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Nano-Fibrillated Cellulose as a Green Alternative to Carbon Nanotubes in Nano Reinforced Cement Composites

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Abstract**: *In this paper, a study for the availability of utilizing nano cellulose, as an alternative material for carbon nanotube in nano reinforcing of cement based materials was conducted. One of the main disadvantages of carbon nanotube is its high production cost. Nano-fibrillated cellulose is a natural material extracted from cotton fibers can be used as a nano fiber material for the cement paste. The comparison is done through studying the effect of nano-cellulose on the mechanical properties; through compressive, flexural, and splitting tensile strength and on the microstructure analysis; through a scanning electron microscope, atomic force microscopy, and x-ray diffraction, and comparing with the results obtained through using carbon nanotube instead. NCL and CNT were added to cement paste with the same ratios; 0.02 wt%, 0.04 wt%, 0-06 wt%, and 0.08 wt%. Results show that the optimum percentage for both NCL and CNT was 0.04 wt%. NCL had shown to be a great alternative to CNT as Nano reinforcement to the cement composites not only to enhance the mechanical strengths but also in enhancing the microstructure of the cement matrix.***

Keywords**: *Nano-Fibrillated Cellulose, Carbon Nanotube, Compressive Strength, Flexural Strength, Splitting-Tensile Strength, Microstructure Analysis***

I. INTRODUCTION

Advanced researches are now going in improving the tensile strength and ductile of cement-based materials. Cementitious materials are characterized by low tensile strength, low strain capacity, and as a brittle material. Short fibers are widely used to improve cracking in concrete [1]. Many different types of fibers added to improve the tensile and flexural strength [2-3].

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There are many obligations which face the use of different kinds of fibers; the poor interface, low corrosion resistance, and high cost. For glass fibers, the bond between glass fibers in addition, the cement matrix is relatively weak, have to influence on the tensile and flexural strength, and low alkaline resistance [4-5]. While the production process for carbon and polymer fibers is too expensive which retard their use in the construction industry [6]. Natural fibers have a good impact on the environment, enhance the mechanical properties of the cement matrix, and much cheaper than other types of fibers [7-8].

The demand for using more sustainable and durable materials leads to the use of products in the nano-scale. Nanotechnology can produce materials with a higher specific area and more advanced mechanical and durability properties than its conventional form [9-10-11-14]. Many studies worked on improving the dispersion of the nano-materials in the cement matrix to avoid the agglomeration occurred to the nano-materials when added to water. Serag et al. [15] had found that indirect sonication is more reactive than using direct sonication in improving the dispersion of nano-materials. While Passant et al. [16] had stated that the specific surface area is the main factor to determine the optimum dispersion time and dosage of nano-silica. Sharobim et al. [17-18] had found that the optimum solid to liquid ratio for nano-silica is 1:10, with an indirect sonication time 5 minutes. Nehal et al. [19] had studied the effect of sonicated nano-clay on the mechanical properties of concrete. The results indicated that using sonicated nano-clay particles produce a dense and well-compacted cement matrix. The gain in compressive, split tensile, flexural, and bond strengths with sonicated nano-clay mixes ranged between 1.42 and 3.74 times of the gain of those of using nano-clay without sonication.

Carbon nanotubes (CNT) are sheets of carbon atoms arranged in a hexagonal shape and rolled to form a cylinder [1-20]. Carbon nanotubes have high tensile strength, very low specific gravity, and high surface area. Carbon nano-tubes enhance the mechanical properties and the microstructure of the cement matrix. This can be attributed to the reduction of the possibilities of segregation occurred due to the good dispersion of carbon nano-tubes in the cement matrix, and the formation of a denser microstructure because of a reduction of the pores and enhancing the strength of the transition zone [21]. Luo et al. [22] had studied the flexural strength of cement mortars using MWCNTs and found that the flexural strength and the stress-intensity factor of the cement composites were significantly improved.

Singh et al. [23] found that adding 0.1% CNTs by weight of cement enhanced the flexural strength and flexural modulus of cement mortar by 7% and 72%, respectively than cement mortars without CNTs. Konsta et al. [24] got 25% and 12.5% improvement in flexural and load carrying capacity, respectively by adding 0.048 wt% of MWCNTs. Gdoutos et al. [25] investigated the effect of MWCNTs on the mechanical properties cement mortars and found that an improvement in flexural strength, Young's modulus and fracture toughness by 87%, 95%, and 77%, respectively.

However, adding CNTs to the cement composites had two problems; the agglomeration or the clumping of the nanotubes together, the week cohesion between them and the cement matrix, and the high price of CNTs, which make their addition to the cement composites, expensive [21].

