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Remediation Trend of Engineering Behavior of Wastewater Contaminated Clay Using Iron Nano Oxide: Experimental Studies

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ABSTRACT

Soil polluted with urban wastewater due to defect of wastewater disposal and leakage from wastewater channels is a common type of pollution in urban areas which in addition to environmental damage, has significant effects on soil engineering parameters. In present study, effects of municipal wastewater on mechanical behavior of soil and clay microstructure was studied, and then effects of iron oxide nanoparticles on remediation trend of contaminated soil was investigated. To achieve this, unconfined compressive strength (UCS), collapse and SEM analysis tests were performed on contaminated samples containing 20%, 60% and 100% wastewater at 1, 3 and 5 months and also on contaminated soil remidiated with 0.5-4% Iron nano oxide. Results showed that wastewater reduces shear strength of clay and this decreasing trend increases with increasing percentage and contamination duration. UCS of soil contaminated with 100% wastewater decreased by 49% after 5 months of contamination. Also, wastewater in the soil caused to soil collapse after 5 months . SEM images showed the clay structure became clotted after contamination and soil pores increased compared to natural soil. Improvement phase results showed that by addition of Iron nano oxide to contaminated soil, shear strength significantly increased, and optimal percentage of Iron nano oxide was 3% in which UCS increased by 105.2%. By increasing the percentage of Iron nanoxide, intensity of collapse index of contaminated soil decreases. Best case senario, final strain of soil decreases by 43.4% compared to contaminated soil. Therefore, utilizing Iron nanooxide is recommended to improve engineering behavior of contaminated clay.

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INTRODUCTION

Soil is one of the most important materials in nature that is used in engineering projects. All construction is done inside or on the ground, but all soils are not suitable for construction and engineering works and contaminated soils are in this group of soils. Contamination can damage the environment in the short or long period as a unit or combination of several chemical pollutants and cause soil pollution [1-3].

Soil pollution can occur due to the entry of chemicals caused by agricultural fertilizers, industrial activities, waste disposal, solvents, industrial wastewater, and municipal wastewater [4-7]. On the other hand, the infiltration of urban wastewater in some cities due to lack of development of municipal wastewater network, or lack of proper wastewater disposal system, leakage from wastewater channels has caused underground false water trips. Municipal wastewater contains suspended solutes and soluble substances (organic and inorganic) such as feces, urine, soluble proteins, drugs, detergents, salt, ammonia, hydrogen sulfide, thiocyanate, thiosulfate, etc., and each of them is the cause of soil pollution [8, 9].

Researches also show the negative effects of pollutants on changing the physical, chemical and mechanical behavior of soil [10, 11]. Therefore, studying the geotechnical behavior of soil in the presence of pollutants is a necessary task to prevent land settlement

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and prevent damage to engineering structures and technical buildings or to select a suitable method for improving this type of soil.

Huang and Keller [12] showed that organic acids dissolve clay minerals and cause changes in soil structure. Cyrus et al. [13] indicated that the shear strength of soil contaminated with ammonium chloride and ammonium phosphate decreases with increasing contaminant percentage. Ramakrishnegowda et al. [14] in their study, investigated the effect of sodium hydroxide solution on the shear strength parameters of fine-grained soil and in their results stated that with increasing the percentage of contamination, the cohesion and the internal friction angle of the soil decreased and increased, respectively. Li et al. [11] investigated the effect of natural leachate on the geotechnical behavior of clay. They stated that with an increase in the percentage of leachate, the hydraulic conductivity and soil cohesion decrease and the angle of internal friction increases. Karkush and Resol [15] in their studies claimed that industrial wastewater has increased cohesion and has reduced the angle of internal frictionof sandy soil.

Clay is able to interact with contaminants due to its large specific surface area, which makes it possible for clays to absorb some or all of the contaminants. On the other hand, the geotechnical behavior of contaminated soils is complicated and depends on factors such as the type of contaminant, duration of contamination, type of clay mineralization and the reaction that occurs in the soil-contaminant system [16].

