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Effects of Silica Fume and Nano-silica on the Engineering Properties of Kaolinite Clay

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ABSTRACT: The improvement of geotechnical properties of weak soils is of interest through the resources shortage. Therefore, this study focused on the effect of silica fume as industrial waste products and nano-silica on geotechnical characteristics and micro-structural properties of kaolinite clay as a soft soil with poor strength properties. Silica fume was added to the kaolinite clay to enhance the strength with 5, 10 and 15%. Moreover stabilized soil with nano-silica were fabricated with 1, 2 and 3% by dry weight of the soil. Then, Atterberg limits, standard proctor, unconfined compressive strength, and California bearing ratio tests were conducted. In addition, the micro-structural changes of soil samples through the stabilization were examined using scanning electron microscope. The results indicated that silica fume and nano-silica increase the optimum water content and decrease the maximum dry density of the stabilized soils. Addition of 15% silica fume and 3% nano-silica to kaolinite clay improved the unconfined compressive strength at curing age of 28 days by up to 70% and 55%, respectively. Also, the results of soaked California bearing ratio test after 7 days of curing demonstrated that 15% of silica fume and 3% nano-silica increased the California bearing ratio values about two times more than the raw soil. Scanning electron microscope (SEM) images were then utilized to evaluate the effects of additives on the kaolinite clay soil. It was concluded that silica fume and nano-silica filled pore space between clay particles and a dense matrix were formed. This textural event caused an improvement in compressive strength of stabilized soils.

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1- Introduction

Based on the increasing the usage of the resources and population growth, the soil with suitable properties for civil engineering project such as pavement construction are not available. Therefore, it is obligatory that the enhancement of local soils properties should be appropriately deliberated. Soil improvements are generally classified into two processes, soil modification or soil stabilization. “Modification” involves changing the texture and moisture sensitivity of the soil, usually by a change in the soil’s plasticity. “Stabilization” refers to a long-term strength gain of the compacted soil, which may occur in addition to the modification [1, 2].

In recent years, soil stabilization using the waste material in geotechnical engineering has been considered from an environmental point of view. Substantially the application of industries by-products reduces the cost and saves the energy in addition to satisfy the requirement of environmental awareness. Silica fume (SF), as defined in ACI 116R [3], is “very fine non-crystalline silica produced in electric arc furnaces as a byproduct of elemental silicon or alloys containing silicon production”. Among all the stabilizers, Silica silica fume as a very reactive pozzolan attracted growing attention due to its fine particles, large surface area, and the high silicon dioxide content [4]. The Silica silica fume was utilized as a partial replacement for Portland cement in concrete [5, 6]. In addition, it has been argued that silica fume is a potential and viable candidate to improve the geotechnical properties of clayey soils as stabilization agent by increasing unconfined compressive strength and reducing the permeability coefficient [7-10]. In this regards, the effect of SF on the geotechnical properties of high plasticity clay was examined and showed that this stabilizer can enhance the permeability, swelling pressure and compressive strength of composite [7]. Furthermore, Vakili et al. [8] used pozzolanic additives to improve the characteristics of expansive soils. The results demonstrated that these additives can considerably decrease the dispersivity potential and plasticity index, and increase unconfined compressive strength (UCS) of the soils after curing process.

Nowadays, nano-material and nano-particles are frequently used additives to enhance the characteristics of materials used in various engineering applications especially in civil engineering [11, 12]. Using nano-materials as a stabilizer have considerable merits. A relatively larger surface area can make materials more chemically reactive and affect

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their strength or electrical properties. Furthermore, Quantum quantum effects can lead to dominate the behavior of material at the nano-scale exclusively affecting the optical, electrical and magnetic behavior of materials [13]. Reducing energy consumption, economic savings, time savings, improving the quality of products have been introduced as the most important advantages of nano-material to increase quality of life and healthier lifestyles [14]. Based on the aforementioned benefits, nano-materials are presumed to be a potentially powerful stabilizer to betterment the properties of soils [14-18].

