

## Effects of Nano-Carbon Reinforcement on the Swelling and Shrinkage Behaviour of Soil

(Kesan Pengukuhan Nanokarbon terhadap Sifat Pembengkakan dan Pengecutan Tanah)

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### ABSTRACT

*In this study, the performance of two types of nanocarbons (NCs), namely carbon nanotubes (CNTs) and carbon nanofibers (CNFs), on the three-dimensional shrinkage and swelling properties of three clayey soils were investigated. The specimens of soil mixed with clay with bentonite contents of 0, 10 and 20% by weight of dry soil. NC contents of 0.05, 0.075, 0.10 and 0.20% were chosen to investigate the influence of different NC types, CNTs and CNFs. All soil specimens were compacted under maximum dry unit weight and optimum water content conditions by using standard compaction tests. The physical and mechanical characteristics of the reinforced samples were then determined. These included the desiccation cracking area, used to determine the crack intensity factor (CIF), as well as the shrinkage and swelling. The CIF for the soil specimens without NCs were higher than the soil specimens with NC additives. These results show that NCs decrease the development of desiccation cracks on the surface of compacted samples. The shrinkage and swelling tests showed that the rate of volume changing of the compacted soil specimens reduced with the increasing of NCs.*

*Keywords: Compaction; desiccation cracks; nano-fiber reinforcement; volume change*

### ABSTRAK

*Dalam kajian ini, prestasi dua jenis nanokarbon (NC), iaitu tiub nano karbon (CNT) dan serat nano karbon (CNF) terhadap sifat pengecutan tiga dimensi dan sifat pembengkakan tiga jenis lempung dikaji. Spesimen tanah dicampur dengan lempung pada kandungan bentonit 0, 10 dan 20% daripada berat tanah kering. Kandungan NC sebanyak 0.05, 0.075, 0.10 dan 0.20% dipilih untuk mengkaji pengaruh jenis NC yang berbeza iaitu CNT dan CNF. Semua spesimen tanah dipadatkan di bawah unit berat kering maksimum dan keadaan kandungan air yang optimum dengan menggunakan ujian pepadatan piawai. Ciri fizikal dan mekanik sampel tersebut ditentukan. Ini termasuk kawasan retak pengeringan yang digunakan untuk menentukan faktor keamatan retakan (CIF) serta pengecutan dan pembengkakan. CIF untuk spesimen tanah tanpa NC adalah lebih tinggi daripada spesimen tanah dengan bahan tambah NC. Keputusan ini menunjukkan bahawa NC mengurangkan pembentukan kesan retak pengeringan pada permukaan sampel yang dipadatkan. Ujian pengecutan dan pembengkakan menunjukkan bahawa perubahan kadar isi padu pada spesimen tanah yang dipadatkan dikurangkan dengan peningkatan NC.*

*Kata kunci: Pepadatan; pengukuhan nano-fiber; perubahan isi padu; retak pengeringan*

### INTRODUCTION

The changes in moisture content result in large volume changes in the fine soil. Swelling-shrinkage behavior, as well as desiccation cracks, are more apparent in soil near the ground surface because that is the medium directly impacted by seasonal and environmental changes (Nahlawi & Kodikara 2006). To suppress this in the landfill linear area, the soil is normally compacted to reach maximum dry density ( $\gamma_{d\ max}$ ) and optimum water content ( $w_{opt}$ ) to reduce shrinkage strain after saturation (Albrecht & Benson 2001). Accordingly, the long term performance of cover barrier systems regarding cracking of the compacted clay liners due to dryness is significant since the cracks in the compacted soil liner can severely decrease the sealing effect of the cover system (Witt & Zeh 2005). Ige (2009) and Leroueil and Hight (2015) in studying the relationship between the hydraulic

conductivity and volumetric shrinkage of compacted soil, concluded that for the construction of liners, volumetric shrinkage strain must be smaller than 11% to prevent noticeable increase in the hydraulic conductivity in soil. However, the changes in moisture of the soil can influence the internal stress equilibrium between the soil water and clay particles, and this mechanism of shrinking and swelling of fine soil is quite complex and can be influenced by several factors for example plasticity, climate, moisture content and clay content (Chen 1975; Houston et al. 2009).

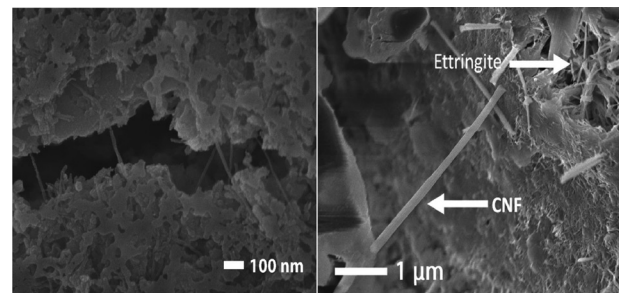
Since the late 1980s, studies using polymeric fibers were conducted (Saran 2010). Furthermore, the use of synthetic fibers, such as polypropylene, polyamide and polyester fibers, in improving the mechanical properties and failure characteristics of soil have been confirmed (Diambra et al. 2010; Hataf & Rahimi 2006; Kumar

et al. 2006; Michalowski & Cermák 2002; Park & Tan 2005; Yetimoglu et al. 2005). Mixing soil with high tensile strength fibers results in fiber-reinforced soil with improved engineering properties.

