

Compressive and Flexural Strength Behavior of Ultra-high Performance Mortar Reinforced with Cellulose nano-fibers

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Abstract— Cellulose fibers, because of their chemical and physical characteristics, are compatible with other materials to be used for the production of building components. This paper presents the influence of using cellulose nano fibers (CNFs) made from plant-derived cellulose as a reinforcement in ultra-high performance (UHP) mortar. In this study, the dispersion method of CNFs using manual and mechanical mixing was also observed. The effects of different dosage of CNFs, namely, 0.005%, 0.01% and 0.015% by wt. of binders (premixed low-heat cement and silica fume) with a constant water to binder ratio of 0.15, were evaluated based on the compressive and flexural strengths at the seventh day after steam curing. Results show that the highest compressive strength value of 184 MPa was reached by UHP mortar sample containing 0.005% CNFs by wt. of binders. However, addition of more CNFs content up to 0.015% did not result in further improvement. Based on load-CMOD curves, UHP mortar reinforced with 0.005% CNFs was found most effective in enhancing the energy absorption capacity and toughness index with flexural strength at peak load of 14.44MPa (36% higher than control UHP mortar). The results indicate the well-post crack behavior of CNFs mortars in comparison with control UHP low-heat cement mortar. Moreover, Scanning Electron Microscopy (SEM) analysis shows that the phenomenon of bridging effect of CNFs could not be significantly detected since the short fiber might be fractured under loading due to less bonding. Furthermore, this study concludes that even a low volume fraction, i.e 0.005%, of CNFs is very sufficient in increasing the ductility of ultra-high performance mortar.

Keywords— cellulose nano-fibers; compressive; flexural; dispersion; SEM.

I. INTRODUCTION

Concrete is generally considered brittle material with low tensile strength, limited ductility and poor strain capacity when compared to other building materials which are normally used in construction i.e.s metals and polymers. The use of steel with its high tensile strength to reinforce concrete offsets this limitation and provides high resistance on compressive and tensile strength, and much greater ductility and toughness of concrete.

Recently, the incorporation of fibers has been shown to improve the properties of cement-based materials through its characteristics as reinforcement in controlling crack initiation and propagation resulted from external applied stress or deformation from the environmental effects, including thermal and shrinkage strains that cause volumetric instability. The inclusion of fibers in concrete improves the fracture, fatigue and impact properties of material from brittle to ductile. The diffusion of liquids and gases in concrete can also be eliminated and, therefore,

effectively improves the resistance of fiber-reinforced concrete on carbonation and corrosion attack leading to enhance the durability and lengthen the service life of the concrete structure [1]. Furthermore, the presence of fiber reinforcement shows potential to be a high-performance material with cost-effectiveness in repair and rehabilitation of building construction.

Fibers that are normally used in cement-based material can be steel, polypropylene, nylon, basaltic, glass etc, depending on its source [2]. Other forms are carbon, asbestos, wood, cellulose, and variation of synthetic fibers with some of the fiber materials are commercially available. Some studies on the use of fibers have been intensively established and showed interesting results. For example, a study conducted by [3] reported an improvement of fiber-reinforced concrete on tensile and bending strength by 15% and 20%, respectively. Other studies on carbon nano-fibers in mortar and concrete properties are also reported. Reference [4] investigated that the use of carbon nano-fibers increased the flexural strength up to 45% and observed

improvement in Young's modulus of at least 50% over plain cement sample. It was also explained through Scanning Electron Microscopy analysis that the incorporation of carbon nano-fibers reinforced the cementitious matrices by bridging nanopores and nanocracks. The usage of carbon nano-fibers also improved fracture resistance when compared to the multiwall carbon nanotubes (MWCNTs) samples. Similar observation was also reported as in [5]. It was found that the flexural strength, Young's modulus and toughness increased up to 40%, 75%, and 35%, respectively, with the incorporation of carbon nano-fibers in cement composites. The study also concluded that carbon nano-fibers can be used at a concentration as 0.048wt% with a water cement ratio of 0.5. Another study as in [6] reported superior improvement in flexural strength (87%), Young's modulus (95%), and fracture toughness (119%) of cementitious nano composites reinforced with well dispersed carbon nano-fibers.