Natural fibers are an alternative solution which enhances the mechanical properties of the cement composites and had a minimal carbon footprint of infrastructural materials at low cost [7-8]. Cellulose nanofibers (NCL) are composed of fibers with width in nano-scale and length in macro-scale. Nano-cellulose made from renewable forest and agricultural resources, which has demonstrated exceptional performance in Nano composites [26]. Nano-cellulose characterized by their unique morphology, low density, large surface to volume ratio, high surface area, high Young's modulus, high tensile strength, and low coefficient of thermal expansion (CTE) [27-28]. Onuaguluchi et al. [29] reported that adding 0.1 wt% of the nano-cellulose hand increase the heat of hydration and enhanced the flexural strength and the absorption energy of the cement paste by 106% and 184%, respectively. Cao et al. [30] had explained the performance of nano-cellulose in the cement matrix, was due to increase in the cement hydration, which was a result of two reasons; the first one is the steric stabilization which is responsible for the dispersion of cement particles. In addition, the second one, that nano-cellulose provides a channel for water transporting through the hydration products ring to the unhydrated cement particles and thereby improving hydration. Xie et al. [31] had found that adding 2-16% by weight millimeter long cellulosic fibers leads to 20-50% improvement in flexural strength and fracture toughness. In this paper, the effect of NCL and CNT studied on the mechanical properties of cement mortars; through compressive, flexural, and splitting tensile strength. In addition, the microstructure of the cement paste will be investigated through a Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM), and X-Ray Diffraction (XRD).

II. EXPERIMENTAL PROGRAM

A. *Materials*

Ordinary Portland cement (CEM I/42.5R) used in this research in accordance with ASTM C150 [32]. The chemical compositions of Portland cement shown in Table 1. Natural sand used as fine aggregate with particles size below 0.5 mm, specific gravity of 2.58 g/cm3, and fineness modulus of 2.25. Carbon Nano-Tubes (CNT) used in this research are made of graphene sheets wrapped in a cylinder shape with a length ranges from 10-100 mm with internal diameter from 1.5 to 15 nm and external diameter of 50 nm. Figure 1 shows the Transmission Electron Micrographs (TEM) of the used carbon nano-tube, while NCL used extracted from cotton fibers. Figure 2 shows the TEM of NCL. The ratio of cement to sand in this cement mortars is 1:2 by weight. The used high range water reducer is an aqueous solution of modified polycarboxylates.

Table 1: Chemical Composition of Portland Cement

Element	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	So_3	Na_2O	K_2O	L.O.I
Cement	20.13	5.32	3.61	61.63	2.39	2.87	0.37	0.13	1.96

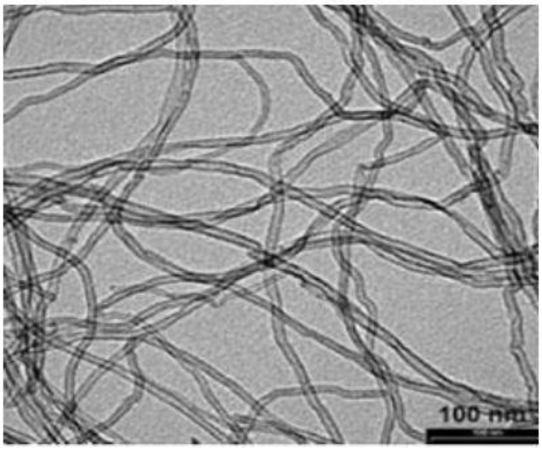


Figure 1: TEM of Carbon nano-tube (CNT)

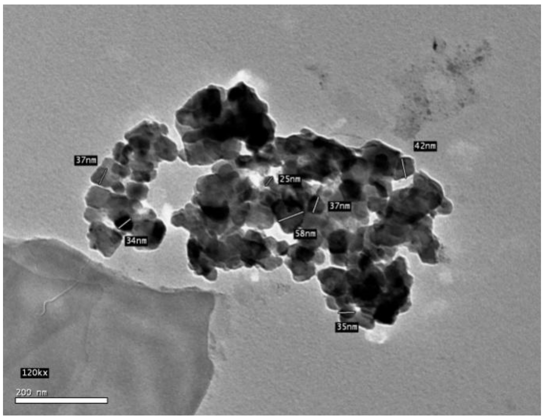


Figure 2: TEM of Nano-Cellulose (NCL)