In recent years, there has been an increase in interest in the use of fibers to suppress the problem of desiccation cracks. In this regard polypropylene fiber has become one of the most commonly used synthetic materials to reinforce concrete and soil (Hejazi et al. 2012; Plé&Lê 2012). Its primary attractiveness is its low cost (Etter et al. 2011). Tang et al. (2007) reported that using fibers as additives can heighten the strength and ductility and reduce the stiffness of cement-stabilized clays. Moreover, Mirzababaei et al. (2013), in studying the effects of carpet waste fibers on the expansiveness of compacted clays, disclosed that as the percentage of fiber increased, the swelling pressure would decrease quickly. The interface between the fiber surface and the hydrated products (in fiber-reinforced cemented soil) has a noticeable impact on the strength of the interface. Nano-reinforcement

The development of nano-materials in soil reinforcement could result in considerable benefits as these materials have shown to give significant advantages, especially in concrete technology (Al-Mansob et al. 2017; Govindasamy et al. 2017). Because of their unique mechanical properties carbon nanofibers (CNFs) and carbon nanotubes (CNTs) are gaining popularity as promising nanomaterials. The nanoscale and high aspect ratio of CNTs and CNFs imply that they can be dispensed on a much finer scale than the usually utilised micro-reinforced fibers (Alsharef et al. 2016; Nochaiya & Chaipanich 2011; Taha & Alsharef 2017). Hence, microcracks can be intercepted faster during their proliferation in a nano-reinforced matrix. Thus, much smaller cracks are produced at the point of first contact between the moving crack front and the reinforcement. Widely researched in the last few decades, researchers originally investigated the usage of macro-to microfibers to control the formation of cracks in cementations materials such as concrete, cement paste and cement grout (Li et al. 2007; Mangat et al. 1984; Wang et al. 2008). Al-Rub et al. (2011) used scanning electron microscopy to analyze the microstructure of cementitious materials, both unreinforced and NCs reinforced and found that the nanofibers enhanced the mechanical properties of the cementitious material. Figure 1 shows the bridging of a nano-sized crack by CNTs and CNFs. The CNTs have a diameter of approximately 10 nm, but they can bridge large cracks because of their high aspect ratio (Figure 1(a)). Figure 1(b) shows both treated CNFs and small needle-like ettringite formations, which could explain the degradation in mechanical properties observed with the acid-treated NCs.

Judging from the results seen in concrete, NCs may have potential to be excellent alternative to micro fibers as nano-reinforcement in high-performance soil. The effectiveness of NCs depends on two primary features; the distribution of NCs within the material and the bonding strength i.e. energy between NCs surface and the material



a) Untreated CNT embedded in cement paste bridging a nano-sized crack      b) CNF and ettringite formations

FIGURE 1. SEM image of different phases of cement shows the fracture surface of the sample containing untreated NCs, a) CNT and b) CNF (Al-Rub et al. 2011)

(Alsharef et al. 2017a; Nochaiya & Chaipanich 2011). NCs are strongly drawn to each other due to their high van der Waals forces, Moreover, CNTs are more influenced by van der Waals forces than CNFs since their surface-to-volume ratio is higher. CNTs are more prone to bundling and agglomeration because of this higher attraction (Yazdanbakhsh et al. 2010).

#### DISPERSION OF NANOCARBONS

The problem of dispersing carbon nanoparticles in soil is one of the current critical hurdles that has hindered the widespread use of such materials in soil composites. The advantageous properties of CNFs and CNTs cannot be harnessed without controlling dispersion. A common method to improve CNFs and CNTs dispersion is by the use of surfactants. Dispersion of NCs in liquid media is a well-researched topic and to accomplish this, many surfactants, combined with sonication, have been investigated (Alsharef et al. 2017b; Moore et al. 2003). However, most effective surfactants are not compatible with soil material (Taha et al. 2005) or cement hydration and their presence in cement paste usually results in a weak material in which significant quantity of air is entrapped. If not controlled properly, it can damage, shorten, or even dissolve the NCs material (Al-Rub et al. 2011; Yazdanbakhsh et al. 2010). Where surfactants prove to be inadequate in achieving dispersion in soil and concrete, ultrasonication has shown some promise. Ultrasonication can roughen the fiber surface thereby improving their bond with composites (Yazdanbakhsh et al. 2009). This bond strength is controllable and can even surpass a threshold value, which, if achieved, prevents fiber slippage. It is reported that the use of ultrasonication to effect considerable dispersion of nano-particles in aqueous solutions is frequently achieved. In particular, it has been shown that CNTs and CNFs can be effectively ultrasonically dispersed in a water solution (Cui 2013). Moreover, Firoozi et al. (2015) used ultrasonics for the dispersion of soil particles without pre-treating or adding a dispersing surfactant. The aim of this research was to investigate the performance of NCs additives on the

compacted soil specimens. More specifically, the effect on NCs on the desiccation cracks (CIF), volumetric shrinkage behavior and liner shrinkage on compacted soil specimens is investigated. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are utilized to examine the cracked surface of soil samples in order to understand how the NCs affects the mechanical properties of the soil specimens.

## MATERIALS AND METHODS

### MATERIALS

Residual soil from the vicinity of the National University of Malaysia, Bangi, was chosen for the study. The soil was sampled at depth of 1 m  $\pm$  20 cm below the ground surface. The soil was mixed with different ratios of bentonite namely S1= 0% bentonite, S2 = 10% and S3 = 20% bentonite to create three kinds of soil samples with different PI. The physical and chemical properties of each soil sample are listed in Table 1. The bentonite used in this study, for which properties are listed in Table 2, is a high sodium bentonite containing sodium montmorillonite.