Reference [7] investigated the effect of carbon nano-fibers addition on the mechanical properties of cement mortar. In this study, a dosage of 0.2% carbon nano-fibers with water/cement ratio of 0.35 to 0.5 was utilized with appropriate sonication techniques. It was found that the addition of carbon nano-fibers increased the 28-days compressive and flexural strengths of cement mortar up to 217% and 50%, respectively. These results are also in line with the work in [8] which found an improvement of average flexural strength up to 82% higher than control cement paste due to the inclusion of 0.1 and 0.2% carbon nano-fibers.

In terms of durability of cement pastes with carbon nano-fibers, a study, as in [9], performed a molecular dynamic simulation and found the superior improvement in interactions between cement pastes and surface treated carbon fibers, making the carbon nano-fibers composite more ductile, retaining some residual strength post peak load. Reference [10] studied the effectiveness of using cellulose and polypropylene fibers on chloride diffusion induced corrosion. The results showed that the use of 0.1% and 0.3% volume fractions of non-metallic fibers limited the amounts of free chlorides thus delayed the initiation of corrosion in reinforcing steel. Among the two types of fibers investigated, cellulose fibers appeared to be more effective in chloride binding than the polypropylene fiber. The inclusion of cellulose fibers in cementitious materials was also effective in mitigating drying shrinkage-induced cracking and significantly reduced the crack width propagation. When compared to cellulose nano crystals, cellulose nano fibers were also found better with high yield and low-cost character [11].

Based on some available results, the beneficial effect of using nano-fibers on the mechanical and microstructure characteristics of cement composites materials can be explained by factors such as: 1) the enhancement effects of nano-fibers to react chemically with cement components; 2) the contribution of nano-fibers to formation of a dense microstructure thus improving the pore structure and controlling nanoscale cracks; and 3) the improvement of the interfacial interaction between the nano-fibers and the cement phases. Despite the benefits of using nano-fibers on cement composites, it is very important to control their dispersion method when added into the mixture. Some

dispersion methods have been evaluated through grinding, micro-fluidization, acid hydrolysis and homogenization using various solvents to isolate the cellulose nano-fibers and avoids self-agglomeration [12]. Proper dispersion will maximize the benefit of using nano-fibers to reduce the fiber free area in the mixture and improves the resistance properties of samples on durability including autogenous and drying shrinkage cracking [13]. However, the dispersion method of nano-fibers remains challenging since it depends on the characteristics of nano-fibers which should be also controlled and optimized to create a well-dispersed nano-fibers material.

Nowadays, nano technology development in construction materials engineering has led cellulose nano-fibers to be one of the most advanced green reinforcement materials due to its high strength properties, relative low cost and availability.

Cellulose nano-fibers (CNFs) are grouped as natural fibers obtained from the processing of wood and plants that can be isolated through homogenization, grinding, micro-fluidization, acid hydrolysis and oxidation process [12]. Reference [11] reported on the CNFs production that the uniform dispersion of cellulose nano-fibers can be achieved through oxidation process of hydroxyl groups of from the cellulose fibers to carboxylate groups that has a negative charge on the CNFs surface that can prevent agglomeration due to the electrostatic repulsion force. Environmentally, cellulose nano-fibers has more advantages compared to other nano-fibers such as carbon nano tube [14]. In comparison to the traditional fibers, cellulose fibers are more in elastic modulus with high length-to-diameter ratios, making them effective in stabilizing cracks.

In terms of nano size, cellulose fibers are expected to increase the toughness and the fracture energy performance of the material [15]. Based on previous research, the addition of CNFs may provide extraordinary flexural and compressive strength increase as well as improving the microstructure and degree of hydration of the matrix. The flexural and compressive strength of cement pastes containing 0.015% CNFs achieved 15% and 20% higher strength, respectively, compared to control paste [11]. The use of CNFs can also modify the performance of cement paste by increasing the flexural strength of approximately 30% with only 0.2% by weight of cement [15]. From the study by [16] the combination of 3% micro and nano-cellulose fibers in concrete was found to improve the fracture energy by more than 50%.

