limit

According to Fig. 5, when textile sludge was added to soil at a rate of 24%, the liquid limit value of the collapsing soil increased from 30% to 41% (Abbas et al., 2021).

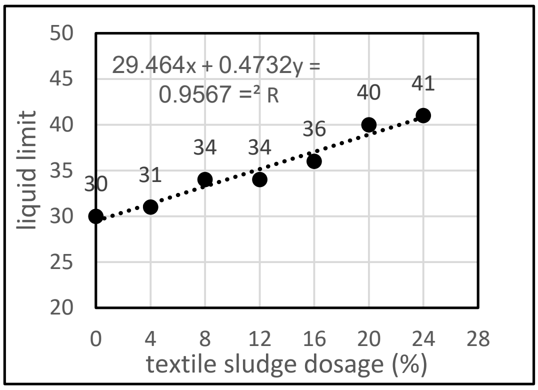

Fig. 5 Effect of textile sludge o liquid limit value

4.2 Chemical and Microstructure results

According to the information above, textile sludge plays a part in decreasing the value of collapse potential while enhancing the values of cohesiveness and liquid limit. The characteristics of sludge, the characteristics of soil that has been combined with 24% of sludge, and the collapsing behaviour of soil are all explained in this part, but from the perspective of the microstructure.

XRD analysis

The XRD micrographs of collapsible soil, textile sludge, and soil treated with 24% sludge were illustrated in Fig. 6. The main minerals identified in collapsible soil are calcite, quartz, kaolinite, and feldspar. The structure of collapsible soil is held together by cementing bonds occurred by clay minerals (kaolinite) or calcium carbonate (calcite), as a result of saturation and compressive load these bonds are weakened and consequently collapse occurs (Kalantari, 2013).

Only crystalline fingerprint phases were discovered at angles (29.25, 33.95, and 38.15) in the textile sludge XRD findings, illustrating the sludge's amorphous nature. The primary identified mineral is clinoptilolite-Na (Al4.08 Ca0.66 H7.96 K0.36 Na1.56 O43.96 Si13.92), and its presence

International Journal of Advances Engineering and Civil Research

Print ISSN 2974-4385

VOLUME 2, ISSUE 1, 2022, 72 - 90.

Online ISSN 2974-4393

appears to be necessary for the absorption of various heavy metals from textile sludge and compliance with environmental regulations (Kocasoy & Ahin, 2007). According to Ahmadi and Shekarchi (2010), clinoptilolite exhibits unique properties including cation exchange capacity, high specific surface area, and high SiO2 and Al2O3 content, similar to other pozzolanic materials. The presence of such materials most likely results in cementitious behaviour, which improves collapsibility.

For soil sample mixed with 24% textile sludge, a new peak appears at 2ϴ =27.5 and it could refer to Clinoptilolite (Molla et al., 2019). The existence of the fingerprint phases of Clinoptilolite in the soil mixed with 24% of textile sludge and the reduction of the intensities refers to the phases of the clay minerals claims the function of such material that replaces the clay minerals and form cementitious bonds. That fits well with the increase of soil cohesion and reducing collapse potential.

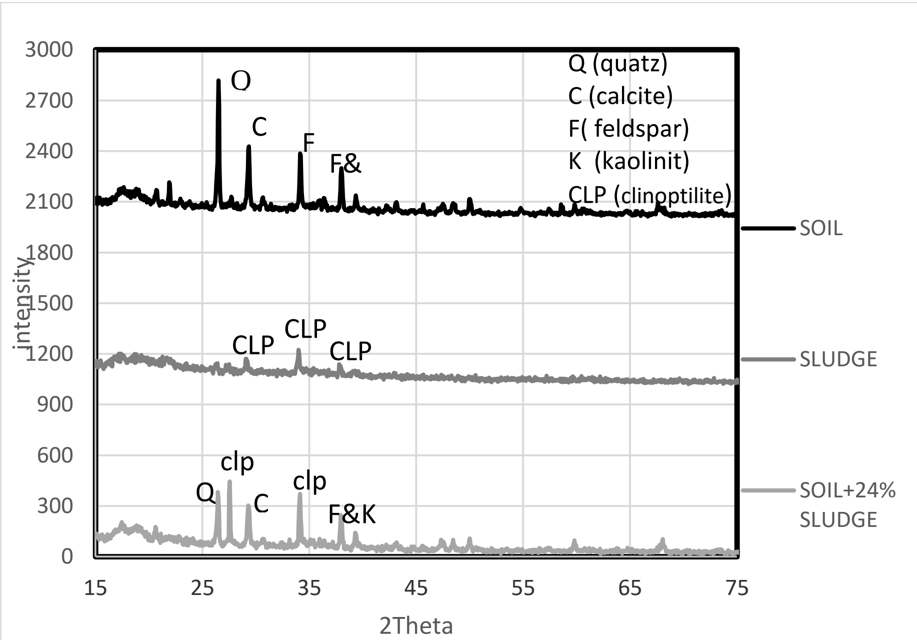

Fig. 6 XRD results of collapsible soil, textile sludge and soil +24% textile sludge