As a result, in addition to understanding the changes in the geotechnical properties of contaminated soils, it is important to select the appropriate method to improve these soils to prevent damage related to engineering projects. According to past studies, traditional additives such as cement, lime, fly ash, etc. have been used to stabilize and improve soil engineering behavior and prevent soil settlement [17-20]. Despite the high useage of cement to stabilize contaminated soils, the results of previous studies showed that the use of cement is limited [21]. For example, a large amount of CO_2 gas is released during the cement production process, which causes significant environmental damages [22, 23]. Also, in the process of stabilization of contaminated soils aiding cement, in general, materials in contaminated soils such as sulfates and organic matter are considered as a slowing factor in the stabilization process, resulting in less resistance than natural soils [24].

Therefore, thanks to nanotechnology, replacing traditional additives by nanoparticles such as nanoclay, nano-silica, nanocopper, nano-magnesium, nano-aluminum and nanoclay to improve the strength of materials and improve soil engineering behavior has been studied by various researchers [25-27]. For example, in their studies, Majeed and Taha [28] stated that adding 0.5-1% of nanomass, magnesium nanoxide and nanoclay to fine-grained soil leads to an increase in soil resistance parameters. Moayed and Rahmani [29] in their study showed that unconfined compression strength of clay increased by adding 1-5% nano SiO_2. Gao et al. [30] also studied on the shear property of nano-MgO-modified soil. Hayal et al. [31] examined the addition of 2.5-10% nano-clay and 0.5-3% nano silica to gypsum-contaminated clay, and showed that the collapse Index decreases with the addition of any amount of nano-clay, but increases with the addition of more than 1% nano-silica.

Among nanomaterials, Iron nano oxide has been used less to improve soil engineering parameters. But these nanomaterials were recommended by researchers to remove pollutants from the environment. However, regarding the role of nano-Iron in the removal of pollutants, researchers have concluded that nano-scale Iron can eliminate soil pollution[32, 33]. For example, according to Chang [34] study, nano-iron can be utilized to remove many pollutants, heavy metal, chlorinated methanes, inorganic anions, etc.

MATERIAL AND METHODS

Material

Soil: Geotechnical parameters of utilized fine-grained soil such as classification, etherberg limits, maximum dry density, specific gravity, unconfined compressive strength, pH, electric conductivity according to ASTM D2487, ASTM D4318, ASTM D698, ASTM D854, ASTMD2166, ASTM D4972, respectively and ISO 11265 determined and presented in Table 1.

Wastewater: In the pollution phase, urban wastewater has been used to make contaminated soil. The results of chemical and biological analysis of wastewater consumption are presented in Table 2.

Iron oxide nanoparticles (N.I): The additive used in this study to improve contaminated soil is iron oxide nanoparticles powder in alpha phase with chemical

Table 1. Engineering properties of clay soil used in the study

Parameter	Values
Liquid limit (%)	30.40
Plastic limit (%)	15.60
Plasticity index (%)	14.80
Soil classification	CL
Maximum dry density (kN/m^3)	17.30
Optimum moisture content (%)	18.70
Specific gravity	2.63
Unconfined compressive strength (kPa)	71.6
pH	7.60
Electric conductivity (ms/m)	270

Table 2. Results of chemical and biological analysis of wastewater

Parameter	Values
COD (mg/l)	566
BOD_5 (mg/l)	320
TSS (mg/l)	137
TDS (mg/l)	249
pH	7.8
EC (µS/cm)	525
Cl (mg/l)	248.5
NO_3^- (mg/l)	29.4
NO_2^- (mg/l)	0.13
NH_3 (mg/l)	103.3
PO_4^{3-} (mg/l)	41.9
TN (mg/l)	118.65

formula (Fe_2O_3), whose physical characteristics are presented in Table 3. Used iron nano oxide powder contains more than 98% iron oxide. The amount of other elements included sodium, phosphorus, sulfur, manganese, aluminum, chromium, silica and calcium, are 0.0005%, 0.016%, 0.12%, 0.095%, 0.0002%, 0.037%, 0.134% and 0.024% are in this powder.