Among the various results on the properties of cement composites with the inclusion of cellulose nano-fibers, there are very less reports and knowledge available on the behavior of ultra-high performance mortar (UHPM) containing CNFs. Ultra-high performance mortar typically consists of cement, supplementary cementitious materials, high range water reducing admixtures, sand and reinforcing fiber with low water cement ratio [17]. UHPM has superior strength (maximum compressive strength is 200 MPa), high workability, higher packing density and durability which can be used for repair work, restoration and maintaining existing building or new construction [18], [19]. However, limited application of UHPM products is largely due to the high economic cost and difficulties in manufacturing process, hence, conducting experimental works on the behavior of

CNFs as reinforcement in ultra-high performance mortar is still very much required.

This study investigates experimentally the effect of cellulose nano-fibers (CNFs) on mechanical properties of ultra-high performance mortar based on the 7-day compressive and flexural strengths test. The dispersion method of CNFs was also evaluated by conducting mechanical and manual mixing using some available laboratory instruments. The evaluation of dispersion method is very important to bring about greater knowledge that can be applied when large volume of dispersed CNFs is considered. Furthermore, the findings will give more understanding that CNFs can offer higher contribution as an alternative material in cement-based systems with potential to advance both performance and sustainability with low cost production in the construction field.

II. MATERIALS AND METHODS

A. Materials

The materials used in this experiment are the premixed low-heat cement (C) and silica fume (SF) at mass content of 0.82 and 0.018. A natural silica sand with average size of 0.212 mm and cellulose nano-fibers (CNFs) with suspension (98% water) made from plant-derived cellulose produced by SUGINO machine's using ultra-high pressure water jet technology are also used in this study. The CNFs are in a gel-like form as can be seen in Fig. 1 with their properties are presented in Table 1.



Fig. 1 Image of CNFs used in this experiment

TABLE I
PHYSICAL PROPERTIES OF CELLULOSE NANO-FIBERS

Specifications	CNFs
Raw materials	Cellulose
Length	Standard
Width (nm)	10-50
Specific surface area (m ² /g)	120
Viscosity (mPa·s)	6,000

TABLE II
MIXTURE PROPORTIONS (WT.%)

Series	Mix	W/B	SP/B	D/B	S/B	CNFs/B
1	Control	15	1.5	0.02	48	-
2	CNFs-0.005	15	1.5	0.02	48	0.005
3	CNFs-0.010	15	1.5	0.02	48	0.010
4	CNFs-0.015	15	1.5	0.02	48	0.015

Note: W= Water; B= Binder; SP= Superplasticizer, D= Defoaming agent, S=Sand, CNFs= Cellulose Nano Fibers

Ultra-high performance mortar containing different percentage of 0.005%, 0.01% and 0.015% CNFs by wt. of binders (C+SF) with a constant water/binder ratio of 0.15 were prepared with the composition is listed in Table 2. In order to reduce the water and air contents, a polycarboxylate-based superplasticizer (SP) and polyether-based anti-foaming agents (D) with density of 1.05 were added into the mixtures.

B. Methods

The experimental works are divided into three parts. Part 1 of investigates the dispersion method to obtain well-dispersed cellulose nano-fibers before adding into UHP mortar mixture. Three dispersion methods were conducted by using: manual laboratory hand-mixer (M), omni mixer homogenizer (NISSEI ACE Type AM-3 (H)) and ultrasonic homogenizer (SONICTAR 85 (U)). The images of the instruments can be seen in Fig. 2, with the blades of each instrument are shown in the inset. At first, 10 minutes of mixing time was selected to evaluate the dispersion degree of CNFs on each method based on 7-day flexural strength of UHP mortars. The CNFs percentage of 0.005% by wt. of binders was used for this purpose. After testing, the method which produced UHP mortar with highest flexural strength result was then selected for further investigation in Part 2, which is on the mechanical properties including compressive and flexural strength tests of UHP mortar with 0.01% and 0.015% CNFs addition.

