RESEARCH PAPER

Influence of nano-material on the expansive and shrinkage soil behavior

Mohd Raihan Taha · Omer Muhie Eldeen Taha

Abstract This paper presents an experimental study performed on four types of soils mixed with three types of nano-material of different percentages. The expansion and shrinkage tests were conducted to investigate the effect of three type of nano-materials (nano-clay, nano-alumina, and nano-copper) additive on repressing strains in compacted residual soil mixed with different ratios of bentonite (S1 = 0 % bentonite, S2 = 5 % bentonite, S3 = 10 % bentonite, and S4 = 20 % bentonite). The soil specimens were compacted under the condition of maximum dry unit weight and optimum water content (w_{opt}) using standard compaction test. The physical and mechanical results of the treated samples were determined. The untreated soil values were used as control points for comparison purposes. It was found that with the addition of optimum percentage of nano-material, both the swell strain and shrinkage strain reduced. The results show that nano-material decreases the development of desiccation cracks on the surface of compacted samples without decrease in the hydraulic conductivity.

Keywords Nano-material · Nano-copper · Nano-alumina · Desiccation crack · Volume change

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Introduction

Desiccation causes clayey soils to shrink. This occurs due to the loss of soil water content which developed matric suction in soil that increases the shrinkage strain in the soil and normally followed by cracks (Nahlawi and Kodikara 2006). According to the Albrecht and Benson (2001) the soil, for example in the area of landfill linear should be compacted at the maximum dry density (max. γ_d) and optimum water content (w_{opt}), to achieve the lower shrinkage strain after saturation.

An important parameter related to desiccation is hydraulic conductivity. In general, for soil liner construction, the soil should be compacted to achieve a hydraulic conductivity of 1×10^{-9} m/s or less. So the acceptable zone of water content and dry unit weight that yielded lower hydraulic conductivity is the line of optimums and some parts of wet side of it. The volume change increase varied greatly between the dry and wet sides of the line of optimum where it increases towards the wet side and decreases on the dry side (Daniel and Wu 1993).

One of the methods to reduce the shrinkage strain and hydraulic conductivity is by increasing the compaction efforts. The increase in the compaction effort increases the dry density and decreases the w_{opt} but for higher water content, the increase in the compaction effort does not reduce the shrinkage strain significantly (Osinubi and Nwaiwu 2008). Another method to reduce the shrinkage is to use additives.

Mixing clayey soils with sand is one of the cheapest methods. The addition of sand effectively decreases the shrinkage while it increases the hydraulic conductivity. The same behavior was observed when lime was used as an additive material (Omidi 1993). Another material used to reduce the shrinkage strain is fibers. Mixing the soil with fiber can reduce the shrinkage strain up to 90 % and increase strength of soil up to 70 % (Kumar et al. 2006) but the fiber increase the hydraulic conductivity up to 50 times the hydraulic conductivity of the soil without fiber (Rifai 2000).

According to Nalbantoğlu (2004), fly ash is one of the treatment materials used to reduce soil swell pressure and soil cation exchange capacity with considerable environmental benefits, reducing land, air, and water pollution. However, treatment by fly ash bring the soil to a more granular nature and decrease the water absorption potential.

Omidi (1993) found out that cement decreases both shrinkage strain and hydraulic conductivity. Treatment by cement also provide some protection from the adverse effect of saturation on strength (Miller and Azad 2000). The ideal soil for cement treatment should have a plasticity index between 5 and 15 (Walker 1995). Soils with a plasticity index above 20–25 are not suited to cement stabilization due to problems with excessive drying shrinkage.

The difference between the particle size contributes to inter particle filling or interlayer filling, which reduce the void ratio. Based on Montesh (2005), Na-saturated bentonite has a swelling potential greater than Ca-saturated bentonite, while the raw bentonite has a swelling potential between Na and Ca because of the raw bentonite have mixed Na–Ca interlayer filling.

The development of nano-materials could result in considerable benefits since the material have shown to give significant advantages especially in concrete technology. According to Li et al. (2006), in cement composites nano-alumina (Al_2O_3) filled up the pores of loose net structure around the sand particle, and decreased the porosity and increased the density of the interfacial transition zone during the formation of C–H–S (C-CaO, S-SiO_2, and H-H_2O) gel. As a result, the elastic modulus of mortars increased. Figure 1 shows the hydration of mortars with and without nano-alumina. Around the aggregate, cracks can be observed at contact area between aggregate and cement paste (Fig. 1a).

However, there is a dense zone with noticeable cracks around the aggregate upon the addition of 5 % mortars (Fig. 1b).

In addition, at nanometer scale, the contacting surfaces can be nearly ideal so that comparable effects are not partially averaged out by many differently oriented domains. It has been shown that friction can vary over an order of magnitude depending on the relative arrangement of the contacting surfaces. In some cases, the particle is restricted to move in one direction. In other cases, the particle rolls, rotates in-plane or slides, which can increase shear forces (Falvo and Superfine 2000).

The expansive and shrinkage strains in the soil causes cracks which increase the permeability of soil, and the soils with high permeability has less strength to resist sliding.

The main objectives from this research were to reduce the volumetric strains (shrinkage and expansive strains) and reduce the crack intensity factor (CIF) of the soil without increase in the hydraulic conductivity of the soil.

Materials

Bentonite

Bentonite used in the present investigation is a high swell sodium bentonite containing sodium montmorillonite and its properties are listed in Table 1. In general, it has a specific gravity of 2.655, plasticity index of 394.6, and cation exchange capacity of 90 meq/100 g.

Soil

The soil is a residual soil taken from National University of Malaysia campus in Bangi, Selangor, Malaysia. This soil is termed as UKM soil. The soil was sampled from 0.5 to 1 m below the ground surface. The UKM soil was mixed with different ratios of bentonite (S1 = 0 % bentonite, S2 = 5 % bentonite, S3 = 10 % bentonite, and S4 = 20 % bentonite) to produce four types of soil samples with different plasticity index. The physical and chemical properties of UKM soil (S1) and other type of mixed soil samples are listed in Table 2.