The multi-walled CNTs, supplied by Arkema Inc. are produced under the trade name Graphistrength. Typical dimensions are 10-15 nm in diameter (5-15 walls) and 1-10 microns in length. The other nano-fiber material, PR-24-XT-LHT, a CNF from Pyrograf, was also used. The properties and other relevant information of the CNTs and CNFs, as provided by the companies, are shown in Tables 3 and 4, respectively. TEM images showing the shape of the NCs can be seen in Figure 2.

TABLE 1. Chemical and physical features of soil samples

Characteristics	Value and descriptions		
	S1	S2	S3
Specific gravity	2.60	2.61	2.61
Plasticity index (%)	13.6	31.4	64.5
Liner shrinkage (%)	8.9	14.0	19.8
Passing no 200 sieve (wt. %)	45.4	55.1	64.9
Unified soil classification system (USCS)	CL	CH	CH
Optimum water content (%)	14.3	15.7	18
Chemical composition			
SiO <sub>2</sub> (%)	62.1	55.2	53.1
Al <sub>2</sub> O <sub>3</sub> (%)	29.5	21.3	23.5
Fe <sub>2</sub> O <sub>3</sub> (%)	5.7	5.2	5.2
MgO (%)	0.58	1.5	1.1
CaO (%)	0.03	1.23	1.0
TiO <sub>2</sub> (%)	1.17	0.9	0.9
Na <sub>2</sub> O (%)	--	1.2	0.8
K <sub>2</sub> O (%)	0.8	0.7	0.7
Other	0.1	0.4	0.3
Heat loss	0.2	11.1	12.3
Organic matter (%)	4.2	--	--

TABLE 2. Bentonite properties

Property	Value
Plastic limit	70.0
Liquid limit	464.6
Specific gravity	2.65
Cation exchange capacity	90.0 meq/100 g of dry soil
Plasticity index	394.3
Chemical composition	
Formula	Concentration (%)
SiO <sub>2</sub>	60.9
Al <sub>2</sub> O <sub>3</sub>	14.8
Fe <sub>2</sub> O <sub>3</sub>	4.4
CaO	3.7
Na <sub>2</sub> O	3.1
MgO	3.1
K <sub>2</sub> O	0.8
TiO <sub>2</sub>	0.6
Other	0.4
Heat loss	8.2

TABLE 3. Properties of CNT – (Graphistrength® C100)

Property	Value
Average Diameter (nm)	10-15
Average length ( $\mu$ m)	1-10
Carbon purity (%)	>95
Apparent density (kg/m <sup>3</sup> )	50 – 150
Relative density (g/mL) at 25°C	2,1
Aspect ratio	600-700
Applications	Reinforcements

TABLE 4. Properties of CNF -PR-19-XT-LHT

Property	Value
Average diameter, (nm)	200
Average length ( $\mu$ m)	50-200
Carbon purity (%)	>98
Apparent density (kg/m <sup>3</sup> )	30-300
CVD carbon overcoat present on fiber	No
Nanofiber wall Density, g/cc	2-2.1
Iron, ppm	12,466
Aspect ratio	1300-1500
Applications	Mechanical and electrical

### PREPARATION OF NANOCARBON-REINFORCED SOIL MIXTURES

Sieve analyses and particle size distribution analyses based on the unified soil classification system, indicated that the soil is sandy with low plasticity clay index (CL). When the bentonite was added to the soil with dry weight percentages of 10% and 20%, the soil is classified as high plasticity index clay (CH). Sieve analyses for the three kinds of soil are displayed in Figure 4.

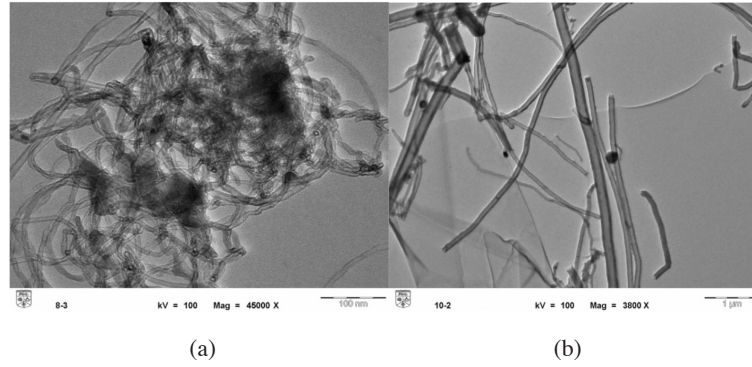


FIGURE 2. TEM micrograph showing shape of NCs: (a) CNT and (b) CNF



FIGURE 3. S3 Soil samples after compaction, the darker sample contains 0.2% CNF while the lighter sample has no NC

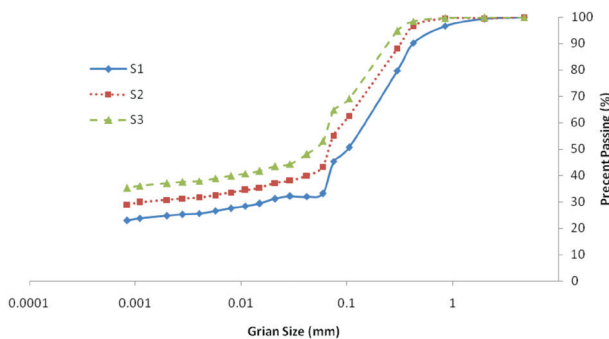


FIGURE 4. Grain size distribution for soil (S1) and soil with 10% (S2) and 20% (S3) bentonite