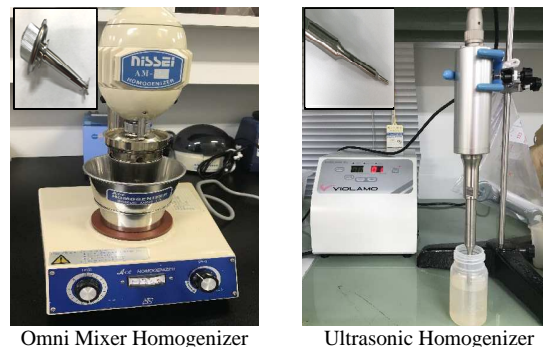


Fig. 2 Instruments used for Cellulose Nano Fibers dispersion

Mortar cylinder specimens in size of 50mm in diameter and 100mm height for compressive strength test and 40x40x160mm³ mortar bars for flexural strength test were cast after mixing the mortar constituents in an omni-type laboratory mixer (capacity of 5 L). In the mixing procedure, cement and sand were firstly dry-mixed for 1 min, and the dispersed liquid containing water+SP+DA+CNFs were then added into the mixtures and mixed for 3 min with medium speed and lasted approximately 4 min with high speed to separate doughlike clumps that was formed during the first minutes until the mixture reached the pastelike state. The workability of UPH mortar was also evaluated through flow table test conducted according to [20]. After casting, the specimens were then placed into the oven for standard steam curing i.e. 48 hours at a constant temperature of 90°C. Afterwards, the samples were stored in the curing room with temperature of 20°C and RH approximately 95% until the testing days. In the flexural strength test, the three-point

bending test on single-edge notch ultra high performance mortar containing cellulose nano-fibers was conducted based on JCI-S-002-2003 recommendation with a loading rate of 0.01 mm/min. The experimental set-up can be seen in Fig. 3. The notch was made by wet sawing at mid-span under the mortar prism samples with 20 ± 1 mm depth. The load and the crack mouth opening displacement were measured through the load cell and attached clip gauge on the UHP mortar specimens using Instron universal testing machine (UTM) with capacity of 30 kN. In part 3, microstructural analysis was performed using Scanning Electron Microscopy method, conducted on the small piece of fractured specimen taken during compression test.

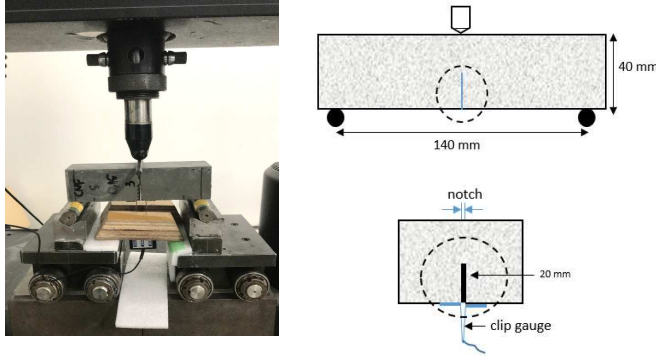


Fig. 3 Experimental set-up on notched beam under third-point loading

The energy absorption capacity (g) of the ultra high performance mortar containing CNFs that reflects the effectiveness of CNFs in improving the performance of UHP mortar was calculated based on the area under the load-deformation curve from zero strain to ϵ_{ts} (strain at the peak load obtained from flexural strength test) using equation (1).

$$g = \int_0^{\epsilon_{ts}} \sigma(\epsilon) d\epsilon \quad (1)$$

The formula to calculate the residual flexural strength is shown in equation (2), where f = the residual flexural strength corresponding with CMOD that provides an indication of the crack tolerance and post-crack behavior of UHP mortar; P = the load corresponding with CMOD; l = the length of span (mm); b = the width of specimen (mm); h = the distance between the tip of the notch and the top of the prism mortar specimen (mm).