(a) without nano-alumina
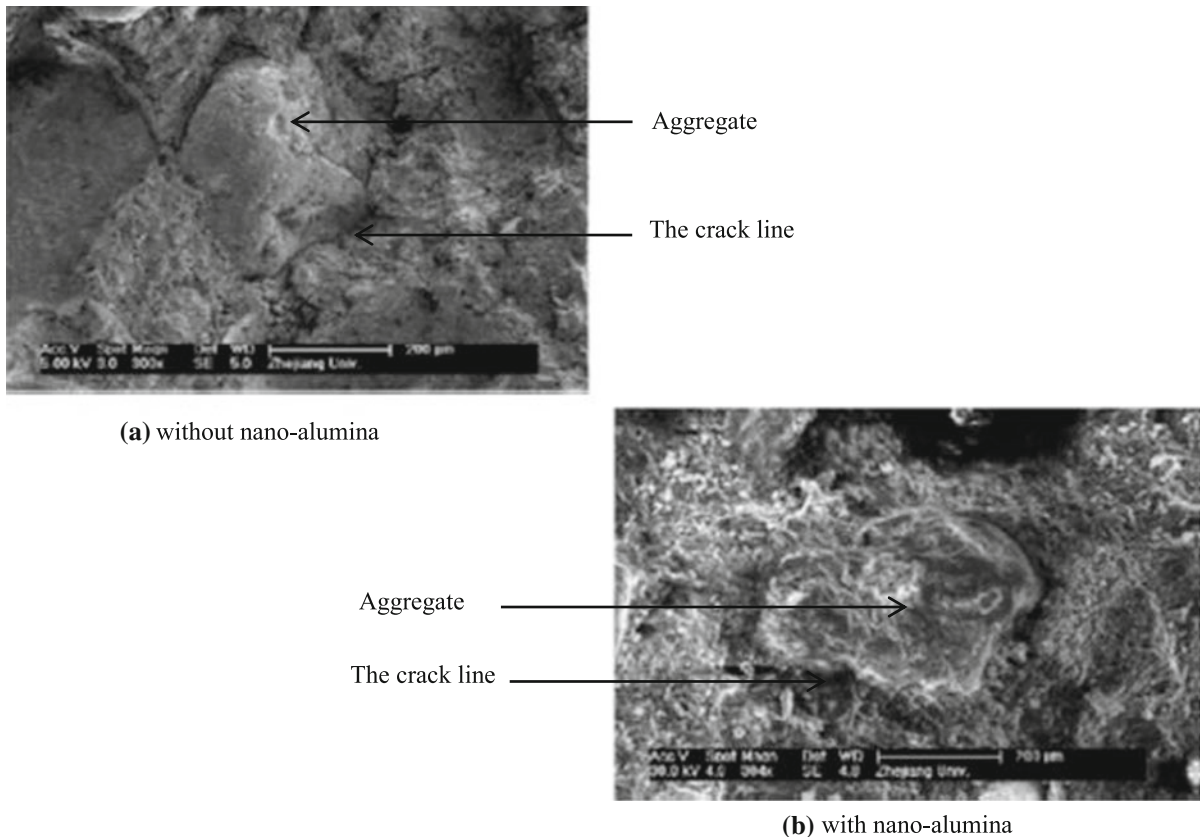

(b) with nano-alumina

Fig. 1 SEM image of mortars hydrating for 28 days (after Li et al. 2006)

Nano-materials

Three types of nano-materials used in this study, i.e., nano-clay, nano-alumina, and nano-copper. Some properties of the nano-materials are discussed below.

Nano-clay

The nano-clay material was supplied by Southern Clay Products, Gonzales, TX, USA with the product known as Nanoclay Cloisite® Na⁺. The specification and all the information provided by the company is shown in Table 3. A scanning electronic microscope (SEM) shows the nano-clay layers (Fig. 2). The measured size was 5–15 nm thickness and 20 nm to 10 μm diameter.

Nano-copper and nano-alumina

The other nano-materials used in this study were ultrapure gamma-alumina (γ-Al$_2$O$_3$) powder with

purity of 99.99 % and nano-copper oxide with purity of 99.99 %, supplied by Inframat Advanced Materials, Manchester, CT, USA. Its general properties are shown in Table 4.

Preparation of soil–nano-material mixtures

The amount of nano-alumina selected was 0, 0.05, 0.075, 0.1, 0.15, and 0.3 % of the total dry weight of the soil, while the amount of nano-copper was 0, 0.15, 0.3, 0.5, and 0.7 % of the total dry weight of the soil. The nano-clay was used only with UKM soil (S1) because the other type of soil already had montmorillonite (bentonite) as one of its constituents. The nano-clay content was 0.0, 0.05, 0.1, 0.15, 0.3, and 0.5 % of the total dry weight of the soil. The reason for using different amounts for different nano-materials was to take the advantages of different particle density and size of the nano-materials. The amount of nano-material was expressed by percent multiply by total

Table 1 Properties of bentonite

Property	Value
Specific gravity	2.655
Liquid limit	464.6
Plastic limit	70.0
Plasticity index	394.6
Cation exchange capacity	90.0 meq/100 g of dry soil
Chemical composition	

Formula	Concentration (%)
SiO_2	60.85
Al_2O_3	14.82
Fe_2O_3	4.38
CaO	3.67
Na_2O	3.13
MgO	3.09
K_2O	0.79
TiO_2	0.61
Other	0.44
Heat loss	8.22