B. *Mix Design Proportions*

In this study, nine cement mortars mix designed; control mix is made of cement, water, sand, and superplasticizer. For CNT mixes, designed in the same way but by adding CNT to the mix with 0.02%, 0.04%, 0.06%, and 0.08% by weight of cement. Then subjected to two different types of sonication; direct sonication through the homogenizer then followed by indirect sonication through bath sonicator [16]. While for NCL mixes was prepared by the same method and with the same replacing ratios. Then subjected only to sonication in an equal amount of water through bath sonicator to improve the dispersion of cellulose nano-particles in the cement matrix [33-34]. Figures 3 shows the solution of CNT and NCL after subjected to the sonication method. The water to binder ratio was a constant value of 0.43 in all mixes. The mix proportions of CNT and NCL cement mortars shown in Table 2.

Table 2: Mixes Components

Mix	Cement (gm)	Sand (gm)	Water (gm)	S.P. (ml)	CNT	NCL
C	1500	3000	650	2.8	-	-
0.02%CNT	1500	3000	650	2.8	0.02 %	-
0.04%CNT	1500	3000	650	2.8	0.04 %	-
0.06%CNT	1500	3000	650	2.8	0.06 %	-
0.08%CNT	1500	3000	650	2.8	0.08 %	-
0.02%NCL	1500	3000	650	2.8	-	0.02 %
0.04%NCL	1500	3000	650	2.8	-	0.04 %
0.06%NCL	1500	3000	650	2.8	-	0.06 %
0.08%NCL	1500	3000	650	2.8	-	0.08 %

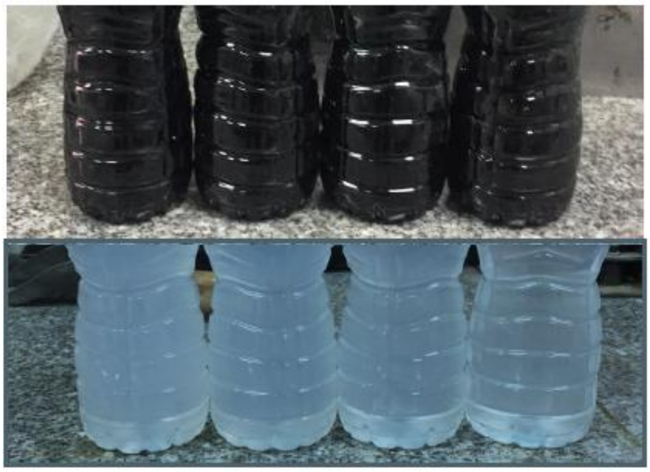


Figure 3: CNT and NCL solutions after sonication and directly prior to adding to the cement paste

C. *Mixing Procedure:*

Before introducing the cement mortar ingredients to the mixer, Nano-cellulose was first added to 600 ml of water and then subjected to ultrasonic waves through bath sonicator (indirect sonication) for 5 minutes and with a power of 135 W and a frequency of 40 kHz. While for carbon nanotube was also added to 600 ml of the mixing water and then subjected to direct sonication through a magnetic stirring of homogenizer for 2 minutes, then subjected to indirect sonication through bath sonicator for 5 minutes and the same speed, power, and frequency that nano-cellulose had subjected. Firstly, cement added to the tilt mixer and then add half of the mixing water for 2 minutes. Then add the remaining of the mixing water with the superplasticizer to the mix and mix for 2 minutes. Finally, fine aggregate added and this was for the control mix. While for nano-cellulose and carbon nanotube, cement added to the mixer then was added with CNT or NCL solution and mix for 2 minutes and then the remaining of mixing water with the superplasticizer was added and mix for 2 minutes. Then fine aggregate added and continue mixing for 2 minutes.

D. *Preparation of Specimens, Curing, and Testing*

After finishing the mixing process, cubes of 50x50x50 mm^3 were prepared for compressive strength test after 7 and 28 days of water curing according to ASTM C109 [35]. Compression tests performed by a loading rate of 2400N/s on a cement paste specimens. For flexural and cyclic loading test, 40x40x160 mm^3 prisms of cement mortars were prepared. The three-point bending tests performed through a microcomputer machine with a loading rate 50N/s. The three-point test was applied for flexural strength on specimens of 40x40x160 mm^3. While splitting-tensile test applied on samples with dimensions 50x50x50 mm^3. The flexural and splitting-tensile test applied on specimens after 28 days from water curing. Figure 4 shows the splitting tensile test setup for the cement mortar specimens.