In further preparing the soil mixtures, the dispersion solution was then prepared by mixing 50 g of natural soil with 100 mL distilled water in a beaker. A soil-to-water mass ratio of 1:2 (by weight) was selected in order to assess the influence of water-filled volumes between soil particles on the movement of NCs. The quantity of NCs incorporated into the soil was 0%, 0.05%, 0.075%, 0.1% and 0.2% of the total dry weight of the soil. These NCs were first added to the water-soil solution and then manually mixed for 5 min. An ultrasonic tip was placed in the beaker and the mixture was sonicated for 4 min to disagglomerate the NCs while avoiding possible tube fragmentation (Vaisman et al. 2006). This time is also considered sufficient to accomplish dispersal of soil in the aqueous solution

(Firoozi et al. 2015), while preventing the suspensions from overheating (Lee et al. 2007). For the sonication process, the Sonic Ruptor 250 Ultrasonic Homogenizer was used. A piezoelectric ultrasonic transducer transforms a sinusoidal electrical voltage into a mechanical longitudinal resonance vibration with the resonance frequency of 20 kHz. The ultrasonic tip used has a cylindrical-shaped medium-intensity ultrasonic processor with a diameter of 19 mm and a length of 10.5 cm. The device was operated at 50% of its maximum amplitude, delivering energy to the samples at a rate of 1900-2100 J/min. Energy was applied in cycles of 2 s to prevent the suspensions from overheating. Figure 5 shows the soil-water-NC suspension after sonication.

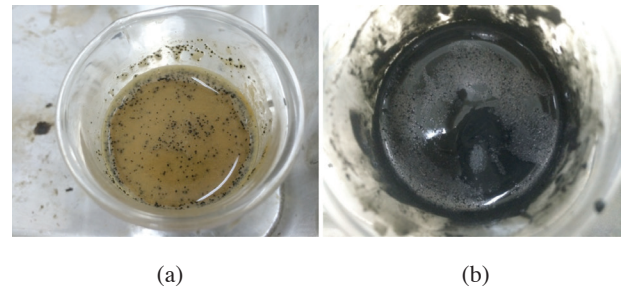


FIGURE 5. Effect of sonication on soil-water mixture: (a) mixture after manually mixing, (b) suspension after 4 min of sonication

#### SONICATION PROCESS

The sonication process disperses both soil and NCs concurrently, resulting in a suspension. As the wide distribution of soil particles and NCs are dispersed, they overlap with each other while submicron clay particles act as wedges between the NCs and the larger soil particles. After sonication, the suspension was mixed thoroughly with the dry soil to achieve uniformity. This suspension incorporated with dry soil was then allowed to stand for 24 h, as prescribed by ASTM D698-7, to allow water in the suspension to be thoroughly absorbed into the dry soil particles. From the homogeneous color observed after compaction, as shown in Figure 3, it can be concluded that this procedure resulted in materially homogeneous samples.

#### ATTERBERG LIMITS

The Atterberg limits (i.e. the liquid limit, the plastic limit and the plasticity index) of each of the natural and treated soil samples were determined in accordance with BS 1377, part 2, 1990. The liquid limit tests were performed using a penetration cone assembly, which consisted of a stainless steel cone with a cone angle of  $30 \pm 1^\circ\text{C}$ . The plastic limit tests were performed using a manual method. Each sample was rolled at a sufficient pressure on a glass plate to form a thread with a uniform diameter of 3.2 mm along the full length of the sample. The plasticity index was calculated as the difference between the water contents at the liquid and plastic limits.

#### MAXIMUM DRY DENSITY TEST

The dry mixture was mixed with optimum moisture content (obtained through standard Proctor test) required to achieve maximum dry density for each NCs concentration. The standard Proctor compaction equipment was utilized to compact the soil samples at various water contents according to ASTM D698-70. The soil utilized for compaction was dehydrated in an oven and pounded with a rubber hammer and soil passing US No. 4 sieve was used for sample preparation. A spray bottle was used to dampen the soil with distilled water and stirred with a trowel while mixing to make sure there was an even distribution of water. Next, the soil was packed in plastic bags and allowed to saturate for minimum 24 h before compaction. Compacted specimens were utilized for expansive strain, shrinkage strain and desiccation crack tests. Volume change measurement was carried out to measure shrinking and expanding features. After compaction, the soil specimens were divided into two portions. The first portion was left to dry in an oven temperature  $\sim 34 \pm 2^\circ\text{C}$  to measure shrinkage strain and the second portion was saturated with water to examine the expansion strain.

#### VOLUMETRIC SHRINKAGE TEST

Shrinkage occurred when wet soil dried up as three-dimensional volumetric shrinking happened. Thus, the samples were dried from all sides according to ASTM C 157 after curing. The change in volume was used to establish the volumetric shrinkage strain of the soil samples. The volumetric shrinkage strain is noted as the change in volume ( $\Delta V$ ) to the total volume of the soil sample ( $V_i$ ), which is written as:-

$$\text{Volumetric shrinkage strain} = \frac{\Delta V}{V_i} \times 100\% \quad (1)$$

#### THREE-DIMENSIONAL FREE SWELL TEST

This test recommended by National Lime Association (2004), in which larger specimens can be tested was chosen to determine the increase in volume due to capillary soaking. The change in volume was utilized to define the

volumetric expansive strain of the soil samples. Similarly, the volumetric expansive strain is noted as the change in volume ( $\Delta V$ ) to the initial volume of the soil sample ( $V_i$ ), which is written as:-

$$\text{Volumetric expansive strain} = \frac{\Delta V}{V_i} \times 100\% \quad (2)$$

#### DESICCATION CRACK TEST

In order to carry out desiccation crack test, soil samples were prepared at optimum water content using 152.4 mm (diameter) cylindrical mould with 116.43 mm in height. The drying process was carried out at a temperature of about  $34 \pm 2^\circ\text{C}$  for 10 days using an oven with fan. The surface dimensions of cracks were measured using gauge wires to find the crack intensity factor (CIF) to access the magnitude of desiccation cracks that form in the soils, which is written as (Harianto et al. 2008; Kleppe&Olson 1985; Peng et al. 2006):

$$\text{CIF} = \frac{A_c}{A_t} \quad (3)$$

where  $A_c$  is the desiccation crack area and  $A_t$  is the total surface area of soil sample.