Table 2 Physical and chemical characteristics of the soils

Characteristics	Values and descriptions			
	S1	S2	S3	S4
Specific gravity	2.607	2.609	2.612	2.617
Plasticity index (%)	16.96	28.255	36.182	69.01
Linear shrinkage (%)	8.2	12.86	15.9	23.57
Passing no. 200 sieve (%)	47.16	49.802	52.444	57.728
Clay content (<2 μm) (%)	18	22.9	28.5	38.5
Unified soil classification system (USCS)	CL	CL	CH	CH
Compaction properties				
Optimum water content (%)	14.29	16.013	18.625	20.3
Maximum dry unit weight (kN/m³)	18.05	17.697	16.93	16.117
Chemical compositions				
Compound				
SiO_2 (%)	62.07	62.86	55.96	54.62
Al_2O_3 (%)	29.46	25.81	21.46	23.06
Fe_2O_3 (%)	5.70	5.48	5.21	5.18
MgO (%)	0.58	1.45	1.51	1.11
CaO (%)	0.03	1.23	1.54	1.00
TiO_2 (%)	1.17	1.04	0.96	0.94
Na_2O (%)	–	1.01	1.17	0.77
K_2O (%)	0.76	0.78	0.74	0.67
Other	0.05	0.14	0.37	0.30
Heat loss	0.18	0.2	11.08	12.35

dry weight of soil. Thus, the content of nano-copper was more than nano-alumina because the particle density and particle size of nano-copper was greater than nano-alumina (Table 4). Furthermore, the nano-alumina optimum content was less than 0.3 % while for nano-copper the optimum content was equal or greater than 0.3 % for all types of soil.

Two mixing methods were examined. In the first method, the nano-materials were mixed with required quantity of water and then the mixture was put in a beaker. The beaker was then placed in an ultrasonic bath of water for 2 h as was suggested by Wei et al. (2002). After ultra-sonication, the suspension was mixed with the soil. However, it was too difficult to obtain a homogeneous mixture of the soil–nano-material mixture by this method. In the second method, the dry soil was mixed with nano-materials and then the water was added to the mixture. In this method, mixing is carried out into two stages. Initially premix or hand mixed (To et al. 2011), the quantity of soil was divided into ten layers and each layer was sprayed with the required amount of nano-material. Each layer was mixed alone then put in the pot and then mixing the mixture by horizontal cylindrical mixer for at least 3 h (Jones et al. 2007). This procedure was found to be the best method to obtain

homogeneous samples since homogeneous color was obtained after compaction as shown in Fig. 3.

The dry mixtures were mixed with the required amount of water to obtain the optimum standard Proctor moisture content and maximum dry density. All mixing was done manually, and proper care was taken to prepare homogeneous mixtures at each stage. Moreover, the type of water used for preparing samples was distilled water.

Test procedures

Initially the soil–nano-material mixture was mixed with water and left for curing then the X-ray diffraction (XRD) test was carried out to check if there is a chemical reaction for nano-material on the soil or not. SEM image indicated that there was no gel or fume inside the sample. The same observation was obtained

Table 3 Properties of the nano-clay (nanoclay Cloisite® Na+)

Parameter	Values	Comments
Physical properties		
Specific gravity	2.86 g/cc	
Loss on ignition	7.00 %	
Particle size	≤2.00 μm	10 %
	≤6.00 μm	50 %
	≤13.0 μm	90 %
Mechanical properties		
Hardness, shore D	83	5 % Cloisite® reinforced nylon 6
Tensile strength, ultimate	101 MPa	5 % Cloisite® reinforced nylon 6
Elongation at break	8.00 %	5 % Cloisite® reinforced nylon 6
Modulus of elasticity	4.657 GPa	5 % Cloisite® reinforced nylon 6
Flexural modulus	3.78 GPa	5 % Cloisite® reinforced nylon 6
Izod impact, notched	0.270 J/cm	5 % Cloisite® reinforced nylon 6
Thermal properties		
Deflection temperature at 0.46 MPa (66 psi)	96.0 °C	5 % Cloisite® reinforced nylon 6
Processing properties		
Moisture content	≤2.00 %	
Descriptive properties		
Modifier concentration, meq/100 g clay	92.6	
X-ray diffraction d-spacing (001)	11.7 Å	
Color	Off white	
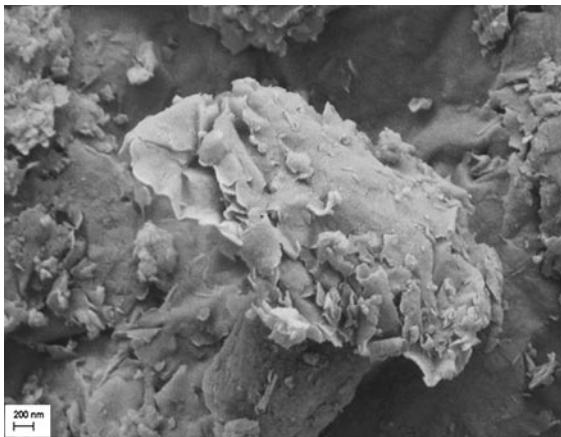
Fig. 2 Nano-clay Cloisite® Na+ layers

by XRD test as shown in Fig. 4. The curves of sample S2 and sample S2 + 0.5 % nano-alumina were both identical. This is because of the nano-material used in this study was insoluble material, and cannot react with soil material by just mixing with water.

The experimental procedures involved determining compaction behavior of unamended soil and soil

Table 4 Properties of gamma aluminum oxide (alumina, Al_2O_3) and copper oxide (CuO) nano powders

Property	Value	
Formula	Al_2O_3	CuO
Particle density (gm/cm³)	3.6	6.3–6.49
Surface area (m²/g)	>150	N/A
Average particle size (nm)	20–50	100
Solubility in water (%)	Insoluble	Insoluble
Melting point (°C)	NE	1,326
Composition		
Al_2O_3	≥99.99 %	N/A
CuO	N/A	99.99 %
Appearance and odor	White to off-white Oderless	Black powder Oderless
Crystal structure	Transitional gamma	N/A

mixed with nano-materials. The compaction behavior curves were determined according to the standard test method (ASTM D698) for both unamended soil and