FTIR RESULTS

FTIR results of soil, textile sludge, and soil mixed with 24% of textile sludge are illustrated in Fig. 7. For collapsible soil, the vibration mode of the bands at 1444, 874, and 715 cm^{-1} indicated the presence of calcite structure in the sample (Galván et al., 2009; Sun et al., 2014). Also, weak peaks around 1790–1800 and at 2518 cm^{-1} may refer to the existence of calcite (Zhan et al., 2018). The strong band at 790 cm^{-1} and the weak band at 690 cm^{-1} refer to the presence of quartz (Sathya et al., 2012). The band at 472 cm^{-1} comply with the Si−O bending band vibration (Lee et al., 2013). The peak at 1032 cm^{-1} derived from in-plane Si–O stretching modes and refer to kaolinite band. Crystallized kaolinite could be also concluded from the presence of OH bands at 3696 and 3616 cm^{-1} (Martens et al., 2002; Yin et al., 2019). The presence of such transmission peaks in the FTIR results of soil shows the main minerals in soil which in turn confirms the XRD results.

Textile sludge spectrum shows bands at 796 and 469 cm^{-1} which confirm the existence of aluminum and silicon in sludge as it refers to vibration modes of O–T–O groups and the bending vibrations of T–O bonds, respectively where (T = Si and Al) (Mansouri et al., 2013). Band at 537 is due to Out-of-plane C=O bending (Kong & Yu, 2007) and bands at 874 and 1425 cm^{-1} indicate a higher amount of carbonates. Two broad bands are found at 1554,1655 cm^{-1} refer to (amide II and amide I respectively), The broad band at 3400 cm^{-1} usually appears in wastes and it assigned to hydroxyl groups and water (Smidt & Schwanninger, 2005). The transmission band at 1039 could refer to C–O stretching, although aliphatic methylene groups are found at 2925 and 2855 cm^{1}. Most of the previous bands represented parts of many organic molecules which supports an important function of textile sludge that could affect the water holding capacity of amended soils and perform a stable structure for soil (Grube et al., 2006).

For soil sludge mixture, the band at 2924 cm^{-1}, may refer to the presence of aliphatic groups (CH) in the mixture (Gokulakumar & Narayanaswamy, 2008). Aliphatic methylene groups and amide bands, could be indication of the presence of organic matter which play a vital rule in fermentation process and production microorganisms in a large amounts to reduce the cost of soil clogging through blocking the pores of soil by suspended particles of sludge (Ivanov & Chu, 2008). The broad peaks at 1633 cm^{-1} and 3426 cm^{-1} refer to the hydroxyl groups and water absorbed on the surface(Cheng et al., 2017). Accordingly, adding textile sludge to soil improves its water holding capacity and as a result, an increase in the initial water content of soil will occur and lead to decrease in suction and consequently decrease in collapse (Ali, 2014). Increasing the water holding capacity also affected on increasing the liquid limit value of mixtures.