The experimental study in order to evaluate the deformation and strength characteristics of Loessial soils stabilized by nanosilicanano-silica demonstrated that by increasing the weight ratio of stabilizer to 3%, the unconfined compressive strength of the improved Gorgan Loessial soil increases 4 times only after 3 days curing time [15]. Moreover, Iranpour and Haddad (2016) investigated the impact of four types of nano-materials (nano-clay, nano-copper, nano-alumina, and nanosilicanano-silica) on the collapsible soil treatment. They argued that using an appropriate percentage of nano-particles could improve soil specifications [16]. In addition, the nanosilicanano-silica have been employed in combination with other chemical stabilizers such as cement and lime for soil improvement [17, 18]. Also in this regard, Seyedi Gelsefidi et al. [18] stated that addition of nanosilicanano-silica significantly improved the California bearing ratio (CBR) of soil-lime. Moreover, the nanosilicanano-silica was found as an additive which can affect the compatibility and strength of cement treated soils positively [17].

Kaolinite clay considered as a soil with poor properties through low strength characteristics [19]. Based on the aforementioned explanations, silica fume and nanosilicanano-silica were utilized as commonly applied additives by other researchers to improve the geotechnical properties of different clay soils [7-10, 14-16]. To the best of the authors' knowledge, there is no comparison between the impact of silica fume and nanosilicanano-silica on kaolinite clay properties. To this end, this study with performing the Atterberg limits, compaction, unconfined compressive strength (UCS) and California bearing ratio (CBR) tests on the stabilized soil aimed to investigate the effect of silica fume and nanosilicanano-silica on some geotechnical properties of kaolinite clay. Scanning electron microscope (SEM) also was employed to identify the micro-structural modification.

2- Experimental and Test Methods

2- 1- Materials

In this research commercial kaolinite clay was used as raw soil. Grain size distribution of the utilized soil meeting ASTM D422-63 [20] is shown in Figure 1. Moreover, Table 1 presents a summary of the engineering properties of the soil. Based on the Unified Soil Classification System (USCS), the soil is classified as CL. The chemical compositions of kaolinite clay, as determined using X-ray fluorescence (XRF) spectroscopy, is presented in Table 2.

With regard to the environmental impacts and geotechnical characteristics, silica fume (SF) and nanosilicanano-silica were added to the soil. In this research, silica fume (SF) obtained from Iran Ferroalloys Industries Company was utilized that its physical and chemical properties are shown in Table 3. To reach the objectives of this research, amorphous nanosilicanano-silica powder manufactured by Evonik Industries (Essen, Germany) with a solid content of more than 99.8%, an average size of 12 nm and surface area of 200 ± 25 m^2/g was exerted.

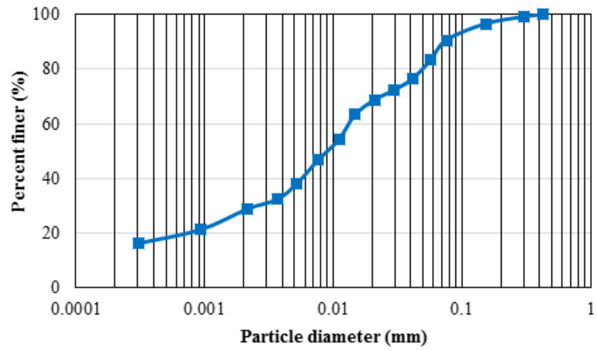


Figure 1. Grain size distribution of the soil

Table 1. Engineering properties of the soil

properties	Standard	Value
Liquid limit (LL), %	ASTM D4318 [21]	29.5
Plastic limit (PL), %	ASTM D4318 [21]	21.5
Plasticity index (PI), %	ASTM D4318 [21]	8
Unified soil classification system (USCS)	ASTM D2487 [22]	CL
Specific gravity	ASTM D854 [23]	2.65
Maximum dry density (MDD), kN/m^3	ASTM D698 [24]	17
Optimum moisture content (%), %	ASTM D698 [24]	16.2
Unconfined compressive strength (UCS), kPa	ASTM D2166 / D2166M [25]	129