Figure 4: Splitting Tensile Test Setup

III. RESULTS AND DISCUSSION

A. *Compressive strength*

Figures 5 and 6 show the compressive strength results of the control mix, CNT mixes, and NCL mixes. The cubes tested after 7 and 28 days of water curing. Generally, the addition of carbon nano-tubes (CNT) to cement mortars had a negative effect on the compressive strength on early age (7 days), except for the mix containing 0.04 wt% CNT. On the other hand, after 28 days the compressive strength enhanced significantly for all the CNT mixes as compared to the control mix. As for the nano-cellulose (NCL) mixes, there is not any positive impact for the presence of nano-cellulose particles on the early strength for all the studied range of contents. However, the late age compressive strength had shown significant enhancement for most of the NCL mixes. At 7 days, the highest compressive strength reached was 45.62 MPa for cement substitution by 0.04% carbon nanotubes with an improvement of 10.1% than the control mix. While the highest early age compressive strength reached 37.26 MPa for cement substitution by 0.02% nano-cellulose, with a reduction of 10.1% lower than the control mix. The relatively low compression strength of nano-cellulose mixes after 7 days can be attributed to that the hydroxyl and carboxyl groups in the cellulose molecules react with Ca^{+2} to form complexes, which can lower the hydration period resulting in retarding the setting time [33]. After 28 days, the maximum compressive obtained for NCL mixes was 51.4 MPa at 0.04% by wt NCL with an improvement of 10.68% than the control mix.

The enhancement in compressive strength after 28 days was due to the high water absorption of nano-cellulose, as they are a class of hydrogels. At late hydration time, nano-cellulose release the trapped water into the cement matrix, which helps in continuing the hydration process of un-hydrated cement particles, which can improve the mechanical properties and the microstructure of the cement matrix [36-37]. While the reduction in 28 days compressive strength more than 0.04% of NCL was due to the formation of clumps, and the agglomeration of NCL particles resulting in weak bond interface with the cement matrix which had a very negative effect on the final mechanical properties of the cement composites [38-39]. As for carbon nanotube mixes, after 28 days, the addition of CNT has a major positive effect on the compressive strength regardless of the ratio of CNT in the cement matrix. Generally, the gain in the compressive strength was a result of the filling effect of CNT in the cement matrix, where CNT fill the nano-gaps within the cement matrix and resulting in enhancing the matrix uniformity. In addition, CNT act as a nucleus to the cement hydration gels which results in the production of chains of reinforced C-S-H. The optimum content of CNT was at 0.04% with a compressive strength of 57.38 MPa with an improvement of 23.58% than the control mix. At using higher contents than the optimum ratio, a slight reduction in the compressive strength was observed but still higher than the control mix; this was due to the agglomeration formed when using larger amounts of CNT particles, these particles got assembled around each other leading to the production of large voids within the matrix and a relative reduction in the compressive strength of the matrix was observed [40]. Theses weak zone is initiated and act as a stress concentrator in the cementitious matrix resulting in premature cracking.

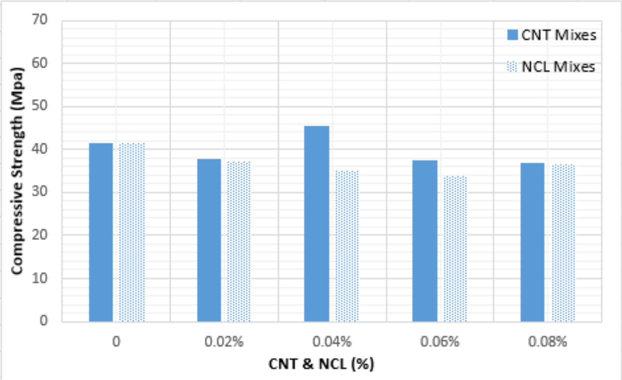


Figure 5: Comparison between 7-days Compressive Strength of CNT and NCL cement mixes

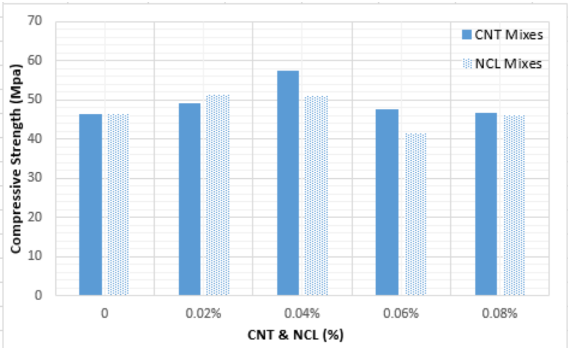


Figure 6: Comparison between 28-days Compressive Strength of CNT and NCL cement mixes