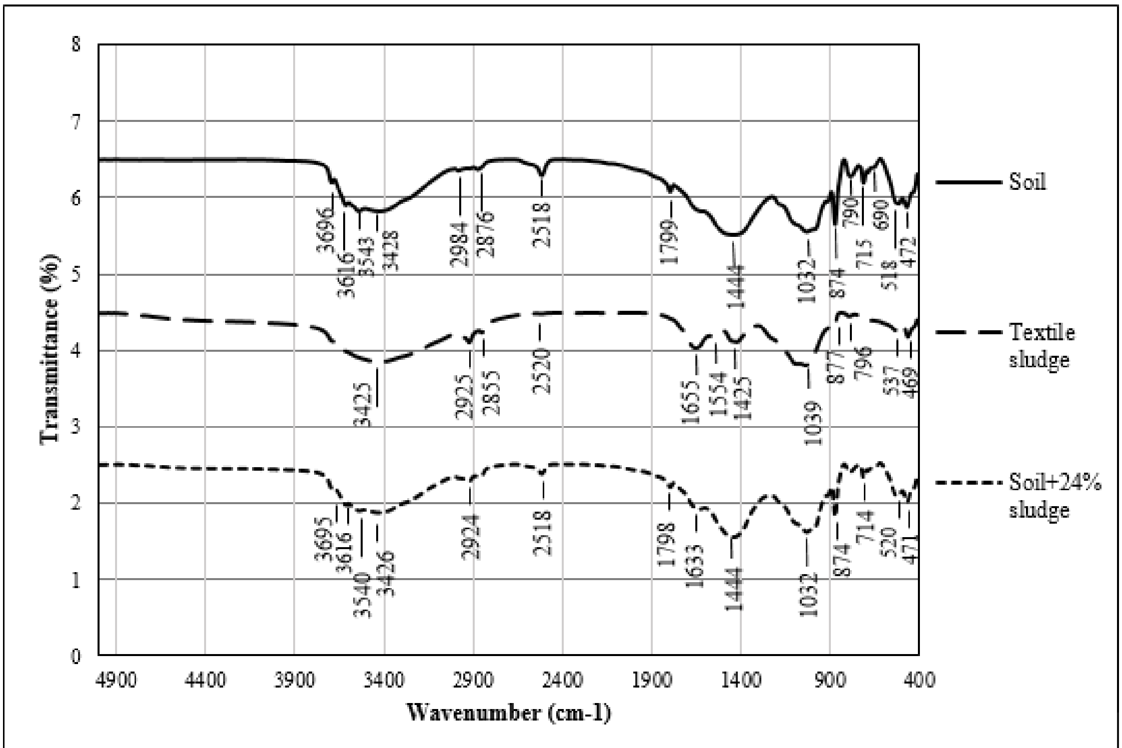

Fig. 7 FTIR transmission spectra of soil, textile sludge and soil +24% textile sludge

International Journal of Advances Engineering and Civil Research
Print ISSN 2974-4385
VOLUME 2, ISSUE 1, 2022, 72 - 90.
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Results of Scanning Electron Microscopy (SEM) associated with Energy-dispersive X-ray spectroscopy (EDAX)

The SEM micrograph of textile sludge in Fig. 8a indicates the aggregate shapes of sludge particles with the non-uniform distribution. In Fig. 8b, the EDAX elemental analysis of textile sludge shows that oxygen(O) and carbon (C) are the main components of textile sludge with additional high peaks of silicon (Si) followed by aluminum (Al), magnesium (Mg), calcium (Ca), iron (Fe). the presence of such elements plus the presence of high oxygen content could refer to the presence of the following oxides (SiO_2, Al_2O_3, MgO CaO, and Fe_2O_3) which are the main oxides to form a pozzolanic reaction (Amiralian et al., 2015). EDAX result of textile sludge also indicates traces of certain elements such as (Na, K, Cl, Ti, Cu, P, and Zn). The SEM micrograph of collapsible soil Fig. 9a shows a large inter aggregates pores, these pores could be classified as bracket pores. This type of pores has a larger diameter than its surrounding particles and has more ability to collapse (Xie et al., 2018). The EDAX spectroscopy in Fig. 9b shows the presence of a high percentage of oxygen (O), calcium (Ca), silicon (Si) and carbon (C) with small proportion of some components such as Al, S, Mg, Fe, Cl, Na, K, and Ti. SEM of soil treated with textile sludge Fig. 10a, bracket pores has been converted into mosaic pores, these pores have a volume smaller than the surrounding particles and less susceptible to compressibility and collapse (Xie et al., 2018), it is also noticeable that after adding sludge, the constituents of the soil as sand, silt, and clay particles cannot be configured out obviously which confirms the cementitious effect of the sludge and confirms also the homogenous distribution of sludge particles through soil samples. The EDAX spectroscopy in Fig. 10b, explains the presence of a high percentage of