Fig. 3 Color of soil samples after compaction, the sample with *light color* is S3 and the sample with *dark color* is S3 with 0.5 % nano-copper
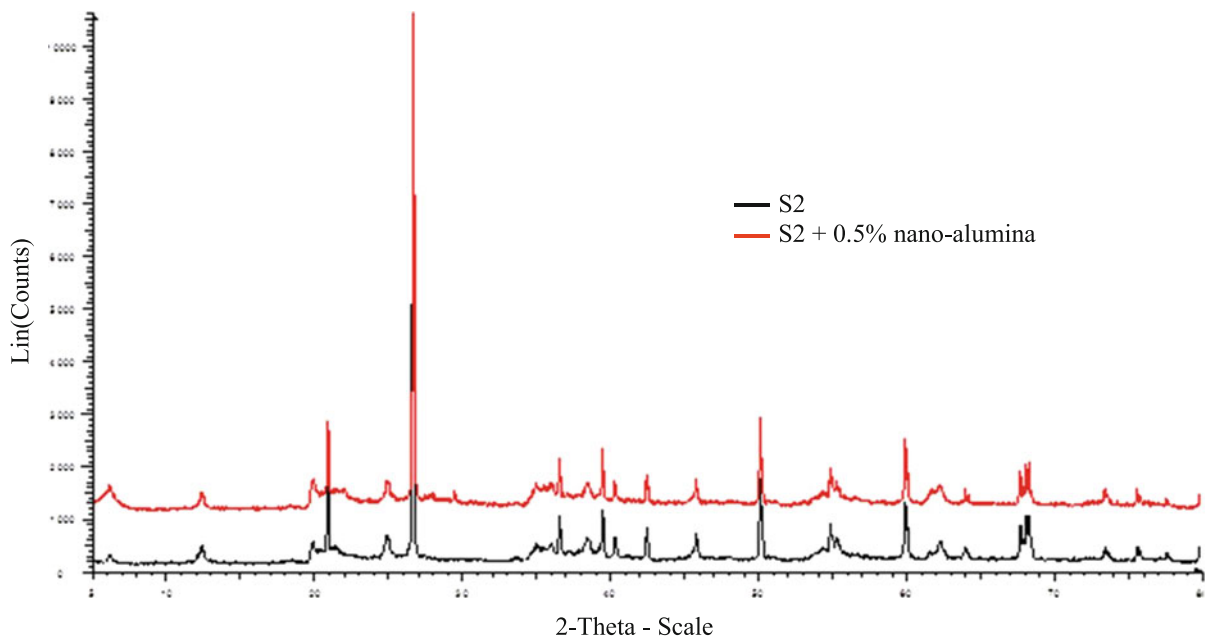

Fig. 4 XRD test

soil with nano-materials. The soil used for compaction was dried in an oven and crushed with a rubber hammer until it passed the US No. 4 sieve. The soils were moisten with tap water using a spray bottle, and stirred with a trowel during mixing to ensure an even distribution of water. Then the soils were sealed in plastic bags and allowed to hydrate for at least 24 h prior to compaction.

Compacted samples were used for the shrinkage strain, expansive strain, and hydraulic conductivity tests. Volume change measurement was made to evaluate shrinkage and expansive behavior. For

hydraulic conductivity tests, the specimens were either immediately placed in permeameters for saturation or wrapped in plastic to prevent drying until they could be saturated.

After compaction, the soil samples were divided into two group. The first group was saturated with water to measure the expansive strain and the second group was left to dry in an oven temperature $\sim 34 \pm 2$ °C to measure shrinkage strain.

Saturation procedure for hydraulic conductivity and expansive strain measurement

Saturation was conducted by soaking the specimens in flexible-wall permeameters using a hydraulic gradient of 10 and an average effective stress of 10.5 kPa following ASTM D 5084. The specimens were permeated with tap water until the ratio of inflow to the outflow was between 0.75 and 1.25, and the hydraulic conductivity was steady. This procedure resulted in specimens saturated close to field condition. When the specimen was removed from the permeameter, measurements were made of its height, diameter, and weight.

Test method for volumetric strain and crack intensity factor

For the desiccation crack test, soil specimens were prepared at max. γ_d and w_{opt}. The drying process was conducted for a period of more than 10 days depending on the sample type. The weight, height, and diameter of each specimen was then measured. The volume change was used to determine the volumetric shrinkage strain of the soil specimens. The volumetric shrinkage strain is defined as the change in volume (ΔV) to the total volume of the soil specimens (V, %), expressed by

$$\text{volumetric shrinkage strain} = \frac{\Delta V}{V} \times 100\,\%. \qquad (1)$$

The surficial dimensions of cracks were measured in order to find the CIF to evaluate the magnitude of desiccation cracks developed in the soils, expressed by (Harianto et al. 2008)

$$\text{CIF} = \frac{A_c}{A_t}, \qquad (2)$$

where A_c is the desiccation crack area and A_t is the total surface area of soils.

Results and discussion

The effect of nano-clay on UKM soil

The influence of nano-clay on the max. γ_d and w_{opt} are shown in Fig. 5. There is a slight decrease in w_{opt} and slight increase in max. γ_d, which indicated that the optimum percentage of nano-clay is 0.1 %. Figure 6 shows that the increase in nano-clay causes an increase in plasticity index and linear shrinkage. This happened because the nano-clay material is an expansive clay. The shrinkage strain slightly decreased at 0.05, 0.1, and 0.15 % nano-clay content then increases beyond this amount. While there was no increase in expansive strain for the nanoparticle contents mentioned. When nano-clay content is increased the expansive and shrinkage strain increases. However, the total volume change was also observed to increase. The total shrinkage strain after saturation was measured relative to the final volume of sample when become fully dry. The total shrinkage strain is the total volume change and approximately equal to the shrinkage volume change and expansive volume change. The increase in nano-clay content of more than 0.3 % gives an increase in total volume change as shown in Fig. 7.