$$f = \frac{3 \cdot P \cdot l}{2 \cdot b \cdot h^2} \quad (2)$$

In order to evaluate the effectiveness of fibers, the toughness index of UHP mortar with and without CNFs was quantified by adapting some parameters as reported in [21] and can be seen in equation (3)-(6):

$$W_{1,2} = \int_0^{d_{ts}} P(d) dd \quad (3)$$

$$W_2 = \int_{d_{fcs}}^{d_{ts}} P(d) dd \quad (4)$$

$$W_3 = \int_{d_{ts}}^{d_{end}} P(d) dd \quad (5)$$

$$W_{1,2,3} = \int_0^{d_{end}} P(d) dd \quad (6)$$

In these equations, $W_{1,2}$ = amount of work up to the peak load, d_{ts} = CMOD at the peak load, d_{fcs} = CMOD at initial cracking, W_2 = amount of work from d_{fcs} to d_{ts} , W_3 = amount of work beyond the peak load, d_{end} = CMOD at the end of loading, and $W_{1,2,3}$ = amount of total work.

III. RESULTS AND DISCUSSIONS

A. Dispersion Method of CNFs based on Flexural Strength of UHP Mortar

This part reports the effectiveness of dispersion method conducted on cellulose nano-fibers based on the flexural strength of UHP mortar bar samples blended with 0.005% CNFs by wt. The results of the 7th-day flexural strength from three mortar bar samples containing 0.005% CNFs prepared using three different methods for 20 min of stirring were obtained. Based on the results, it was found that dispersion of CNFs by using ultrasonic homogenizer (U) provided an improvement on the 7th-day flexural strength of UHP mortar, compared to other methods, which are omni mixer homogenizer (H) and manual laboratory hand mixer (M). The results are reported in Fig. 4. It can be seen that the 7th-day flexural strength of UHP mortar with CNFs prepared using ultrasonic homogenizer is 27 MPa which is higher than the results gained by UHP mortar with CNFs prepared by other methods indicating that ultrasonic homogenizer at a certain power promoted water swelling effect through ultrasonic wave that can broke-up the hydrogen between the cellulose fibers and led to better dispersion.

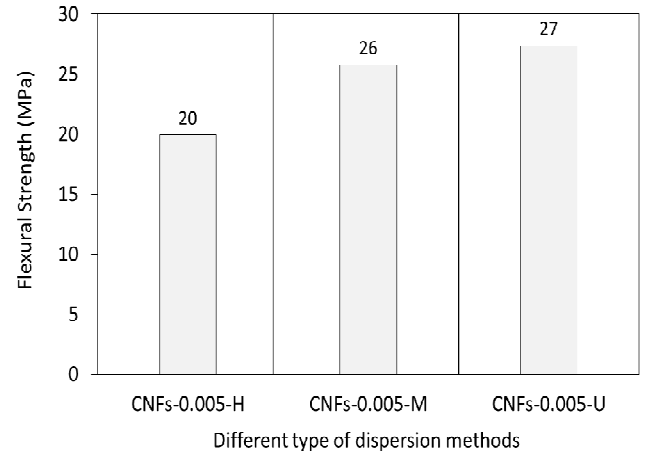


Fig. 4 Flexural strength of UHP mortars containing CNFs prepared with different dispersion methods

However, increasing the mixing time from 10 min to 30 and 60 mins using ultrasonic homogenizer resulted in decreasing the strength performance. As seen in Fig. 5, increasing the mixing time of CNFs up to 30 and 60 mins lower the flexural strength of UHP mortar indicating the re-agglomeration of CNFs occurred which afterwards hindered the potential effect of CNFs when blended into UHP mortar mixtures. Based on the results, it was concluded that 10 min of stirring using ultrasonic homogenizer would be long enough to obtain well-dispersed liquid of CNFs and therefore, this method was used to evaluate the mechanical properties of UHP mortar containing higher percentage of CNFs i.e. 0.01 and 0.015% CNFs.