Sample preparation

To make a sample of soil contaminated with wastewater, a solution of wastewater with concentrations of 20%, 60% and 100% was prepared and then added to dry soil (as much as saturated moisture). Then, in order to maintain moisture and curing the samples, they were stored in a multi-layer nylon bag in the laboratory for 1 , 3 and 5 months.

To make samples for remidiating the contaminated soil, the soil was first dried by contamination of 100% wastewater (after 5 months of contamination). Then, at a rate of 0.5% to 4% of the dry weight of the contaminated soil, Iron nano oxide was added to the soil and mixed in the dry state. Then water (optimum moisture content) was added to the samples and mixed. They were then placed in multilayer plastic and tested after 28 days of curing.

Test methods

In this study, to evaluate the compressive strength of natural, contaminated and improved soil, unconfined compressive strength (UCS) test according to ASTM D2261 standard, has been done on cylindrical specimens with a diameter of 38 mm and a height of 76 mm. Also, to investigate the changes in the collapse index, the collapse test was performed on natural, contaminated and improved soil according to ASTM D5333 standard in the consolidation device. Figure 1 shows some of the curedspecimens, and specimens that have been subjected to the unconfined compressive strength and collapse tests.

Table 3. Physical properties of iron oxide nano

Property	Values
Purity	>98%
Grain Size	20-40nm
SSA	40-60 m^2/g
Bulk density	1.20 gr/cm^3
True density	5.24 gr/cm^3

Figure 1. Cured and tested samples

Based on collapse test, the collapse index, which indicates the rate of soil collapse in percentage at a stress of 200 kPa, is obtained from Equation (1).

$$I_e = \frac{d_f - d_i}{h_0} \times 100 \qquad (1)$$

In Equation (1), I_e, d_f, d_i and h_0 are defined as collapse index, the number of gauge at 200 kPa after saturation, gauge number at 200 kPa before saturation and the initial height of the sample, respectively. In this research, according to ASTM D5333 standard, I_e has been used to assess the severity of soil collapse.

In order to investigate the microstructure changes of contaminated and improved soils, compared to natural soils, scanning electron microscopic (SEM) analysis was performed by SPUTTER device model SC7620.

RESULTS AND DISCUSSION

Unconfined compressive strength analysis

Unconfined compressive strength is a direct measurement of strength for cohesive soils. To investigate the changes in compressive strength in the pollution conditions compared to natural soil, unconfined compressive strength test was performed on samples of natural soil and soil contaminated with 20%, 60% and 100% wastewater at contamination duration of 1, 3 and 5 months. Figures 2-4 show the axial stress-axial strain behavior of natural and contaminated soils. As can be seen, in each percentage of pollution, with increasing pollution duration, the axial stress at the moment of rupture has decreased, which indicates a decrease in compressive strength and shear strength of soil contaminated with wastewater compared to natural soil. As shown in Figure 5, in the short term (1 month) with increasing contamination percentage, the decreasing trend of UCS is low. However, with increasing contamination period, the decrease in UCS compared to natural soil increases. For example, the UCS content of soil samples contaminated with 20%, 60% and 100% of wastewater after 1 month of contamination was decreased 3.6%, 7.1% and 13.3%, respectively, and after 5 months of contamination was declined 33.2%, 42.2% and 49%, respectively compared to natural soil. As a result, the duration of contamination has a great effect on reducing the shear strength of soil, especially for low percentage of contamination. Also, as shown in Figures 2-4, for each percentage of contamination, the failure strain corresponding to the maximum resistance decreases with increasing duration of contamination.

In other words, soil contaminated with wastewater causes soil to fail at lower strains; this is a negative consequence in the mechanical behavior of soils.

The decrease in soil strength can also be attributed to the possible failure of cementation bonds between soil particles after contamination with wastewater. Also, changes in the mechanical behavior of soils occur due to physical or physicochemical interactions between soil and pollutants. Physicochemical interaction occurs when pore fluid reacts with soil minerals. On the other hand, wastewater causes chemical changes in soil pore fluid and in general, the presence of these changes over time causes physicochemical interaction between pore fluid and minerals and thus changes in the mechanical behavior of soil.