The effect of nano-alumina and nano-copper on the plasticity index

A small positive effect can be observed on the plasticity index of the soil mixture. The addition of nano-alumina causes a small decrease in the plasticity index while an increase in nano-copper content gives higher decrease in the plasticity index (Figs. 8, 9). This happened because of the nano-alumina and nano-copper particles have higher density than clay
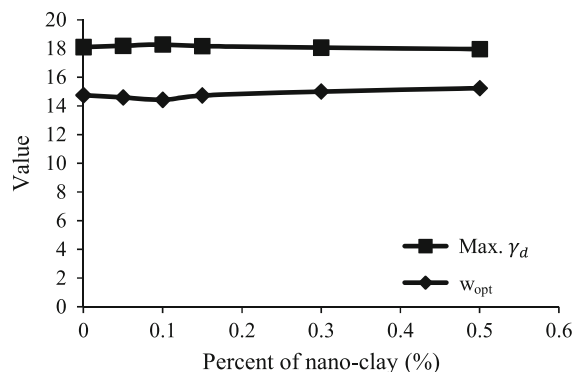

Fig. 5 Effect of nano-clay on the max. γ_d and w_{opt}
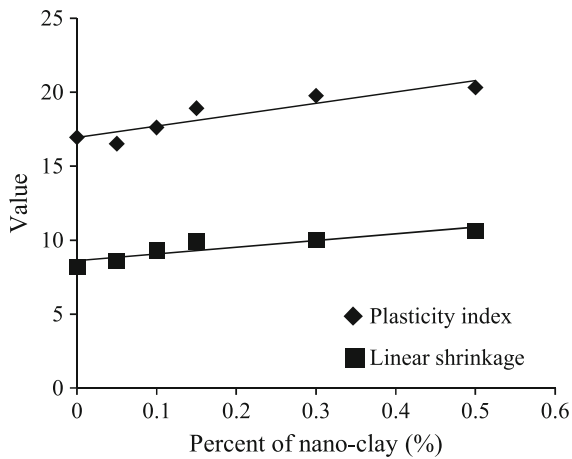
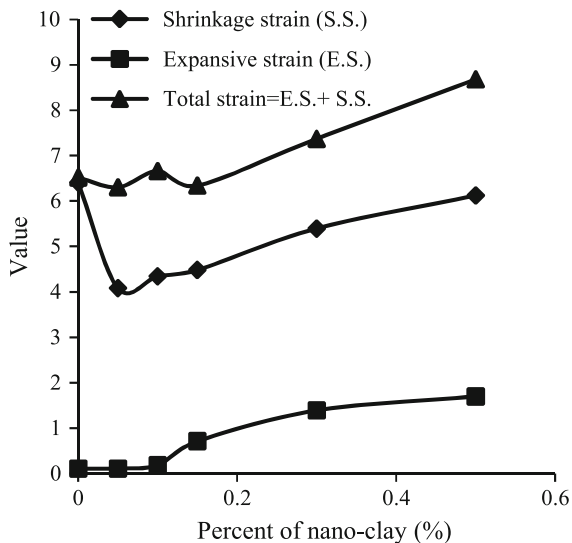Fig. 6 Effect of nano-clay on the plasticity index and linear shrinkage

Fig. 7 Effect of nano-clay on the shrinkage, expansive and total soil strains

Fig. 8 Effect of nano-alumina on the plasticity index

Fig. 9 Effect of nano-copper on the plasticity index

particles, as well as the surface area of nano-alumina is less than bentonite particles (nano-clay) surface area.

The effect of nano-alumina and nano-copper on the compaction conditions

There is no significant effect of nano-alumina on the w_{opt} for S1, S2, and S3 soils for the amount of nano-alumina tested (Fig. 10). However, some measurable changes occurred for S4 soil (20 % bentonite) with a decrease in w_{opt} at 0.1 % and increasing w_{opt} beyond

this. For tested specimens with nano-copper, minimal changes occurred on the w_{opt} as the amount of nano-copper increases (Fig. 11). For the max. γ_d, a greater increase was observed with the addition of nano-copper compared to nano-alumina (Figs. 12, 13). This is because the particle density of nano-copper is greater than the particle density of nano-alumina. However, beyond the peak, increase in nano-materials results in decrease in density and increase in the w_{opt}.

The increase of nano-material more than the optimum limit may possibly result from agglomeration in nano-material particles which in turn causes an increase in the void ratio then decrease in density and increase in water content. According to Ferkel and Hellmig (1999), the agglomeration of nanoscaled powders increase the amount of necks between particles and therefore decreases the density of green bodies. Figures 14 and 15 show the soil samples S3 which was mixed with 0.5 and 0.7 % nano-copper. The nano-copper particle agglomerated when the nano-copper content was increased. Soil sample S4 shows similar behavior (Figs. 16, 17).
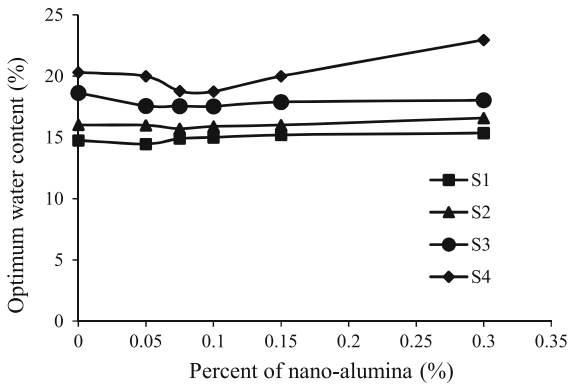
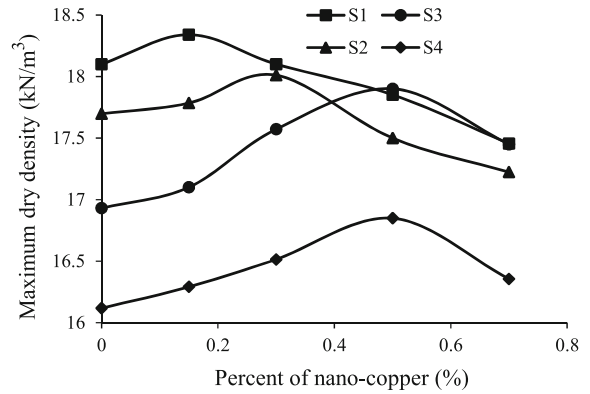**Fig. 10** Effect of nano-alumina on the w_{opt}
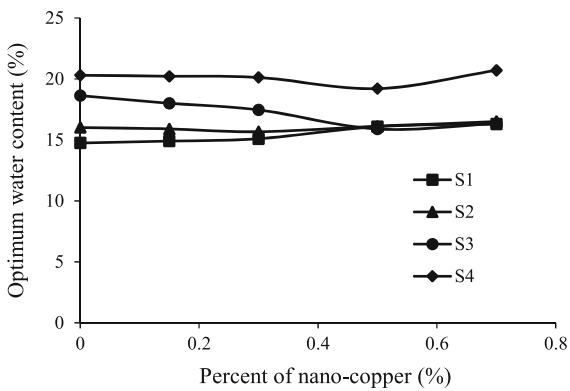