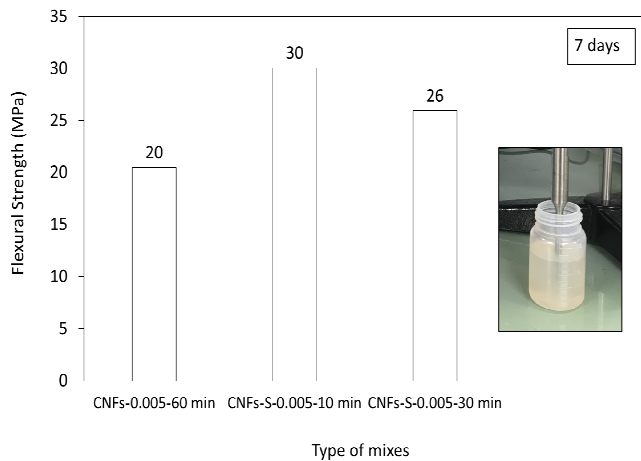


Fig. 5 Flexural strength of UHP mortar containing CNFs prepared by using ultrasonic homogenizer

B. Effects of CNFs on the 7th day compressive strength and workability of UHP mortars

The compressive strength results of UHP mortars with and without the CNFs inclusion can be seen in Fig. 6. The results show an improvement of compressive strength reached by CNFs mortar samples after the 7th day of curing, indicating the positive influence of using CNFs in blended low heat cement mortar. The highest compressive strength value of 184 MPa was reached by UHP mortar sample which contained 0.005% CNFs by wt. of binders. In this case, the compressive strength was about 8% higher than control mortar and approximately 4-8% higher than 0.01% and 0.015% CNFs mortars, respectively. The results indicates the effectiveness of 0.005% CNFs in improving the resistance of UHP mortar on compression load attributed to the distribution of CNFs in the line of compression axis that refines the fracture toughness. The presence of CNFs is presumably provides close spacing and strong bonding to the cement matrix due to its high specific surface that increases density and influences the compressive strength development. The high surface area to volume ratio of CNFs also accelerates the chemical reactivity and promotes the formation of CSH gel in cement composites [22]. However, addition of more CNFs content up to 0.015% did not result in further improvement. The 7th day compressive strength of 0.015% CNFs even had similar strength with control mortar at w/b ratio of 0.015 which is about 170 MPa. Reference [18] also reported the reduction of compressive strength and dynamic of modulus of elasticity when the nano-fibers was increased from 0.1% to 0.25%. Less improvement of compressive strength on CNFs mortar was suggested due to the formation of reactions between the hydroxyl and carboxyl groups in cellulose molecules with Ca^{2+} that can delay the induction period of hydration and setting time, as reported in [11]. In this study, the author reported that the improvement of strength could be obtained at later ages due to the ability of CNFs in releasing water and promoted higher degree hydration between water and cement particles and reduced the quantity of un-hydrated particles thus denser the pore structure of CNFs mortar. This internal curing capabilities of cellulose fibers was also mentioned by [13].

The characteristic of CNFs with hydrogels type that has high water absorption can be explained from the workability results of CNFs mortars as seen in Fig. 7. The figure shows the decrease of flow diameter obtained as the volume fraction of CNFs was increased up to 0.015% by wt. These results can also be an evident on the possible agglomeration occurred as the CNFs content increased. Reference [23] reported that increasing the volume fraction of CNFs led to weaken the bonding interfaces and promoted stress concentration that was assumably resulted from the porosity and compaction difficulties also agglomeration of CNFs that has a lot of hydroxyl groups. Furthermore, further research is still needed to evaluate the dispersion performance of CNFs particularly when using higher percentage. The optimization of dispersion method by using higher amount of superplasticizer or different type of superplasticizer and also the homogenization protocol are some interesting areas to investigate by also considering the later age performance of UHP mortar containing CNFs.