B. *Flexural Strength*

Figure 7 shows the flexural strength of CNT and NCL specimens after 28 days. All NCL mixes exhibited higher flexural strength than the control mixes. For NCL mixes, the higher flexural strength obtained was at 0.04 wt% with strength equals to 152 MPa and with an improvement of about 25% than the control mix. The improvement in the flexural strength can be attributed to; firstly, the result of the previously mentioned above hydrophilic properties of NCL as the hydration of cement accelerated and more CSH formed. Secondly, the higher specific area of NCL improves the nano-fiber matrix interface, which improves the bond between the NCL and the matrix. Thirdly, the nucleation effect of NCL, which improve the cement hydration and produce more CSH volumes [41-42]. Fourth, the indirect sonication process of NCL before adding to the cement matrix, which improves the dispersion of NCL in the cement matrix and improving the nanofiber effect of NCL, and providing bridging effect to resist cracks propagation [38-39]. In addition, the high tensile strength properties of NCL mortar mixes that will be shown later. While for CNT mixes, the maximum flexural strength obtained was 177 MPa at 0.08 wt% with an improvement of about 45% than the control mix, which is higher than the NCL mixes with very remarkable value (almost twice the highest NCL value). This was due to the bridging effect of CNT, which can resist cracks propagation [43-44]. Finally, the slight decrease in flexural strength at the mixes with NCL higher than 0.04% could be due to the agglomeration that occurs in the NCL particles and their poor dispersion in the cement matrix. In which, a weak zone is initiated and act as a stress concentrator in the cementitious matrix premature cracking [45].

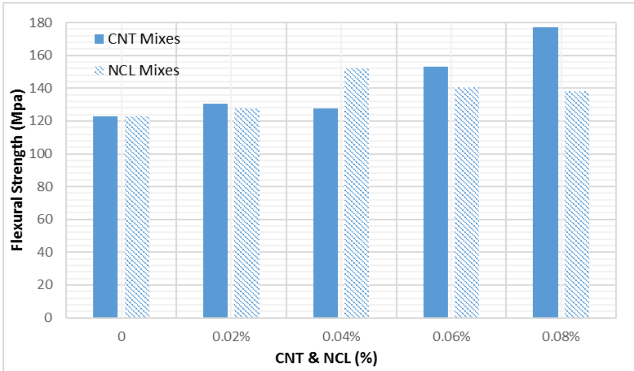


Figure 7: Comparison between Flexural Strength of CNT and NCL cement mixes

C. *Tensile strength*

Figure 8 shows the splitting tensile strength test for CNT and NCL specimens after 28 days of water curing. Generally, both CNT and NCL had improved the splitting tensile strength of the cement mortars. For CNT specimens, the tensile strength increased up to 3.7 MPa with an improvement of 13.1% than the control mix at the substitution level of 0.04 wt% CNT. While NCL mixes reached a higher value with tensile strength equals to 4.52 MPa reporting an improvement of 38.24% than the control mix at the substitution level of 0.02 wt% NCL.

The improvement for tensile strength using CNT and NCL was due to optimum dispersion through the indirect sonication process which they were subjected to prior adding to their mixing with cement paste leading to a denser microstructure and a better performance regarding the crack arrest at the initial level of the nano scale [46]. In addition, the bridging effect of CNT and NCL to the cracks, which suppress crack propagation at the Nano scale [47]. Moreover, the nucleation effect that helps in the production of more Calcium Silicate hydrates (CSH) forming a denser microstructure and can improve all of the mechanical strengths for both CNT and NCL mixes [48]. The enhancement of the NCL was better than the CNT on contrary to the flexural strength results could be due to the better enhancement of the CNT to the compressive strength over the NCL that results in better flexural capacity for the beams with CNT over those with NCL.

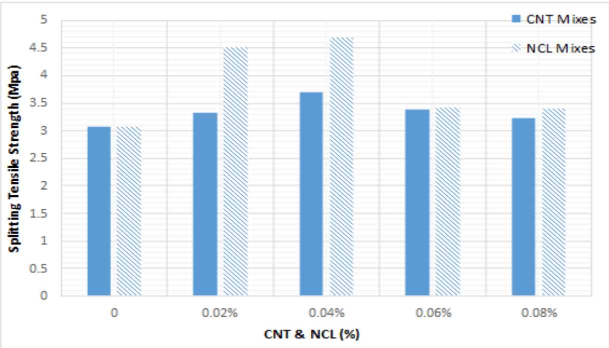
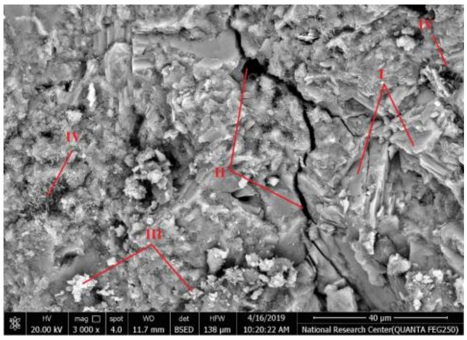