Table 2. Chemical composition of Kaolinite clay

Compound	Weight (%)
SiO_2	72.5
Al_2O_3	18.07
Fe_2O_3	0.36
CaO	1.15
MgO	0.61
SO_3	0.06
K_2O	0.39
Na_2O	0.25
Other	0.61
Loss on ignition	6.0

Table 3. Physical and chemical properties of silica fume

Chemical properties	Value	Physical properties	Value
SiO_2 (%)	90-95	Color	Grey
C (%)	0.8-3	Diameter (µm)	<1µm
Loss on ignition (%)	1.5-2.5	Specific gravity	2.2
		Surface area (m^2/g)	15-30

2- 2- Sample Preparation

To fabricate the specimens, different percentage of silica fume (SF) at 5, 10 and 15% of dry weight of soil (3 samples) and nano-silica (NS) at 1, 2 and 3% of dry weight of soil (3 samples) were added to the kaolinite clay. Silica fume was mixed with the kaolinite clay under the dry condition as it is commonly applied by other researchers [7, 9, 19]. The nano-silica were added to the required amount of water then prepare a homogeneous solution of them by an ultrasonic bath device. The prepared mix then was sprayed on the different samples to exchange moisture among the particles, forming a homogeneous blend and preventing agglomeration of the nano-particles. The specimens for Atterberg limits tests and proctor compaction test were prepared through the ASTM D4318 [21] and ASTM D698 [24], respectively. Cylindrical specimens (38-mm diameter and 76-mm height) for the unconfined compressive strength tests were prepared at their optimum water content and compacted to 95% of maximum dry density. The compacted specimens were removed from molds and wrapped in plastic bags individually to prevent moisture change for 7 and 28 days. Specimens for soaked California bearing ratio tests were compacted in the CBR mold with compaction energy similar to Proctor compaction test. Finally, prepared specimens were cured inside the plastic bag for 7 days to prevent moisture loss.

2- 3- Test Methods

The main objective of this study is to investigate the effect of silica fume and nano-silica on the geotechnical properties of the kaolinite clay. To elaborate the goals of research the following test methods were conducted:

- Atterberg limits tests were carried out on mixed soils according to ASTM D4318 [21].
- The effect of SF and NS on the optimum moisture content (OMC) and maximum dry density (MDD) of kaolinite clay were examined through the standard proctor test, which is regulated by ASTM D698 [24].
- Unconfined compressive strength tests were conducted on compacted specimens after curing according to ASTM D2166 [25].
- The soaked CBR test was carried out in accordance with ASTM D1883 [26] after 7 days of curing.
- The micro-analysis and failure analysis of fabricated soils were evaluated by Scanning Electron Microscopy (SEM). Three specimens (raw soil, soil+15%SF, and soil+3%NS) cured at 28 days were studied by using SEM analysis. The samples were obtained from the central part of the specimens.

3- Results and discussion

3- 1- Atterberg limits

To evaluate the effects of the silica fume and nano-silica on the plasticity characteristics of the soil, the Atterberg limits test was conducted. Figures 2 and 3 show the mean liquid limit (LL), plastic limit (PL) and plasticity index (PI=LL-PL) of three replicates for each specimen. It can be seen that the plasticity limit for specimens with SF is more than that of the raw soil. However, the liquid limit and plasticity index of kaolinite clay were slightly decreased after addition of SF. This could be dependent on the soil type and associated the cation exchange capacity [7, 27]. These results were in agreement with the previous studies [7, 10]. In this regard, Kalkan and Akbulut demonstrated that the liquid limit and plasticity index of clay soil decrease by increasing the silica fume content up to 50% [7]. Furthermore, it has been shown that plasticity index of smectite clay slightly decreases as the silica fume content increases [10]. Decrease in the LL and the PI due to that SF coats and binds all clay particles which possess little cementitious value and large particles which called the pozzolanic reaction between SF and aluminous material [9].