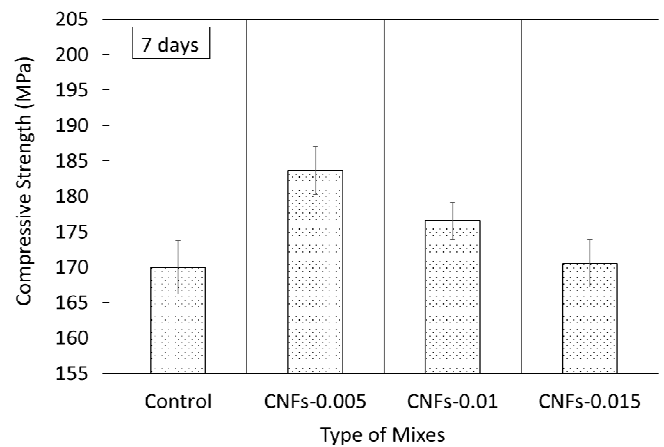


Fig. 6 Compressive strength of CNFs UHP mortars after the 7th day

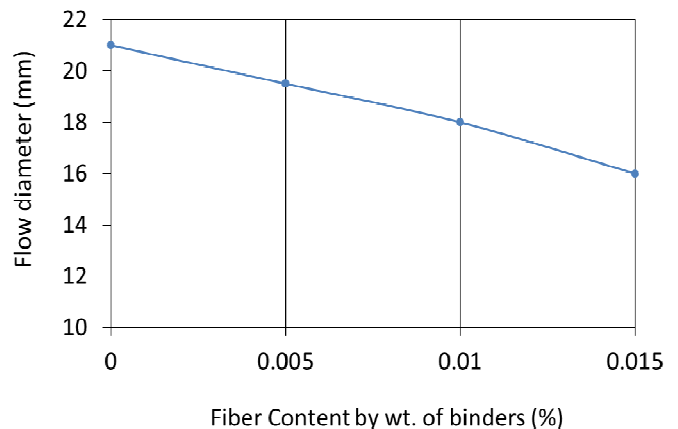


Fig. 7 Flow diameter of UHP mortars with and without CNFs

C. Effects of CNFs on the 7th day flexural strength of UHP mortar

Figure 8 presents the results of the load-CMOD curves of UHP mortars containing different percentage of CNFs. The loading speed of 0.1 mm/min was applied for the experiment.

From the figure, it can be noted that the ultimate load related to the maximum contribution of the fiber can be found in UHP-mortar containing 0.005% CNFs. The ultimate load of mortar containing 0.005% CNFs reached the highest peak load of 1.54 kN (1540 N), followed by 0.01% CNFs which are approximately 36% and 17%, respectively, higher than the ultimate load of control UHP mortar indicating the well-post crack behavior of CNFs mortars in comparison with control UHP mortar. On the other hand, there is no significant difference was observed on the ultimate load of UHP mortar with 0.015% CNFs when compared to control UHP mortar that is assumably due to the agglomeration of CNFs. As seen in Fig. 6 and Table 3, the ultimate load of 0.015% CNFs mortar is 1143 N while the control mortar is 1131 N. In addition, when the residual strength at peak load of each mixes is calculated, the results show that CNFs-0.005, CNFs 0.01 and CNFs 0.015 UHP mortar samples exhibited 14.44 MPa, 12.47 MPa and 10.72 MPa, respectively, showing the reduction of strength as the CNFs content is increased. It can be concluded that minimum of 0.005% CNFs is needed to enhance the ductility of UHP mortar including the post-cracking behavior. In comparison to other study, the experimental work conducted in [24] and [25] showed an improvement in flexural strength when CNFs was blended at 0.1% of volume fraction and reduced when the content of CNFs increased up to 0.4% due to fiber agglomeration. Reference [26] also concluded that in order to improve the mechanical properties, the optimum fiber content will depends on the source of cellulose fiber and the preparation as well as the production method of CNFs.