The effects of iron oxide nanoparticles on the compressive strength of soil contaminated with 100% wastewater are shown in Figure 6. The results show that addition of iron oxide nanoparticles increases the axial stress related to contaminated soil (C100). As shown in Figure 7, as the percentage of iron oxide nanoparticles increases to 3%, UCS increases, and after this value, UCS is reduced compared to the optimal percentage of nanoparticles. UCS of improved soil with 0.5%, 1.5%, 3% and 4% iron nano oxide increased by 33%, 67.9%,

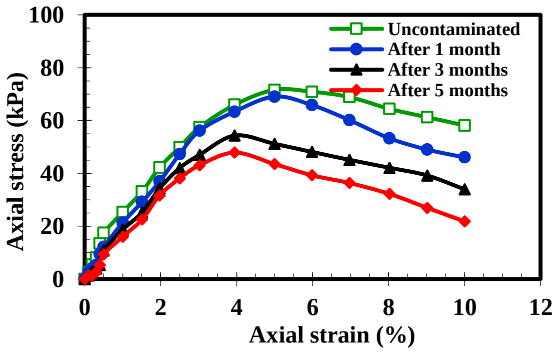

Figure 2. Stress curves versus strain of soil contaminated with 20% of wastewater at different times

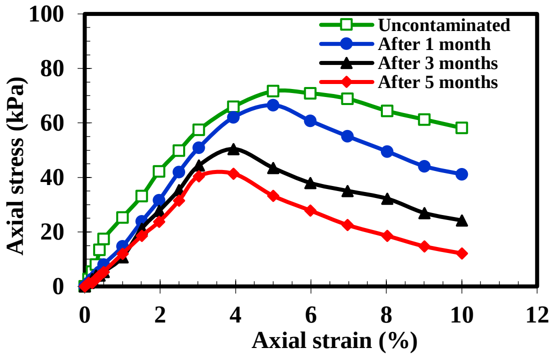

Figure 3. Stress curves versus strain of soil contaminated with 60% of wastewater at different times

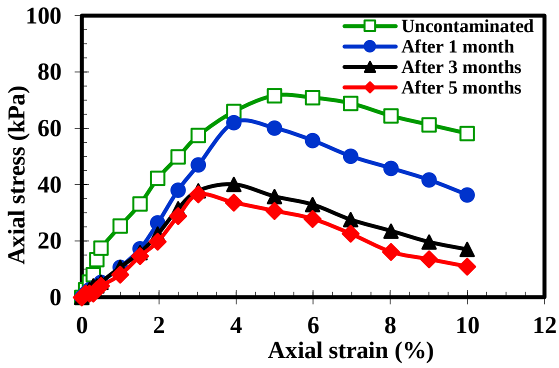

Figure 4. Stress curves versus strain of soil contaminated with 100% of wastewater at different times

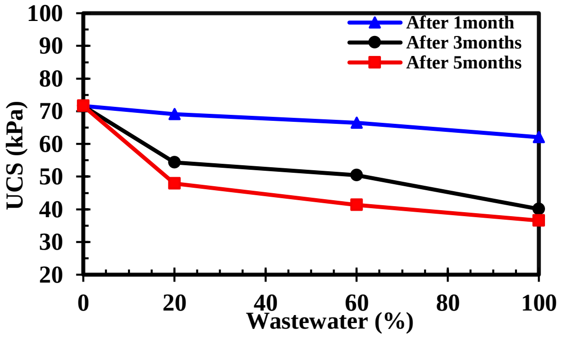

Figure 5. Variations UCS under the amount of contamination at different times of contamination

105.2% and 83.9% compared to contaminated soil, respectively. Increased UCS can be due to the placement of nanoparticles between soil pores and the creation of a communication network between soil particles and iron oxide nanoparticles, and it creates a dense and resistant structure. In addition, nanomaterials have very high reactivity due to high specific surface area (SSA) and electrical charges on the surface [25]. Therefore, nanoparticles can be adsorbed in cementation bonds between soil particles, and on the other hand, because iron oxide nanoparticles have a high hardness, they form bonds with higher strength [35, 36]. As a result, soil shear strength increases.