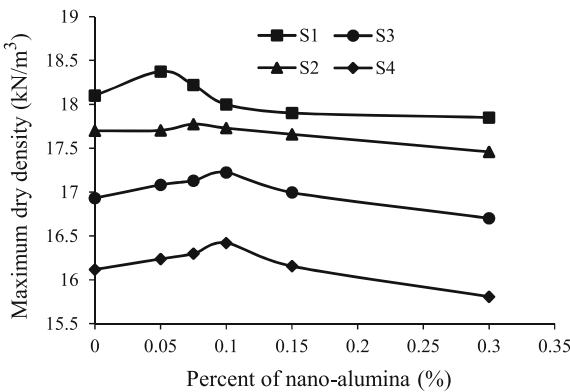**Fig. 11** Effect of nano-copper on the w_{opt}

Fig. 12 Effect of nano-alumina on the max. γ_d

The effect of nano-alumina and nano-copper on the shrinkage and expansive strains

Total strain in soil occurred into two stages. First, expansive strain took place when soil samples were

Fig. 13 Effect of nano-copper on the max. γ_d

exposed to water until saturation, and the second when soil samples were exposed to drying. The summation of the expansive and shrinkage strains give the total strain in soil samples. Nano-alumina was observed to reduced both the amount of expansive and shrinkage strains. This is because the increase in dry density and the reduction in w_{opt}. However, when the nano-alumina content increases the density reduces because the particle becomes agglomerated which normally increases the void ratio and decreases the density which in turn leads to increase in the soil strain (Figs. 18, 19, 20).

Similar behavior was also observed using nano-copper. However, the nano-copper content is more than nano-alumina and the improvement from using nano-copper is better than nano-alumina (Figs. 21, 22, 23). In addition, the reduction in strain by nano-copper of soil samples S3 and S4 are more than the soil samples S1 and S2, especially the shrinkage strain (Fig. 21).

The effect of nano-alumina and nano-copper on the CIF and hydraulic conductivity

The effect of nano-material on the desiccation crack development is shown in Fig. 24. The test results showed that the nano-materials causes some decrease in the desiccation cracks on the surface of S3 and S4 samples. For S1 and S2, however, the cracks cannot be measured. The reduction in CIF when nano-copper is used is more than the soil samples with nano-alumina. This is because the total strain of soil with nano-copper is less than the total strain of soil with nano-alumina. Furthermore, from Table 5, the hydraulic conductivity
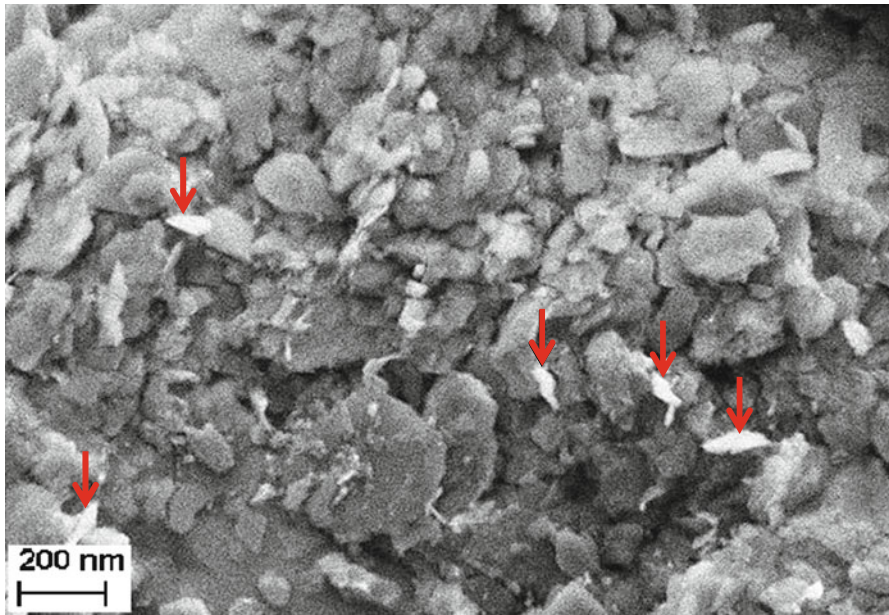
Fig. 14 Distribution of nano-material particle in soil sample (S3) mixed with 0.5 % nano-copper
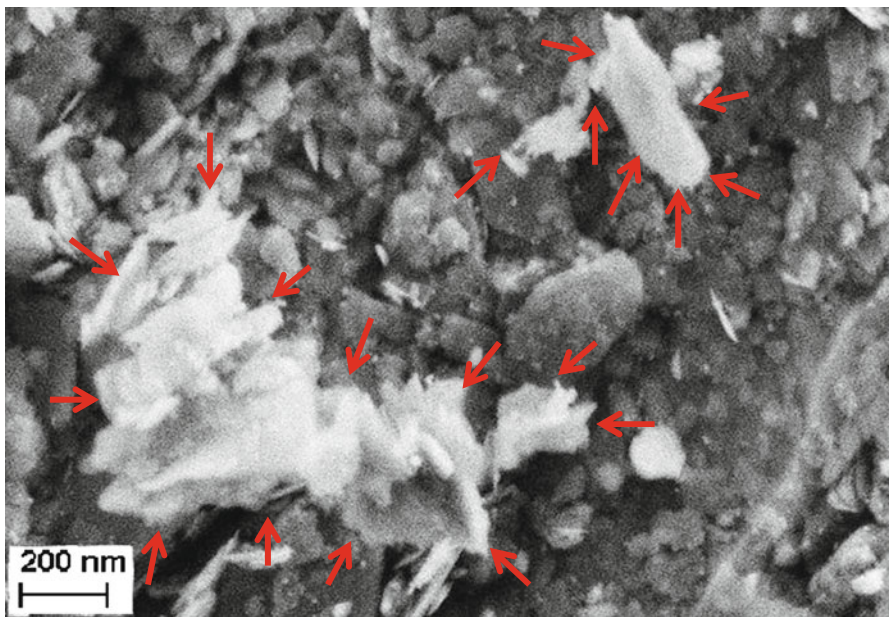