#### RESULTS AND DISCUSSION

The influence of bentonite on the specific gravity is shown in Table 1. The slight increase with the addition of bentonite is due to the fact that the specific gravity of bentonite is greater than that of the original soil. However, the increase in the plasticity index and liner shrinkage with the addition of bentonite is quite significant as shown in Figure 6. It is especially the increase in the plasticity index that resulted in the soil's classification being shifted to that of a high-plasticity clay.

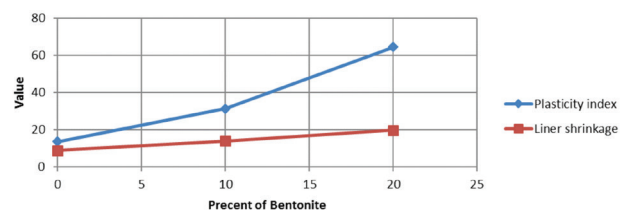


FIGURE 6. Effect of bentonite on liner shrinkage and plasticity index

#### EFFECT OF CNTS AND CNFS ON PLASTICITY INDEX

While the effect of the addition of CNTs to the plasticity index of the three soil specimens is insignificant, the effect of CNFs is significant in that more CNFs contribute to a decrease in the plasticity index (Figures 7 & 8). This behavior is likely due to the fact that CNFs have a higher

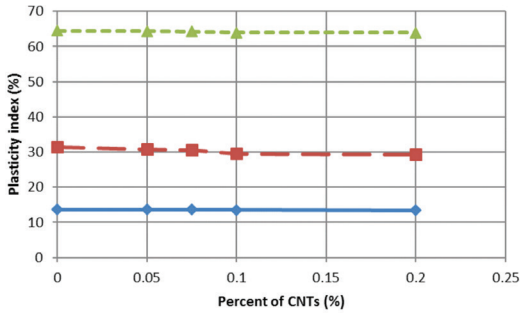


FIGURE 7. Effect of CNTs on plasticity index

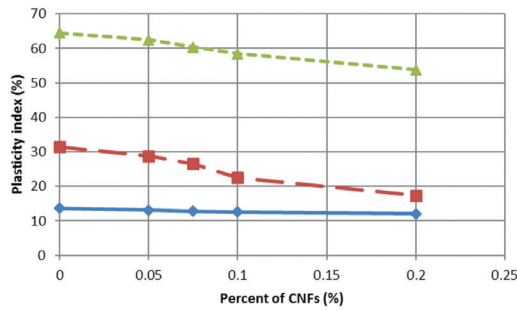


FIGURE 8. Effect of CNFs on plasticity index

aspect ratio than CNTs (Tyson et al. 2011). Generally, also, the NCs filaments have higher density than clay particles and their surface area is less than that of the bentonite particles. The CNFs however, are a super-hydrophobic and chemically inert material (Wang et al. 2008) and therefore does not soak up or respond to natural composite (soil moisture) or leachate as clay particles would.

EFFECT OF NCS ON COMPACTION CONDITIONS

There is a negligible effect of CNTs on the optimum water content ( $w_{opt}$ ) for S2 and S3 soil specimens (Figure 9). However, some noticeable changes occurred for S1 soil (0% bentonite) with a decrease in  $w_{opt}$  at 0.75% CNT and an increase in  $w_{opt}$  for higher CNTs. The change is mainly due to the displacement and rearrangement of soil particles induced by addition of CNTs and then filling soil voids, thereby reducing volume spaces in which water to be adsorbed. The work of Nochaiya and Chaipanich (2011) agrees with this in that they showed that the addition of CNTs was able to reduce the porosity. For specimens with CNFs, more significant changes occurred on  $w_{opt}$  as the amount of CNFs increased (Figure 10). For the S3 soil (20% bentonite), there is a decrease in  $w_{opt}$  up to 0.05% and constant  $w_{opt}$  for higher levels of CNF.

Figures 11 and 12 show that adding NCs influence the maximum dry density ( $\gamma_{dmax}$ ) of the nano-reinforcement soil, in that  $\gamma_{dmax}$  increased with increasing NC, reaching a peak at 0.075%, 0.1% and 0.075% in S1, S2, and S3 soil specimens, respectively, for CNT-reinforced soil specimens and peaking at 0.075%, 0.1% and 0.05% for the