Also, the reason for the decrease in UCS after increasing the nanomaterials more than the optimal level (3%) can be the accumulation of nanomaterial particles, which leads to the formation of pore voids.

On the other hand, a sharp increase in the amount of nanoparticles of the soil increases the number of nanoparticles in a constant volume and poor interaction of nanoparticles. These particles produce weak and unstable masses [37].

Collapsible analysis

In order to investigate the collapse behavior and changes in soil collapse index (I_e) in pollution conditions compared to natural soil, collapse tests were performed on natural soil samples and soil contaminated with 20%, 60% and 100% wastewater at contamination duration of 1, 3 and 5 months. According to the results of collapse test, the collapse index of the studied natural soil is 0.4%, which is in the slight range according to ASTM D5333. As shown in Figures 8-11, the collapse index increases with increasing the percentage and period of contamination, and at a stress of 200 kPa after saturation, the soil undergoes a lot of strain. The results show that the intensity of the collapse index of samples contaminated with 20%, 60% and 100% of wastewater in the short term (1 month, in this study) is in the slight range . But in the long term (5 months, in this study) for the sample contaminated with 100% of wastewater, the intensity of collapse index is in the moderate range. As can be seen in Figures 8-10, the final strain (at 1600 kPa effective vertical stress) for each percentage of contamination increases significantly with increasing contamination duration. In this case, the final strain of soil contaminated with 20% of wastewater, after 1 month, 3 months and 5 months, the rate pollution were 2.9%, 5.3% and 14.6%, respectively. Also soil contaminated with 60% of wastewater, the rate of pollution were 7%, 9.9% and 25.1%, as well as soil contaminated with 100% wastewater the pollution has increased to 9.4%, 13.5% and 41.5% compared to natural soil, respectively.

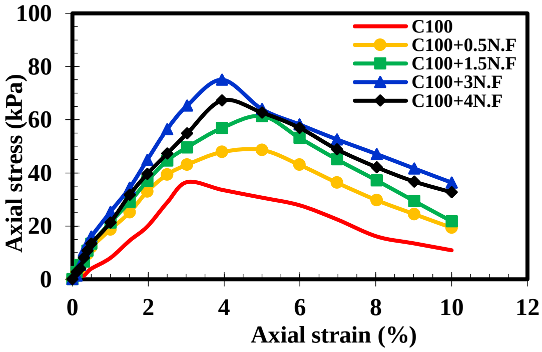

Figure 6. Stress curves versus soil strain remediated with different percentages of iron nanooxide

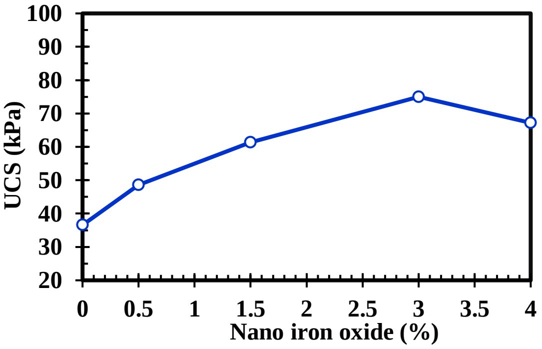

Figure 7. Variations UCS versus different percentages of iron nano oxide

Collapse refers to the sudden fall of soil due to the loss of strength of the bonding agent of soil particles, and the amount of collapse is dependent on the ratio of the initial porosity of the soil as well as the porosity of the soil structure. On the other hand, the more porous the soil structure would be, the greater the collapse occurrence. However, wastewater pollution causes a change in the nature of soil pore fluid and also increasing the concentration of pollution and duration of pollution causes physicochemical interaction of pore fluid and minerals and thus, caused serious changes in the soil structure.

The cause of soil collapse after contamination with wastewater can be considered as a change in the soil structure (increased porosity) as well as the loss or reduction of cementation bond between soil particles (see SEM images for a better understanding).