Fig. 15 Distribution of nano-material particle in soil sample (S3) mixed with 0.7 % nano-copper

with and without nano-material are very close to each other for soils with low bentonite content (low plasticity index), i.e., samples S1 and S2. This means that the nano-materials can be used as a treatment material for cracks without any significant effect on the hydraulic conductivity of soil. Furthermore, the addition of nano-materials in high bentonite soils content causes a reduction in hydraulic conductivity. This differs from other treatment materials which reduces the cracks in soil and increase the hydraulic conductivity, for example, polypropylene fiber and other type of fiber (Rifai 2000).
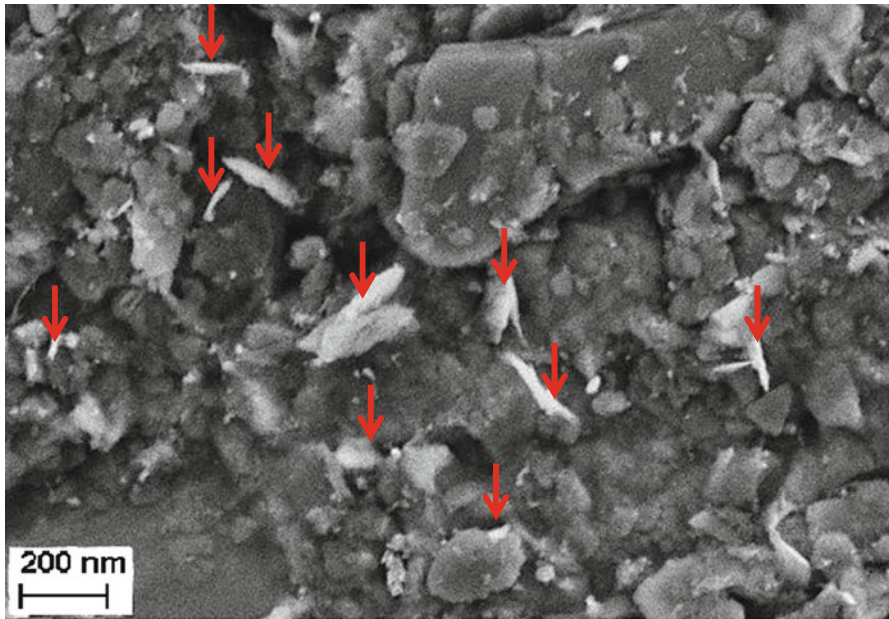
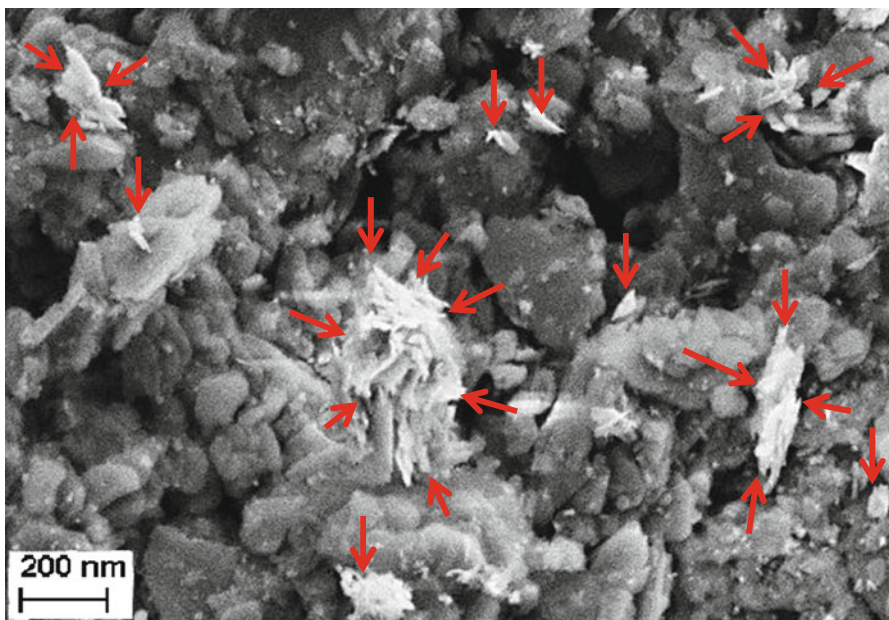**Fig. 16** Distribution of nano-material particle in soil sample (S4) mixed with 0.5 % nano-copper

Fig. 17 Distribution of nano-material particle in soil sample (S4) mixed with 0.7 % nano-copper