Given the testing results which is shown in Figure 3, the PL slightly increased initially but then decreased when nano-silica content increased. It can be attributed to agglomeration of nano-particles when the dosage of nano-silica exceeded 1% [28]. Silica nano-particles have high specific surface due to their very small size. A high specific surface material would increase the wettable surface area and the amount of water adsorbed [29]. These properties can lead to increase the liquid limit. As a result of increasing LL and reducing PL, plasticity index increases.

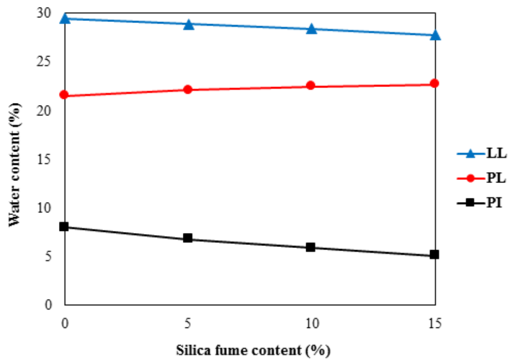


Figure 2. Variation of LL, PL, and PI of the silica fume–clay samples

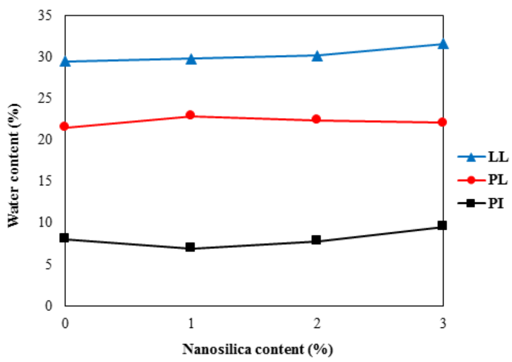


Figure 3. Variation of LL, PL, and PI of the nanosilica–clay samples

3- 2- Compaction characteristics

The compaction behavior of soil+SF and soil+NS is illustrated in Figures 4 and 5, respectively. These figures obtained from five specimens that were tested for each percentages of stabilizers. These figures indicated that addition of SF and NS reduces the maximum dry density and increases the optimum moisture content. The maximum dry densities ranged from 16.5 to 16.8 kN/m3 for soil mixed with SF and from 16.7 to 16.9 kN/m3 for soil stabilized with NS. The reduced MDD result from replacement of soil of higher specific gravity with material with lower specific gravity. Furthermore, silica fume and nanosilicanano-silica increase surface area of samples compared to raw soil. This implies that more water is needed in order to compact the mixtures and then the OMC is increased. Similar trends regarding the impact of SF and NS on the clay soils' compaction have been observed by other researchers [7, 9, 18]. The high optimum water content and the low dry unit weights occurred in 50% silica fume–clay composite samples. The amount of silica fume and the chemical composition of the clay minerals considerably affect the significance of these changes [7]. About the effect of nanosilicanano-silica on the clay soil, it has been indicated that nanosilicanano-silica cause the chemical pozzolanic reaction to increase. This reaction can lead to need the higher amount of water and cause the more amount of water is required. Moreover, the low density of nanosilicanano-silica (30-50 gr/lit) can be resulted in the maximum dry density of the soil to decrease [18].