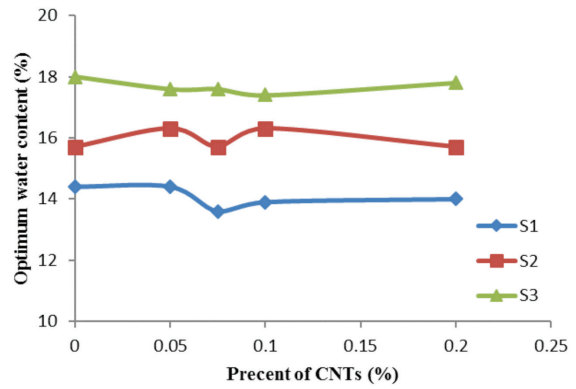


FIGURE 9. Effect of CNTs on  $w_{opt}$

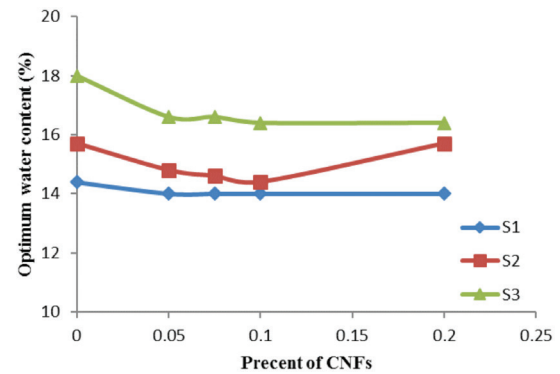


FIGURE 10. Effect of CNFs on  $w_{opt}$

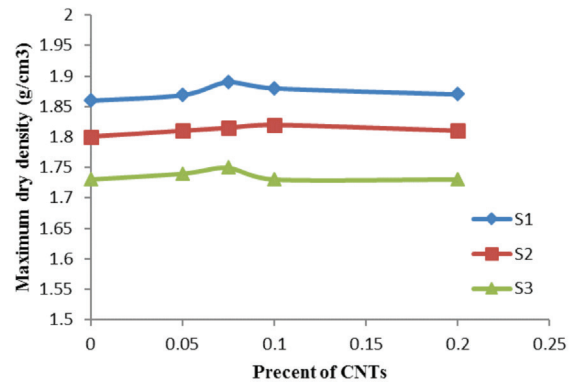


FIGURE 11. Effect of CNTs on  $\gamma_{dmax}$

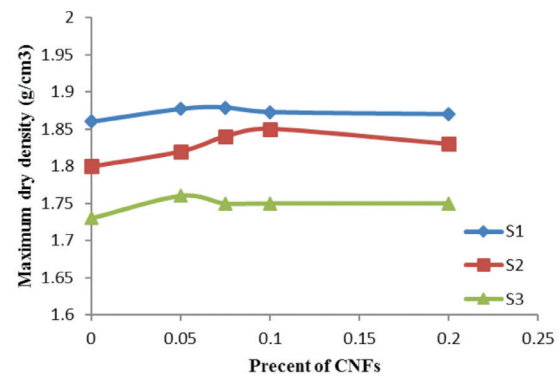


FIGURE 12. Effect of CNFs on  $\gamma_{dmax}$

respective CNF-reinforced soil specimens. It is apparent, however, that the effect of CNFs on  $\gamma_{dmax}$  is more significant compared to effect of CNTs. This is because the diameter and length of CNFs are greater than of CNTs (Blandine et al. 2016). Moreover, there is no increase in  $\gamma_{dmax}$  for NC contents above 0.10%. However, it is observed that after the peak, increasing NC content results in a decrease in  $\gamma_{dmax}$ . It is expected that well-dispersed CNTs and CNFs would lead to filling of pores within soil with the fibers, thereby improving its microstructure. Example of such cases are shown in Figure 13. It is also possible, however, for fibers not to effectively fill the pore spaces or come in full contact with soil particles. In such cases,  $\gamma_{dmax}$  would decrease (Harianto et al. 2008). It could be then that the inclusion of NCs beyond the optimum limit may possibly result from agglomeration of NC filaments. According to Ferkel and Hellmig (1999), increasing nano-material content beyond the optimum limit causes an increase in the void ratio thereby decreasing density and increasing water content. An example of the soil-mixture microstructure for such a case can be seen Figure 14. Such NC filament agglomeration was observed when the NC was incorporated at 0.2% and when the NC was mixed into the soil without sonication.

#### EFFECT OF NCS ON SHRINKAGE AND EXPANSIVE STRAINS

Figures 15 to 20 show the effect that increasing NC content causes on the different types of volumetric strains of the nano-reinforced soil specimens. In all cases, nano-

reinforcement of the soil specimens results in a decrease in strain. This behavior can be explained as follows. Firstly, as the total contact area between the soil particles and NC filaments increases with increasing NC concentration, it provides more resistance, induced by soil-fiber interaction during the desiccation Cai et al. (2006). Secondly, less strain is attributed to the higher dry density and lower  $w_{opt}$ , when compared to soil specimens without nano-reinforcement. Figures 17 and 20 show the summation of the expansive and shrinkage strains, or the total strain of the soil samples. For all the soil specimens, it is noticeable that while strain decreases as NC content increases, the reduction in strain becomes insignificant as NC content surpasses 0.10%. This is likely because the NCs filaments become more agglomerated at higher NC concentrations, which increases the void ratio and decreases the density. It is also observed that NCs are effective in reducing shrinkage and expansive strain of expansive soil specimens (such as S2 and S3) which experience large volume changes due to moisture content variations. Reduction in the volumetric shrinkage strain may be due to the increase in the strength of reinforcement in the soil, particularly the tensile strength as proposed by (Fatahi et al. 2012). Other researchers have also found that the porosity and the total pore volume in reinforcement concrete decrease with the inclusion of CNTs (Siddique & Mehta 2014). When reinforced with NCs, the lowering of pore volume in the soil leads to a denser microstructure, and thus lower shrinkage values compared to that of unreinforced soil.