Figures 12 and 13 show the effect of iron oxide nanoparticles on the collapse behavior and changes in the collapse index (I_e) of soil contaminated with 100% wastewater. As can be seen, by adding iron oxide nanoparticles and increasing the percentage of nanoparticles, the improved soil undergoes less strain than the contaminated soil. The final strain of the improved soil with 0.5%, 1.5%, 3% and 4% Iron nanooxide decreased by 9.5%, 24%, 40.1% and 43.4% compared to the contaminated soil, respectively. Also, in the best case, the intensity of the collapse index reaches from the moderate value to the slight value.

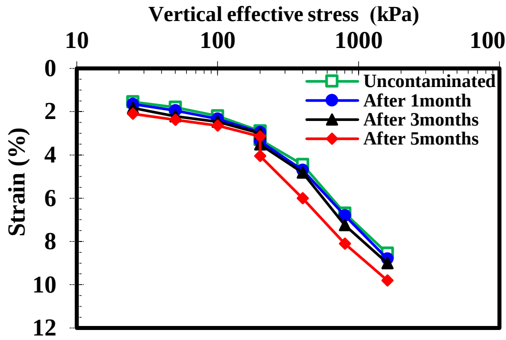

Figure 8. Variations in the collapse behavior of soil contaminated with 20% of wastewater at different times

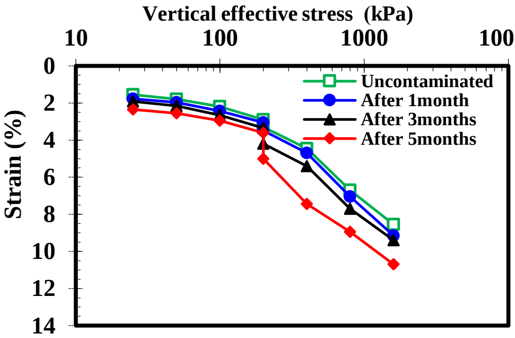

Figure 9. Variations in the collapse behavior of soil contaminated with 60% of wastewater at different time

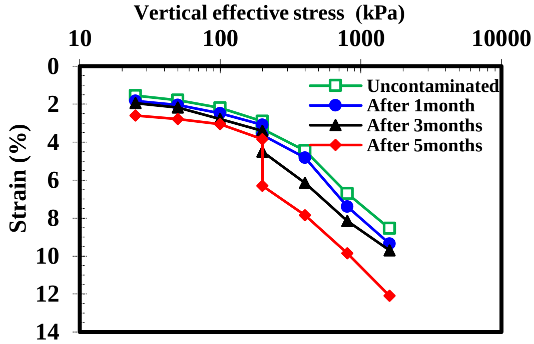

Figure 10. Variations in the collapse behavior of soil contaminated with 100% of wastewater at different times

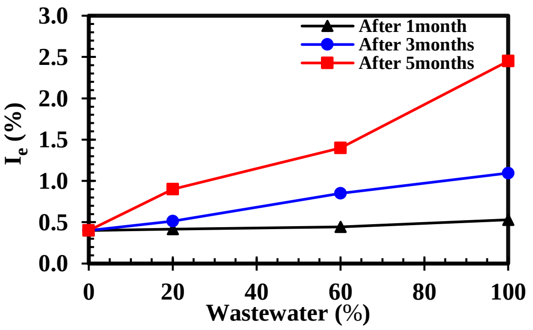

Figure 11. Variations I_e under the amount of contamination at different times of contamination

Materials in the nano scale often show very different physical behavior against atoms and bulk materials. The properties of nanoscale materials cannot necessarily be predicted with respect to the properties of materials on a larger scale. Nanoscale particles have a very high specific surface area due to their very small size. If used in very small amounts in the soil environment, the physico-chemical behavior and engineering properties of the soil are very specifically and significantly affected. The detial about the performance of soil using nanomaterials as addatives were discussed by Khalaf et al. [38].

As a result, the reason for the reduction of strain and the intensity of collapse can be the adsorption of nanoparticles in the cementation bond between soil particles, which creates another type of bond, which leads to increased soil particle cohesion and soil strength [31].