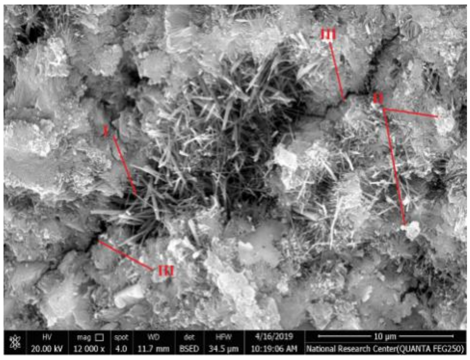
Figure 8: Comparison between Splitting Tensile Strength of CNT and NCL cement mixes

D. *Scanning Electron Microscope:*

Figure 9(a) and 9(b) shows the microstructure of the control mix. In figure 9(a), The CSH phase shown in the form of small isometric grains of hydrated calcium silicates (I). In the middle of the figure 9(a), there is a clear crack (II) with residual bridges formed from the CSH phase. Un-hydrated CSH also observed (III). In addition, Ettringite needles were observed too but with small amounts (IV). In the middle of figure 9(b), the ettringite needles were identified (I). Un-hydrated CSH appears in the form of small grains (II). A micro-crack passing across the entire figure range also observed (III). Figure 10(a) and 10(b) shows the microstructure of cement mortars with CNT at 0.04% wt. It is obvious that the number of un-hydrated cement grains is much less than the control mix and the microstructure is much denser than the control mix. This can be attributed to the uniform dispersion of CNT in the cement paste and the absence of any clumps and this was due to the sonication process of CNT prior to addition to the cement pastes. Moreover, in Figure 10(b), the bridging effect of CNT between the micro cracks of the cement paste is clear and well identified, and this is because of the degree of well dispersion of CNT in the cement matrix, and this can explain the enhancement of the compressive strength of the cement mortars after 28 days for all percentages of CNTs. While Figure 11(a) and 11(b) shows the microstructure of cement pastes with 0.04% wt. NCL. In Figure 11(a), the amount of un-hydrated cement is much less than those in CNT mixes and the control mix. This can be due to, as mentioned previously in the compressive strength, the water absorption effect of NCL, which release late water that helps in the hydration of the un-hydrated cement particles, and consequently enhances the cement matrix via reducing the porosity, and the micro-pores, as well as improving the strength of the cement matrix.

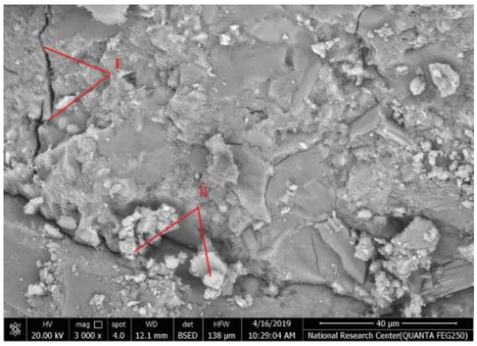


(a)

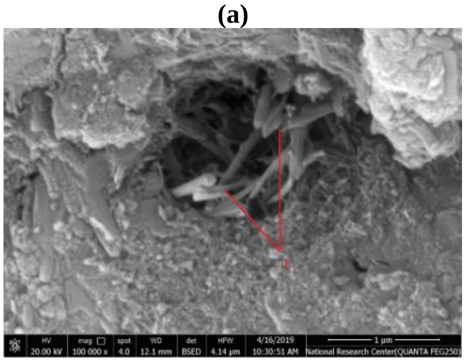


(b)

Figure 9: SEM of the control mix; (a) magnification 40 µm, (b) magnification at 10 µm

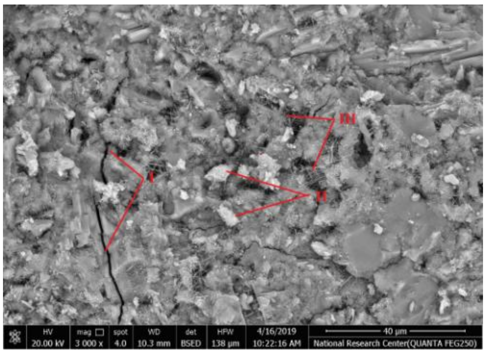


(a)