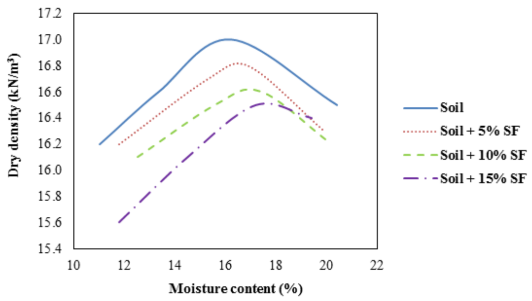


Figure 4. Moisture-density relationships of soil and soil+SF

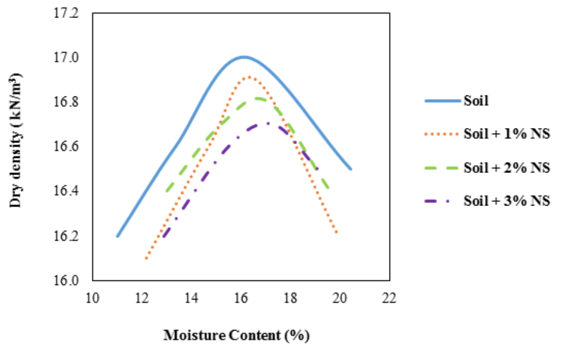


Figure 5. Moisture-density relationships of soil and soil+NS

3- 3- Unconfined compressive strength (UCS)

The effect of silica fume and nanosilicanano-silica on the unconfined compressive strength of the kaolinite clay was investigated as a function of the additive content and curing period. These measurements for three replicates of each sample were averaged out and presented in Figures 6 and 7. It can be concluded that silica fume led to an increase in the UCS. Addition of 15% SF increased the compressive strength of kaolinite clay from 129 to 162 kPa and 220 kPa, at 7 and 28 days curing period, respectively. This can be attributed to the internal friction of silica fume particles and chemical reaction between silica fume and kaolinite clay materials [7]. The UCS results of samples with NS showed that by increasing the NS content, the unconfined compressive strength increases. A similar trend for soil with nanosilicanano-silica has reported by Haeri et al. [15] and Mostafa et al. [30]. Conclusively, with an addition of SF and NS at 7 and 28 days corresponding strain to peak axial stress decreased. Therefore, samples with SF and NS are more brittle compared with raw soil.

One of the major deterioration in flexible pavements constructed on week subgrade is depression. Soil stiffness is a complicated phenomenon can play an important role in this failure. As the non-linear elastic behavior of commonly used soils, the modulus of soils is different from each other. Considering the aforementioned explanations, the elastic modulus of kaolinite clay stabilized with SF and nanosilicanano-silica must be properly evaluated. A common method to determine the soil elastic modulus is using the tangent modulus of the stress–strain curve that obtained from unconfined compression tests. As an alternative method to find this parameter, a secant modulus that determined for 50% of peak axial stress (the stress level estimated to occur in the field) can be utilized. For further details on calculation of soil stiffness interested reader is referred to Figure 8. Figures 9 and 10 show the effect of SF and NS content on the modulus of elasticity at 50% of peak stress, respectively. It can be observed that modulus increases significantly with SF and NS content. Therefore, stabilizing the kaolinite clay with SF and NS will minimize a settlement in week soils.

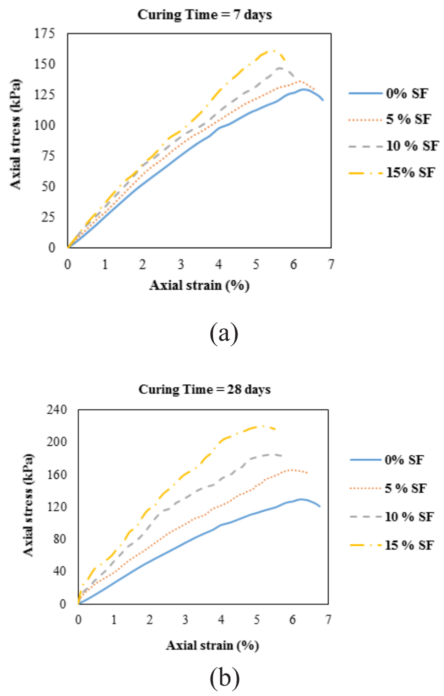
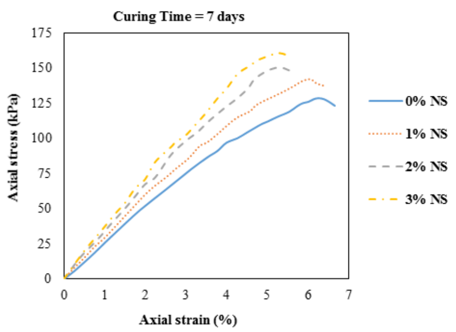
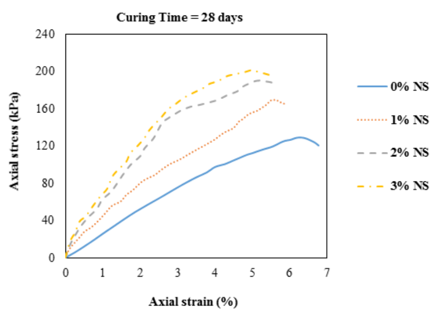