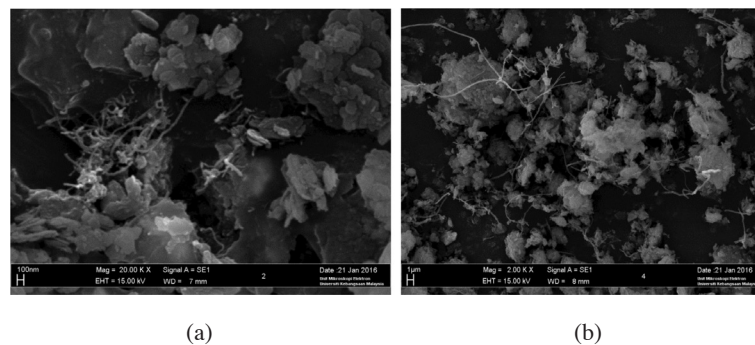


FIGURE 13. Distribution of NCs in soil after compaction: a) CNTs and b) CNFs

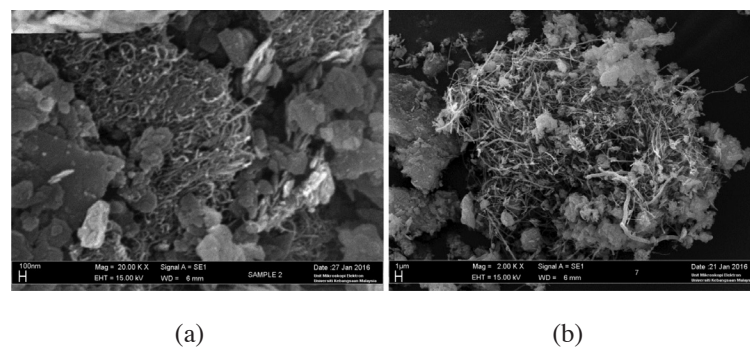


FIGURE 14. Agglomeration of NCs in soil after compaction without sonication: a) CNTs and b) CNFs