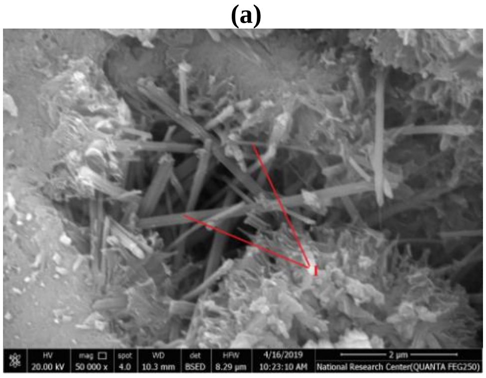


(b)

Figure 10: SEM of the CNT mix with 0.04% wt; (a) magnification 40 µm, (b) magnification at 10 µm



(a)



(b)

Figure 11: SEM of the NCL mix with 0.04% wt; (a) magnification 40 μm, (b) magnification at 10 μm

E. *X-RAY Diffraction*

XRD performed to detect changes in the hydration products due to the presence of Nano-materials. Due to their crystalline nature, calcium hydroxide, calcium silicate, and silica peaks appear clearly in the XRD diagrams, and can directly be detected by this technique. Statistically significant changes in the peak values of crystalline phases, such as calcium hydroxide and calcium carbonate, as well as the appearance of new peaks, are indicative of a change in the phase composition of existing phases or formation of new phases, respectively, in the microstructure. From figures 12 to 14, the CH content in the control mix was higher than that in CNT and NCL mixes, and this can explain the lower compressive strength compared with CNT and NCL cement paste mix. The CH peaks at 2 theta of 34 were much higher in NCL mix than CNT one. This confirms the effect of NCL on increasing the hydration process and increasing the calcium hydroxide content leading to enhancing the tensile and flexural strength, and the microstructure than that of CNT. As well as these results confirm that the major effect of the CNT over the NCL in enhancing the compressive strength is generally related to the filling effect over any other effects.