Figure 6. Effect of the addition of silica fume on UCS (a) after 7 days (b) after 28 days



(a)



(b)

Figure 7. Effect of the addition of nanosilica on UCS (a) after 7 days (b) after 28 days

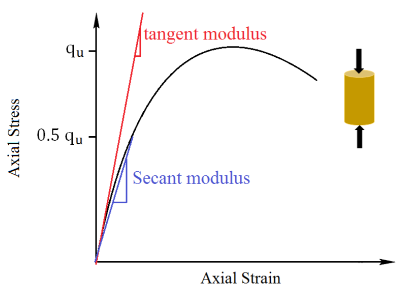


Figure 8. Tangent and secant modulus determination for soil

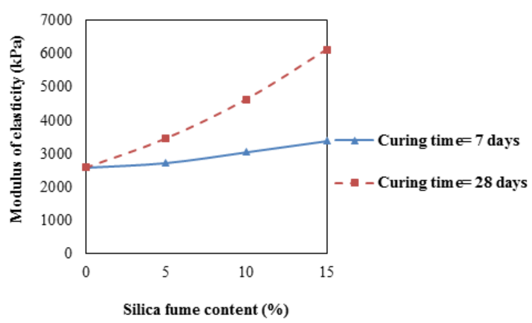


Figure 9. Effect of the addition of silica fume on the modulus of elasticity of kaolinite clay

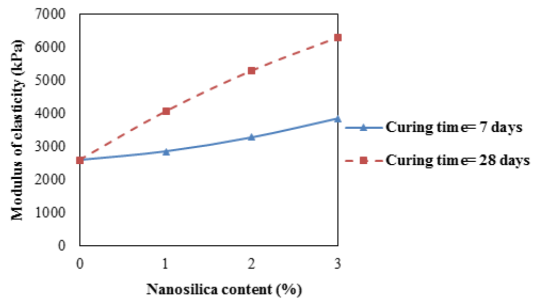


Figure 10. Effect of the addition of nanosilica on modulus of elasticity of kaolinite clay

3- 4- California Bearing Ratio (CBR)

The CBR value is one of the common tests used to evaluate the strength of soils in the design of pavement courses. Three samples from each soil mixture have been tested at soaked conditions after 7 days of curing time. The values of CBR results are averaged out for three replicates and presented in Figure 11. The consequences of the soaked CBR test are in agreement with those of the unconfined compressive strength tests. It is evident from Figure 11 that the addition of 15% silica fume and 3% nano-silica leads to an increase in CBR from 3% to 6.6% and 6.8%, compared with the raw soil. The increases in CBR ratio of soil+SF and soil+NS can be attributed to the formation of flocculation products in bonding between the clay particles.