Conclusions

The mixtures of soil and nano-materials enhance the engineering properties of soils (i.e., compaction characteristics, volumetric shrinkage strain, volumetric expansive strain, and the CIF). Insoluble nano-material does not give chemical reaction with soil by just mixing with water. The addition of some type of nano-material such as nano-clay does not produce significant improvement in soil. However, nano-clay content exceeding certain limits has a negative effect. The improvement by nano-copper was more positive than nano-alumina in terms of
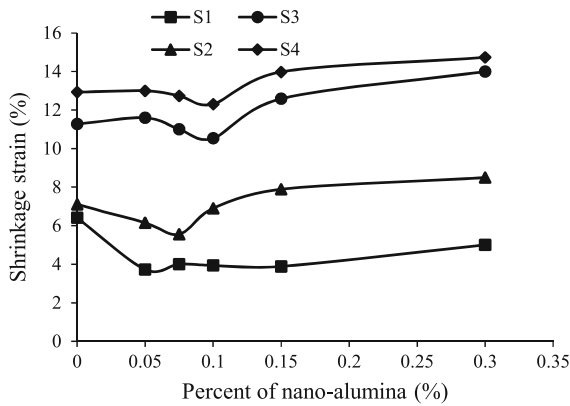
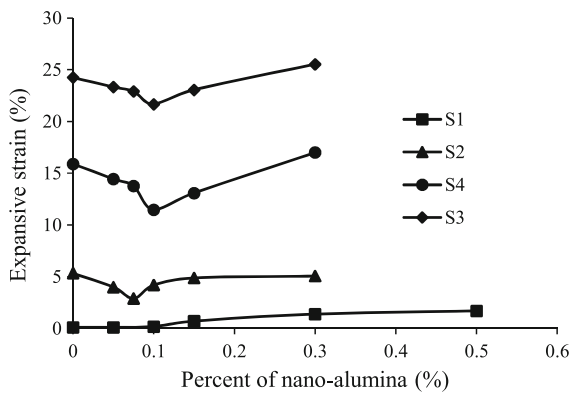Fig. 18 Effect of nano-alumina on the shrinkage strain

Fig. 21 Effect of nano-copper on the shrinkage strain

Fig. 19 Effect of nano-alumina on the expansive strain

Fig. 22 Effect of nano-copper on the expansive strain
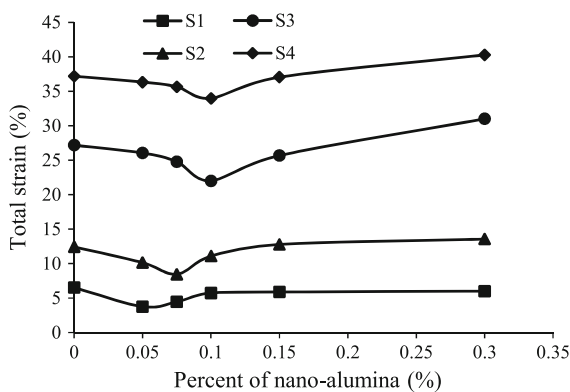

Fig. 20 Effect of nano-alumina on the total strain
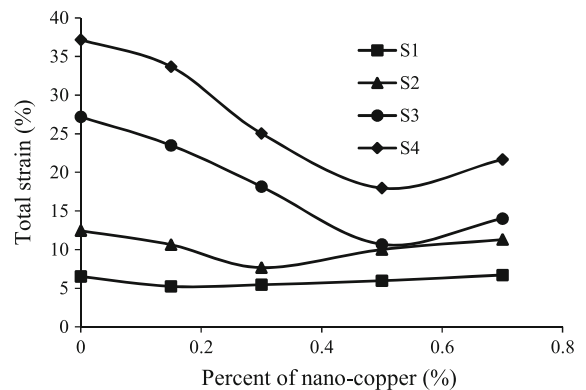
Fig. 23 Effect of nano-copper on the total strain

expansive and shrinkage strain. This is possibly because the particle density of nano-copper is greater than nano-alumina which increases the specific gravity of the soil–nano-material mixture leading to increase in the maximum dry density of the mixture. The increase in the dry density subsequently leads to decrease in the soil
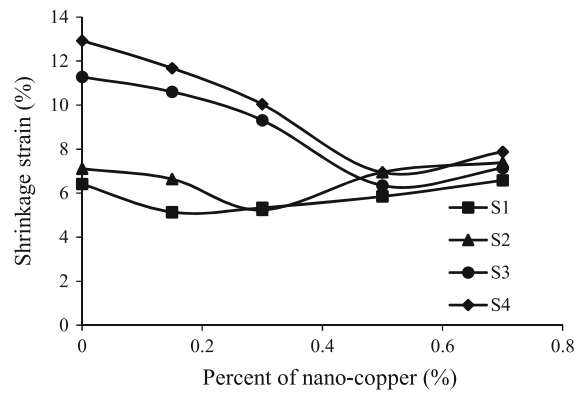
shrinkage and expansive strains. Moreover, the increase in the content of agglomerated particles leads to decrease in the dry density and increase in voids which increase the water content. Therefore, the shrinkage and swell strain also increases. Since the size of nano-copper particles is more than two times size of nano-alumina particles, the
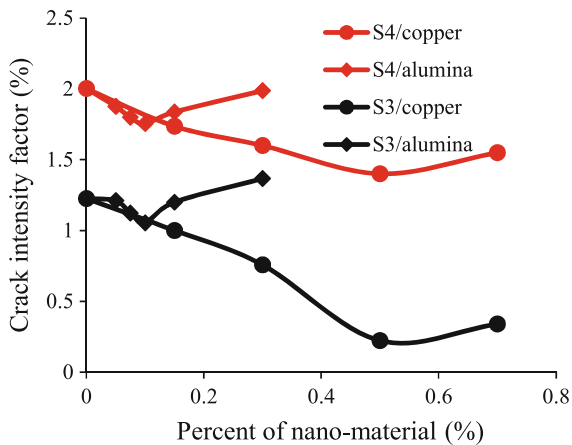
Fig. 24 Effect of nano-material on the CIF

Table 5 Hydraulic conductivity of soil samples with and without nano-materials

Soil type	Nano-material content (%)		Hydraulic conductivity (m/s)
	Al$_2$O$_3$	CuO	
S1	0	0	1.862E−09
S1	0.05	–	1.815E−09
	–	0.15	1.871E−09
S2	0	0	1.244E−11
S2	0.075	–	1.301E−11
	–	0.3	1.227E−11
S3	0	0	1.000E−11
S3	0.1	–	5.401E−12
	–	0.5	8.444E−11
S4	0	0	4.567E−12
S4	0.1	–	1.599E−12
	–	0.5	2.408E−12

nano-copper less agglomeration than nano-alumina thus improving the soil better than nano-alumina. The addition of nano-materials more than the optimum value causes agglomeration of particles that produce negative side effects on the mechanical properties of the soil. The additive of nano-materials does not reduce the hydraulic conductivity of soils. This different from other stabilizer materials which decrease the crack development and increase the hydraulic conductivity (i.e., fiber).

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