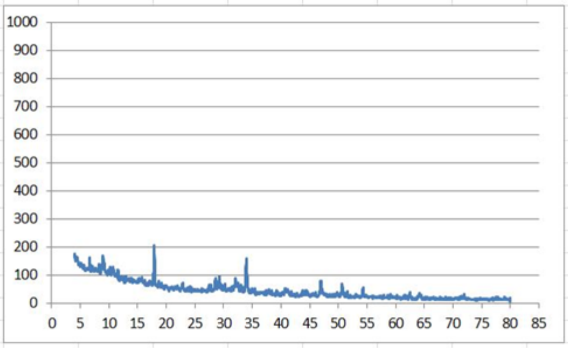


Figure 12: XRD for the Control mix

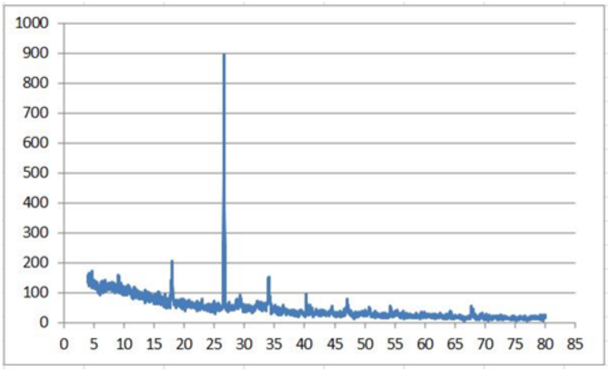


Figure 13: CNT mortar Mix

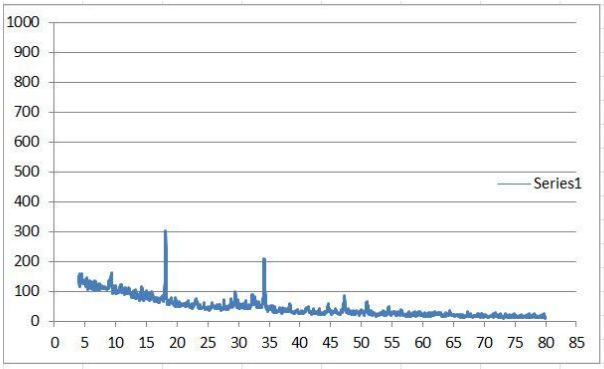


Figure 14: NCL Mortar Mix

F. *Atomic Force Microscopy:*

Atomic Force Microscope used to study the surface roughness of the cement matrix and the effect of inclusion of both CNT and NCL on the microstructure and the surface texture of the cement paste. Figure 15 shows the AFM of the control mix, in which the spherical particles of the C-S-H appears with different sizes as well as the presence of flat areas indicating the presence of the calcium hydroxide crystals. Figures 16 and 17 show the AFM for CNT and NCL cement paste mixes. The spherical particles of C-S-H gel had been interlayered with the CNT particles resulting in arrayed and homogenously reinforced C-S-H with overall particle sizes of 1.07 μm with almost the same height of the control mix particles. On the other hand; the particles size has been increased dramatically to reach 1.71 μm for NCL mixes. The ribbed arrays in the NCL mix were significantly not uniform as compared to the CNT mix, while the C-S-H crumbles were clearly identifiable than the CNT mix. This can be attributed to the effect of NCL on the hydration process, as illustrated in section 3.1, which produce a tight surface, reduce the size of pore structures, and block the penetration of any fluids to the cement paste. While in CNT cement pastes the good dispersion of the nano-carbon tubes had been greatly recognized by the ribbed texture as shown in Figure 16. It can be concluded that CNT had a great influence on the cement composites via their physical performance on contrary to the NCL that their effect appears to be mostly due to their impact on the hydration process.

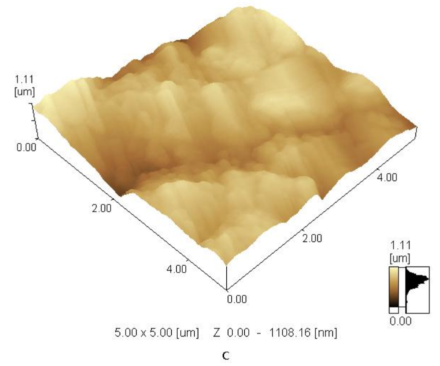


Figure 15: The AFM topographical of control paste

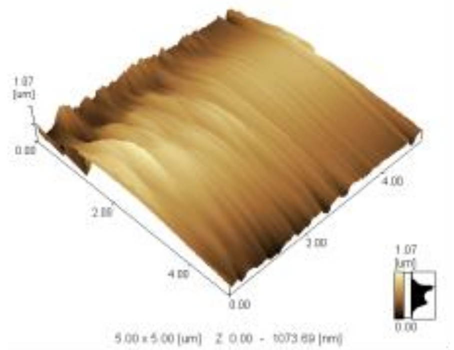


Figure 16: The AFM topographical of 0.04% by wt CNT cement paste

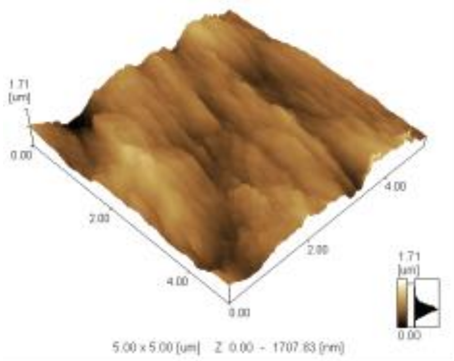


Figure 17: The AFM topographical of 0.04% wt NCL cemnt paste

IV. CONCLUSIONS

This paper focused on the performance of Nano reinforced cement mortars with different Nanofiber particles incorporation; CNT, and NCL. From the test results, conclusions can be summarized as follows:

• Generally, the findings out of the research plan demonstrate the potential utilization of nanofiber reinforcement to modify the mechanical properties of the cementitious matrix.

• The optimum addition of the NCL particles at 0.04% by cement weight helped in improving the tensile, and flexural strengths by about 40%, and 30% respectively as compared to the control mix, while the maximum enhancement of the compressive strength was of about 11% only.

• The optimum addition of the CNT particles at 0.04% by cement weight helped in improving the tensile, strength by about 13%, while the optimum percentage for enhancing the flexural strength was 0.08% with gain of about 45% as compared to the control mix, while the maximum enhancement of the compressive strength was of about 25% for the 0.04%.

• From the AFM micrographs, the distribution of the C-S-H clusters showed to be more ribbed, and uniform with the CNT mix than the NCL mix. These microstructure characters imply that the strength that CNT had a great influence on the cement composites via their physical performance on contrary to the NCL that their effect appears to be mostly due to their impact on the hydration process, which is in accordance with the mechanical strength results and the SEM and XRD results.

• Finally, the Nano cellulose fibers could be a great alternative to CNT as Nano reinforcement to the cement composites not only to enhance the mechanical strengths but also in enhancing the microstructure of the cement matrix.

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