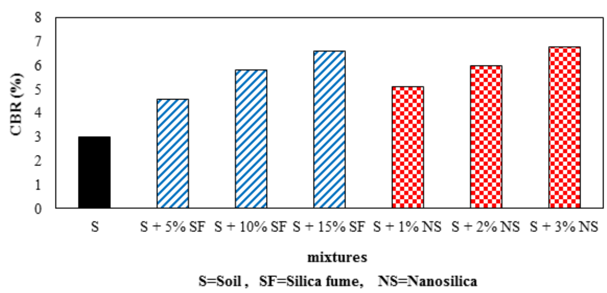
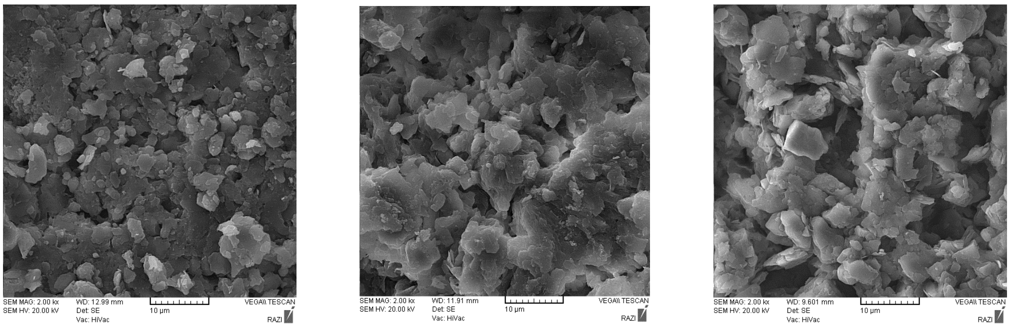


Figure 11. CBR values of the soils with varying silica fume and nanosilica contents

3- 5- SEM analysis

The micro-structural changes in the compacted kaolinite clay due to SF and NS addition after 28 days curing time were analyzed using scanning electron microscope (SEM). Figure 12a shows SEM micro-graphs of the compacted kaolinite clay at a high magnification. It shows hexagonal clay flakes with a discontinuous structure and large voids are exist in the raw soil. Moreover, Figure 12b demonstrates that clay particles were covered by SF and micro-pores were filled. This can cause to flocculation products formation in the vicinity of clay particles which leads the contact and cohesion between clay particles to increase. Therefore, the improvement in compressive strength and CBR tests can be attributed the frictional strength to increase caused by this phenomenon. The specimen with NS has denser matrix than the raw soil and most of the voids were filled (Figure 12c). According to the SEM images, nanosilicanano-silica can reduce the size of flocculation products at the interface between the particles more effectively compared with SF [29].



(c) (b) (a)

Figure 12. Scanning electron micrograph of specimens (a) kaolinite clay (b) kaolinite clay+15% SF (c) kaolinite clay+3% NS

3- 6- Statistical analysis

A statistical analysis is done in this research in order to investigate if the addition of stabilizers is statistically significant in strength characteristics of kaolinite clay. For this purpose, multi-way analysis of variance (ANOVA) that also known as multi-factor ANOVA tests with replication was conducted through Minitab 2018 software. The CBR value and UCS at 7 days curing time along with UCS at 28 days curing period were chosen as response variables to examine the effect of stabilization on the performance responses. Moreover, 0.05 (α=0.05) as a commonly used level in statistical analysis was selected as a significance level to study the null hypothesis which states that all the treatment means are statistically the same. This means that there is a 5% risk of concluding that an effect exists when there is no actual effects existing. P-values lower than 0.05 reject the null hypothesis and provide statistical evidence that the selected parameter is significant. Furthermore, based on the objectives of this research the Tukey-Kramer method was employed to compare all possible pairs of means and find the means that are significantly different from each other.