Print ISSN 2974-4385

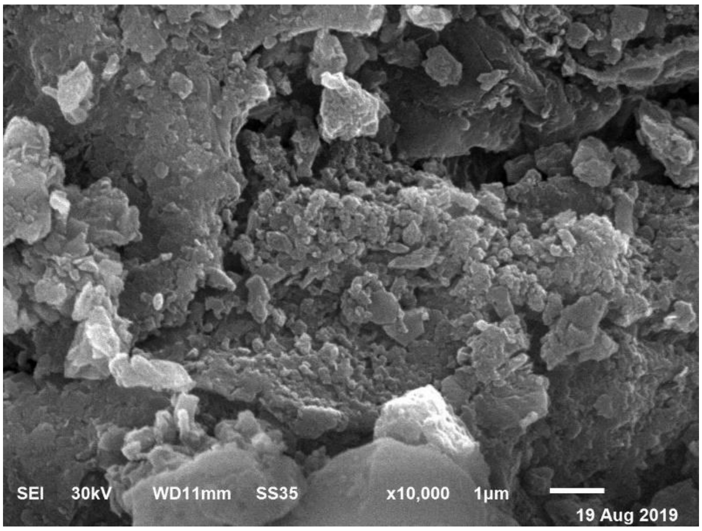
International Journal of Advances Engineering and Civil Research

VOLUME 2, ISSUE 1, 2022, 72 - 90.

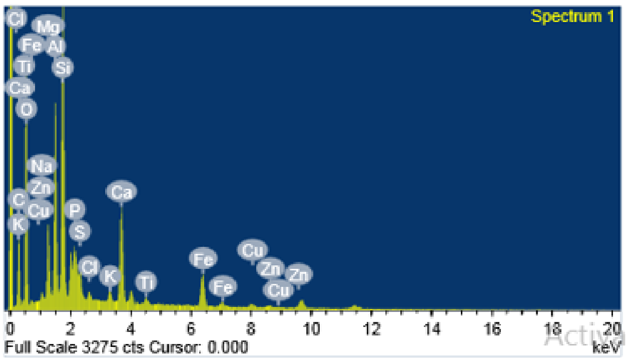
Online ISSN 2974-4393

oxygen (O), carbon (C), calcium (Ca) and silicon (Si) with small proportions of some elements such as Al, Mg, Fe, Na, K, Cl, and Ti.

(a)



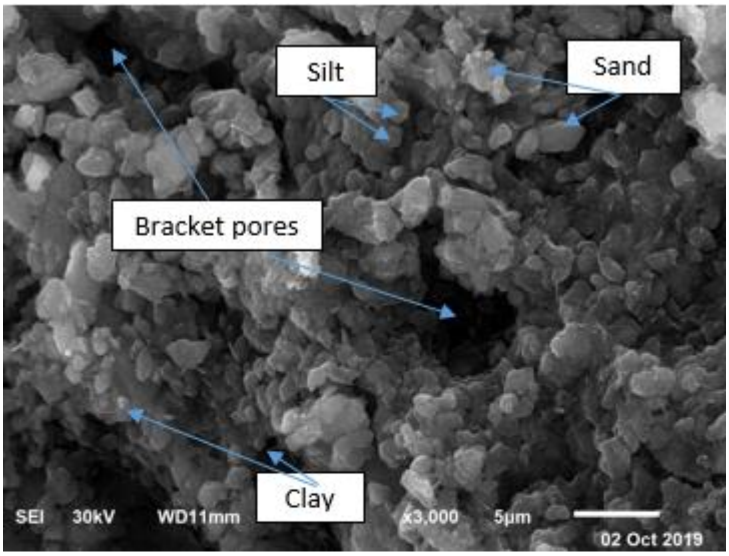
(b)



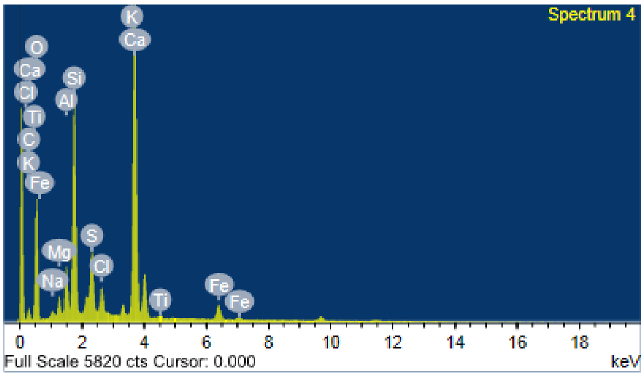
Elem...	Weight%	Atomic%
C K	22.68	31.57
O K	51.92	54.26
Na K	0.51	0.37
Mg K	3.02	2.08
Al K	6.02	3.73
Si K	8.59	5.12
P K	0.78	0.42
S K	0.51	0.26
Cl K	0.26	0.12
K K	0.28	0.12
Ca K	2.77	1.15
Ti K	0.24	0.08
Fe K	1.90	0.57
Cu K	0.32	0.08
Zn K	0.20	0.05
Totals	100.00	