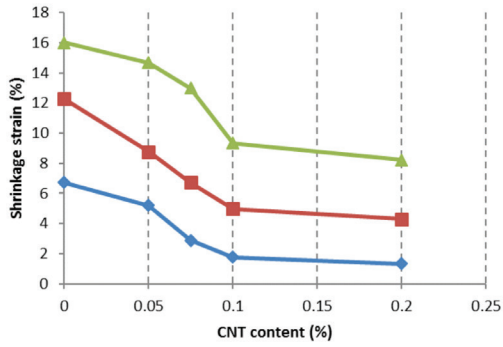


FIGURE 15. Effect of CNTs on shrinkage strain

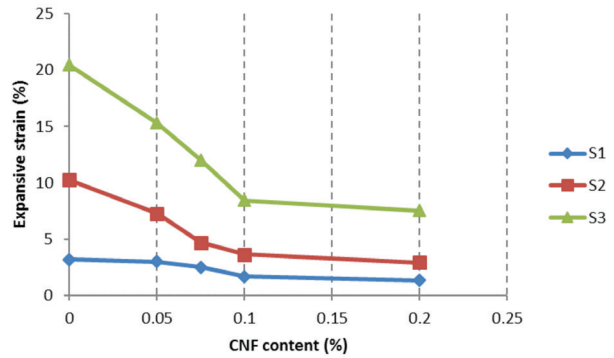


FIGURE 19. Effect of CNFs on expansive strain

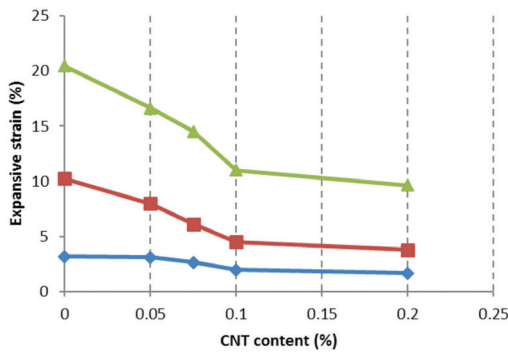


FIGURE 16. Effect of CNTs on expansive strain

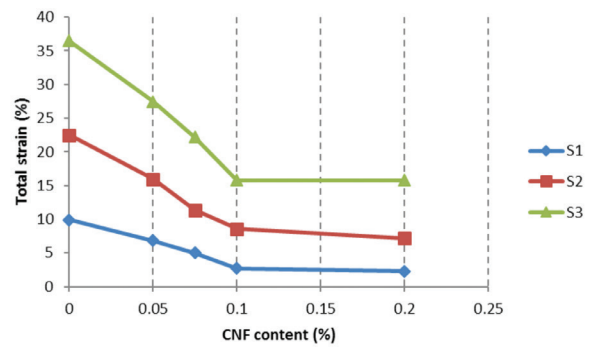


FIGURE 20. Effect of CNFs on total strain

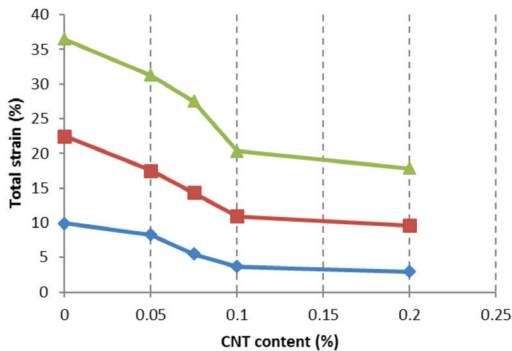


FIGURE 17. Effect of CNTs on total strain

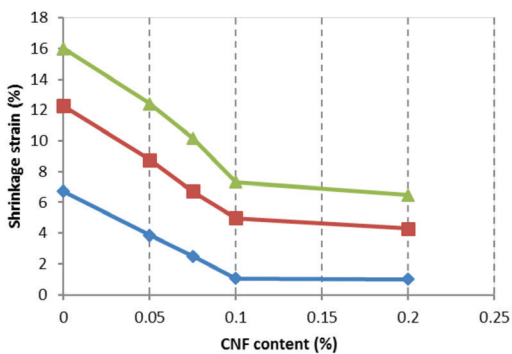


FIGURE 18. Effect of CNFs on shrinkage strain

Similar studies have found that the highest reduction of the volumetric shrinkage and expansive strain was observed for CNFs which were long, generally straight and had diameters of approximately 200 nm (Blandine et al. 2016). It is noticed that for bentonite modified soil specimens (S2 & S3), the volumetric shrinkage strain was reduced by more than 11%, making them suitable to be used as liners (Omidi et al. 1996a, 1996b). Figure 21 shows the schematic diagram (interpreted from Figures 13 and 22) of the mechanical behavior during the interface between NCs and soil particles. The NCs filament surface is interlocked and contacted with many soil particles causing a cumulative effect on the strength and friction between soil particles and NC filaments. Such mechanical behavior can enhance the impact of NCs to soil composite resistance namely interlock force, interfacial force and friction between the soil particles and NC filaments. However, it is worth noting that Cai et al. (2006) in their tests on the inclusion of fiber in clay stated that a rise in the fiber content can lead to a rise in shrinking potential of the chemically treated clay because of fiber slacking in the soil matrix during shrinkage.

EFFECT OF NCS ON DESICCATION CRACKS

The desiccation crack CIF for the sample without bentonite (S1) cannot be measured (low plasticity index), but the CIF for the other soil specimens (S2 and S3) are presented



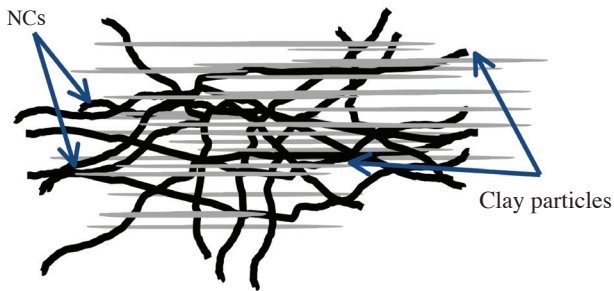


FIGURE 21. Schematic showing the progression of distribution of NCs in compacted soil after sonication

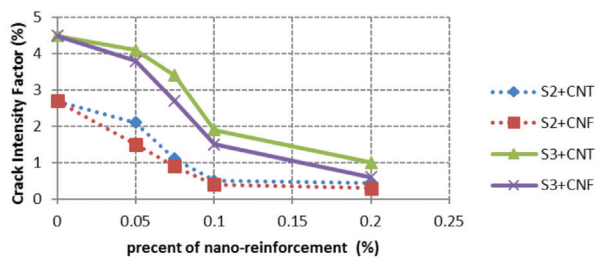


FIGURE 22. Effect of crack intensity factor (CIF) on the soil specimens at various NCs contents

in Figure 22. The maximum value of CIF was about 4.5% in S3 soil and this soil experienced a maximum reduction in CIF of 3.9% with CNF additives. Clearly, while the CIF of soil without NCs additives are significant, the addition of NCs results in a dramatic reduction in the CIF. The reduction in CIF when CNFs is used is slightly more than when CNTs is used. This is because the strain in soil with CNFs is less than the strain in soil with CNTs. Furthermore, as previously mentioned, with the addition of NCs, the friction between soil particles and NCs contributes to the development of resistance to the desiccation process. Whenever there is a tendency for the soil to shrink, it causes the soil-NCs resistance in soil to be activated, effectively suppressing cracks. When there is effective dispersion of CNs in compacted soil, it leads to the filling of pores, thereby improving its microstructure and restricting the propagation of nano-cracks and the formation of micro- and macro-cracks. An example of micro-crack bridging by the NCs within the soil, which leads to a reduction in CIF, is shown in Figure 23. Based on the magnification of the image, the CNFs, having diameters approximately 150 nm, can be clearly seen.

#### CONCLUSION

The effect of NCs filaments reinforcement was positive in terms of expansive and shrinkage strain. This is conceivably due to diminutive dimensions of the nano-material and their high aspect ratios. These properties increase the specific gravity of the soil-NCs mixtures, thus, leading to an increase in the maximum dry density of the mixture. The increase in the dry density subsequently

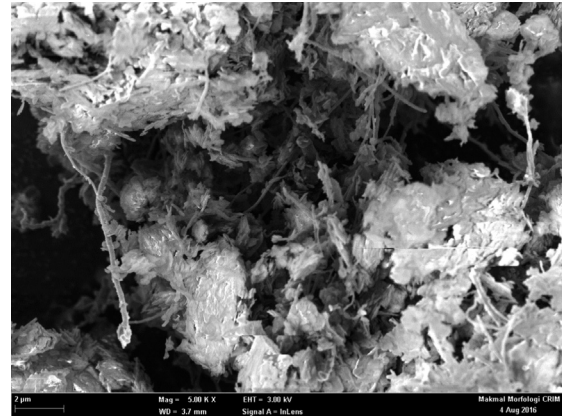


FIGURE 23. Micro-crack bridging by CNFs

results in decrease of soil shrinkage and expansive strains. Additionally, the cracks were significantly suppressed as demonstrated by a decrease in the CIF with increasing NCs content. This differs from other treatment materials like polypropylene and other types of fibre, which reduce cracks in the soil but increase the hydraulic conductivity, suggesting that NCs additives have potential application in soil as a possible procedure to overcome desiccation cracks commonly faced by landfill covered barriers. SEM images verified that there was good dispersion of NCs within the soil particles, confirming ultrasonication as a decent method of mixing.

This work emphasizes the need for further studies on the change in microstructural properties with nano-reinforcement in order to better understand how NCs improve soil strength properties.

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