Scanning electron microscopic analysis

SEM images of natural soil, soil contaminated with 100% wastewater for maximum contamination duration (5 months processing), and soil improved with 3% nano-iron oxide (optimal percentage) are shown in Figure 14 for two different magnifications (100×, 1000×). As can be seen, the soil structure has become discontinuous compared to natural soil after being contaminated with wastewater, and the pores have become larger and the

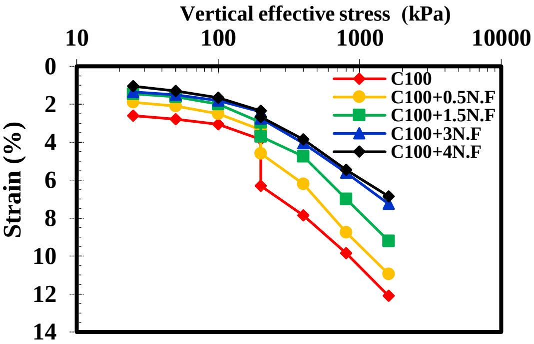

Figure 12. Variations in the collapse behavior of soil contaminated with 100% of wastewater at different times

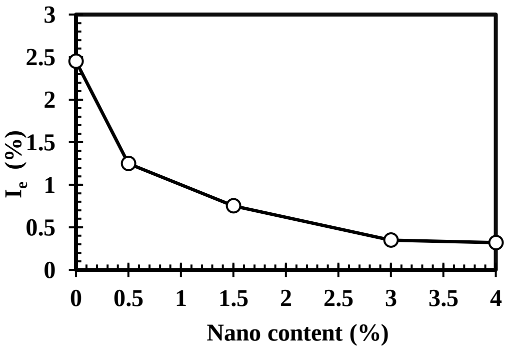

Figure 13. Variations I_e versus different percentages of iron nano oxide

soil structure has flocculated. This is due to the reduction of diffuse double layer (DDL). Because the dielectric constant is directly related to DDL, and on the other hand, due to the presence of organic solutions in wastewater, the dielectric constant of the pore fluid related to pure water decreases [39], resulting in reduced DDL. Moreover, the decrease in DDL leads to soil shrinkage and as a result, clay particles flocculate and soil cavities become larger. Also increase in pores indicates soil prone to change in engineering behavior including decrease in strength and increase in deformation under applied loads [40]. Therefore, soil microstructure images in this study confirm the decrease in soil strength.

As shown in Figure 14, the addition of nanoparticles to contaminated soil fills the soil pores, and the soil structure changes from discontinuous to fully continuous. Additionally the creation of new bonds between nanoparticles and soil particles (cementation bond) is quite obvious. The presence of cementation bond improves the mechanical behavior of the soil, which overlaps with the results of soil geotechnical experiments.

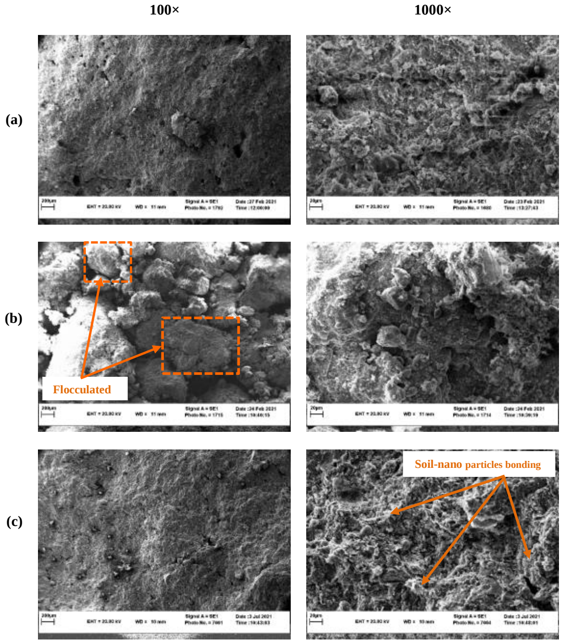

Figure 14. SEM images of (a) natural soil, (b) contaminated soil, and (d) remediated soil

CONCLUSION

In order to better understanding the effects of percentage and duration of wastewater pollution on the engineering behavior of clay soils and its modification using nano Iron oxide, a laboratory study including unconfined compressive strength and collapse tests as well as SEM analysis on wastewater contaminated clay and improved soil was performed with iron oxide nanoparticles. The main findings can be summarized as follows:

- Urban wastewater reduces the compressive strength of clay so that with increasing the percentage of wastewater and the duration of pollution, the compressive strength decreases greatly. 49% reduction in USC which is the largest amount is related to soil contaminated with 100% wastewater after 5 months of contamination.
- The results indicate that soil at low pollution (20% in this study) needs more time to change its mechanical behavior than maximum pollution (100%).
- Urban wastewater can cause clay collapse in the long term (5 months in this study). In the most critical case (sample contaminated with 100% wastewater) the final strain increases by 41.5% compared to natural soil.
- According to SEM images, urban wastewater changes the soil texture and flocculates soil structure, and on the other hand, soil pores are larger than natural soil, which changes the soil engineering behavior.
- In the improvement phase of the research, the results indicate the positive effects of iron oxide nanoparticles on improving the mechanical behavior of contaminated clay.
- By increasing the percentage of Iron nano oxide from 0.5% to 3%, the compressive strength of contaminated clay increases. The highest increase of USC was 105.2% which is related to soil sample modified with 3% Iron nano oxide.
- The intensity of the collapse index is reduced by adding iron oxide nanoparticles to the contaminated soil. In the best case, the intensity of the collapse index of the sample improved with 4% nano iron oxide from moderate reaches the value of slight. In this case, the final soil strain is reduced by 43.4% compared to the contaminated soil.
- Iron oxide nanoparticles reduce the pores of contaminated soil and according to the results of SEM analysis, iron oxide nanoparticles cause a cementation bond between soil particles as well as a more cohesive and integrated structure.

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Persian Abstract

چکیده

خاک آلوده به فاضلاب شهری به دلیل نقص دفع فاضلاب و نشت از کانال های فاضلاب یکی از انواع آلودگی‌های رایج در مناطق شهری است که علاوه بر آسیب‌های زیست محیطی، اثرات قابل توجهی بر پارامترهای مهندسی خاک دارد. در مطالعه حاضر اثرات فاضلاب شهری بر رفتار مکانیکی خاک و ریزساختار رس و سپس اثرات نانوذرات اکسید آهن بر روند اصلاح خاک آلوده مورد بررسی قرار گرفت. برای دستیابی به این هدف، آزمایش‌های UCS، رمبندگی و آنالیز SEM بر روی نمونه‌های آلوده حاوی ۲۰، ۶۰ و ۱۰۰ درصد فاضلاب در ماه‌های اول، سوم و پنجم و همچنین بر روی خاک آلوده اصلاح شده با ۰/۵-۴ درصد نانو اکسید آهن انجام شد. نتایج نشان می‌دهد که فاضلاب باعث کاهش مقاومت برشی خاک رس می‌شود و این روند کاهشی با افزایش درصد و مدت زمان آلودگی افزایش می‌یابد. UCS خاک آلوده به فاضلاب ۱۰۰ درصد، پس از پنج ماه آلودگی ۴۹ درصد کاهش یافت. همچنین فاضلاب موجود در خاک پس از ۵ ماه باعث رمبندگی خاک می‌شود. تصاویر SEM نشان داد که ساختار رس پس از آلودگی لخته شده و منافذ خاک نسبت به خاک طبیعی افزایش یافته است. نتایج فاز به‌سازی نشان می‌دهد که با افزودن نانو اکسید آهن به خاک آلوده، مقاومت برشی به طور قابل توجهی افزایش می‌یابد و درصد بهینه نانو اکسید آهن ۳ درصد بوده است؛ که در آن UCS ۱۰۵/۲ درصد افزایش یافت. با افزایش درصد نانو اکسید آهن، شدت شاخص رمبندگی خاک آلوده کاهش می‌یابد. در بهترین حالت، کرنش نهایی خاک نسبت به خاک آلوده ۴۳/۴ درصد کاهش می‌یابد. بنابراین، استفاده از نانو اکسید آهن برای بهبود رفتار مهندسی خاک رس آلوده توصیه می‌شود.