Fig. 8: (a) (SEM) for textile sludge; (b) (EDAX) for textile sludge

(a)



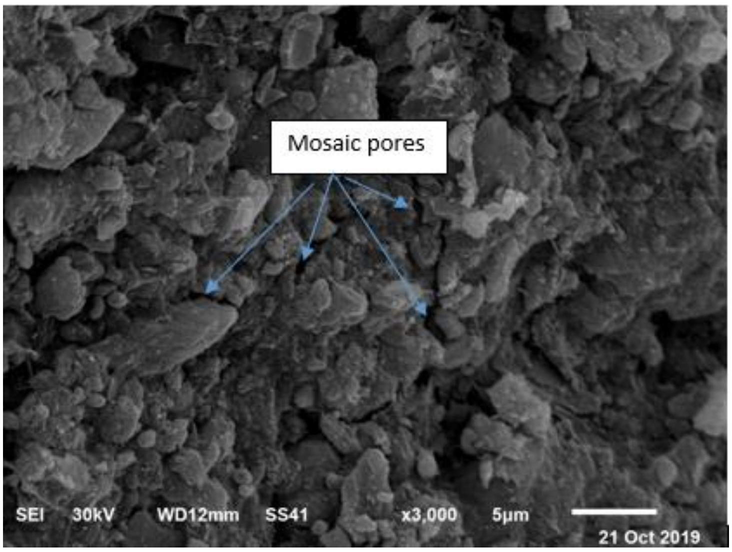
(b)



Elem...	Weight%	Atomic%
C K	7.98	12.62
O K	56.51	67.05
Na K	0.75	0.62
Mg K	1.50	1.17
Al K	2.58	1.82
Si K	9.86	6.67
S K	2.51	1.49
Cl K	1.34	0.72
K K	0.65	0.32
Ca K	14.82	7.02
Ti K	0.15	0.06
Fe K	1.35	0.46
Totals	100.00	

Fig. 9: (a) (SEM) for collapsible soil; (b) (EDAX) for collapsible soil

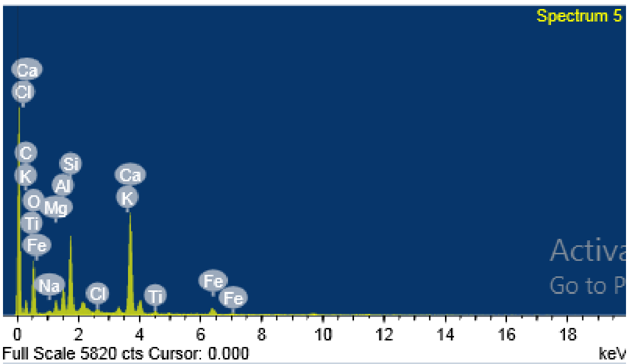
(a)



International Journal of Advances Engineering and Civil Research

Print ISSN 2974-4385 | VOLUME 2, ISSUE 1, 2022, 72 - 90. | Online ISSN 2974-4393

(b)



Elem...	Weight%	Atomic%
C K	21.54	30.37
O K	53.33	56.45
Na K	0.63	0.46
Mg K	1.98	1.38
Al K	2.88	1.81
Si K	7.60	4.59
Cl K	0.35	0.17
K K	0.61	0.26
Ca K	9.47	4.00
Ti K	0.39	0.14
Fe K	1.23	0.37
Totals	100.00	

Fig. 10: (a) (SEM) for collapsible soil treated with 24% of textile sludge; (b) (EDAX) for collapsible soil treated with 24% of textile sludge**.**

Conclusion:

1) The interactions of particles with clay and calcium carbonate bonds have been related to the behaviour of soil collapse by XRD data. After overloading and saturation, these connections are broken. XRD further revealed textile sludge features such as a large specific surface area, citation exchange capacity, and a high concentration of SiO2 and Al2O3. All of these features contributed to a decrease in soil collapsibility and an increase in cohesion value.

2) According to the FTIR data, adding textile sludge to the soil produces organic matter and, hence, microorganisms, reducing soil collapsibility and the expense of soil clogging. Textile sludge additionally improved the soil's capacity to retain water, which raised the liquid limit.

3) The treated soil's SEM results revealed a homogenous compact structure, and the bracket pores had transformed into mosaic pores, which are less prone to collapse.

4) The EDAX results of textile sludge show a significant concentration of oxygen as well as aluminium, magnesium, calcium, silicon, and iron, indicating the existence of these elements in oxide form, which played a part in the pozolanic method.

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