The consequences of the statistical analysis on the strength testing results are shown in Table 4. Each evaluated factor has seven levels including raw soil (s), soil+SF (SF5, SF10, SF15), and soil+NS (NS1, NS2, NS3). As mentioned before, three replicates were tested and the results were averaged out and presented in the figures. These replicate data were used to perform the statistical tests. As the P-values in Table 4 suggest, CBR and UCS test results were affected by stabilization. As Tukey-Kramer statistical test says, the strength parameters of stabilized soils are significantly higher than that of the raw soil. Moreover, the consequences of statistical analysis infer that CBR and UCS test results at 7 days curing time are consistency. About the comparison between different stabilizers, it can be concluded that there is no significant difference between SF10 and NS2, SF5 and NS1. Although there is no significant difference between SF15 and NS3 based on the test results from 7 days curing time, the SF15 considerably affect the unconfined compressive strength of kaolinite clay after 28 days curing period than that of the NS3. This can be attributed to considerable contribution of silica fume to the compressive strength of the soil at later ages which was observed in the concrete [31].

4- Conclusions

In this research, the effect of silica fume and nanosilicanano-silica addition to the kaolinite clay was explored in terms of engineering properties. To this end, the experimental tests including Atterberg limits, standard proctor, California bearing ratio at 7 days curing period, and unconfined compressive strength at 7 and 28 curing time were conducted. Moreover, the SEM micro-graph was carried out to investigate the micro-structural characteristics of stabilized soils. Considering the discussions and the experimental results, the following conclusions are drawn:

- The addition of silica fume slightly decreased both the liquid limit and the plasticity index and increased the plasticity limit of kaolinite clay. NanosilicaNano-silica initially decreased the Plasticity index (at 1% NS) and then increased PI at higher percentages of additives.
- The maximum dry density (MDD) and optimum moisture content (OMC), decreased and increased, respectively, with increasing content of silica fume and nanosilicanano-silica. Silica fume and nanosilicanano-silica increase surface area of samples compared to the raw soil which increases the water absorption.
- The addition of Silica silica fume and nanosilicanano-silica led to increase the unconfined compressive strength of specimens. The UCS of the specimens with 15% SF and 3% NS was improved up to 70% and 55% after 28 days curing time. Compressive strength increases with increasing curing age. The modulus of elasticity (E_{50}) changed in a similar trend as that of unconfined compressive strength. Samples with SF and NS are more brittle compared to the raw soil.
- With the addition of 15% silica fume and 3% nanosilicanano-silica, cured in 7 days, the CBR values of specimen increased about two times more than the raw soil.
- Based on the statistical analysis, it can be concluded from CBR and UCS test results that there is no significant difference between 10% SF and 3% NS as well as 5% SF and 1% NS. Moreover, 15% SF significantly affect the unconfined compressive strength of kaolinite clay after 28 days curing period than that of the 3% NS.
- The SEM micro-graphs inferred that silica fume and nanosilicanano-silica can change micro-structure of kaolinite clay and fill the voids in samples which led to enhance the compressive strength.

Table 4. Statistical analysis on test results.

Test	ANOVA Test Results					Tukey-Kramer Test Grouping Results						
	Factor	Adj. MS	F-Value	P-Value								
CBR - 7	Stabilization	5.20429	79.2	0.000		NS3	A					
				NS3	0.000	SF15	A	B				
				SF15	0.000	NS2		B	C			
				NS2	0.001	SF10			C	D		
				SF10	0.014	NS1				D	E	
				NS1	0.038	SF5					E	
				SF5	0.000	Raw						F
UCS - 7	Stabilization	432.158	48.01	0.000		SF15	A					
				NS3	0.000	NS3	A					
				SF15	0.000	NS2		B				
				NS2	0.060	SF10		B				
				SF10	0.661	NS1		B	C			
				NS1	0.011	SF5			C	D		
				SF5	0.000	Raw				D		
UCS - 28	Stabilization	2479.04	150.89	0.000		SF15	A					
				NS3	0.000	NS3		B				
				SF15	0.000	NS2		B	C			
				NS2	0.001	SF10			C			
				SF10	0.047	NS1				D		
				NS1	0.000	SF5				D		
				SF5	0.000	Raw					E	

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