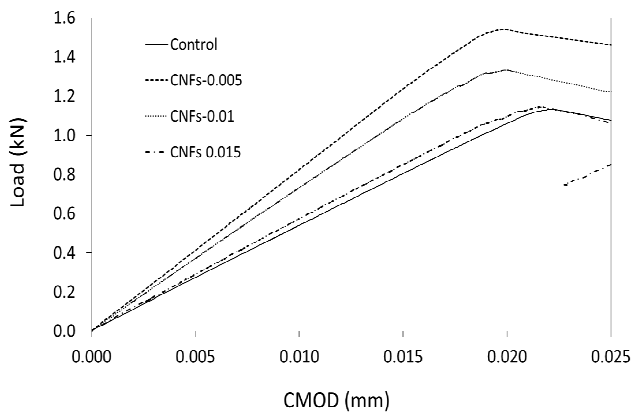


Fig. 8 Flexural strength results of CNFs UHP mortars at the 7th day

D. Energy absorption and toughness index of UHP mortar containing CNFs

This section discusses the behavior of CNFs on energy absorption capacity and toughness index of UHP mortar. For the evaluation of the energy absorbed of UHP mortar with CNFs that refers to the area under load-CMOD curve, three zones are divided as seen in Fig.9. In this figure, load-CMOD curve of 0.01% CNFs mortar is taken as an example. Zone A is the linear elastic stage where the load and crack displacement growth up to the crack initiation load (σ_1) and reached the peak load (σ_2) whereas zone B and C are strain hardening stage and softening stage, respectively. In this study, the strain hardening stage is selected to explain the reinforcing effect of CNFs in the UHP mortar through the

scatter of fiber distribution. Moreover, the toughness index is calculated by dividing the total area of Zone A and B under load-CMOD of CNFs mortars by the area under the same curve of control UHP mortar. The softening stage behavior in Zone C after the peak load is not discussed in this paper.

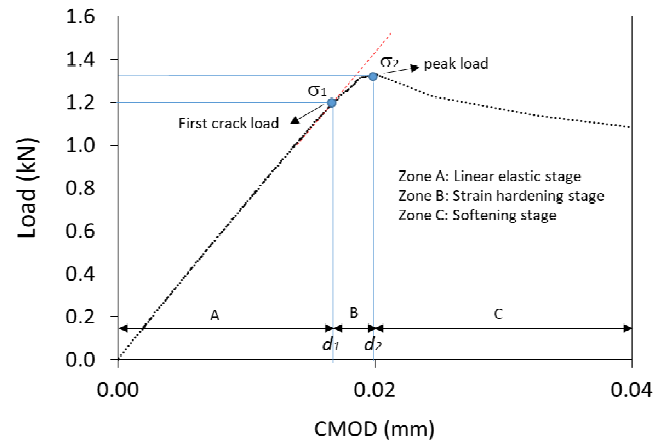


Fig. 9 Load-CMOD curve example on UHP mortar with CNFs

Based on Fig. 9, it can be noticed the phenomenon of slow crack growth to the peak load. Afterwards, there is a sudden drop that can be observed after reaching the peak load when the matrix cracked. The load is then back up to the level and then gradually decreases as the increase of displacement until the test done. This trend could not be found in control UHP mortar containing low heat cement only. Moreover, the area under load-CMOD curve of UHP mortar with 0.005% CNFs is obviously larger than the curve of UHP control mortar indicating more energy is absorbed during cracking of the CNFs 0.005 mortar than in control one. Based on the calculation using equation (1), the energy absorptions of CNFs-0.005 and CNFs-0.01 mortar samples are 14.42 kJ/m³ and 12.23 kJ/m³, respectively, higher than control UHP mortar and there is no significant improvement is observed as the percentage of CNFs was increased up to 0.015% (see Table 3). Moreover, the toughness index of UHP mortar containing CNFs compared with control mortar can be seen in Fig. 10.

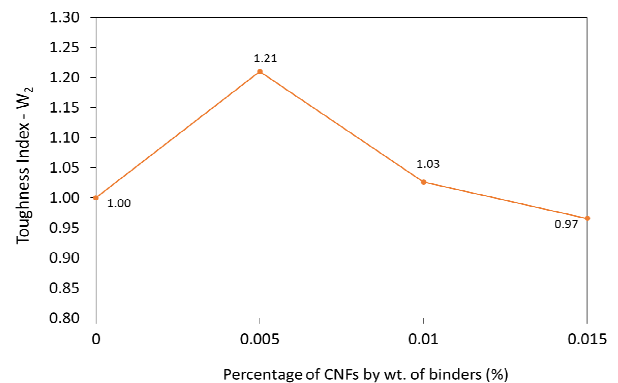


Fig.10 Toughness index of UHP mortar containing CNFs

The figure shows that the UHP mortar with CNFs has higher capability in carrying further loads demonstrating higher tough and more energy absorbed during its deflection. The highest toughness index which is 1.21 was found in UHP mortar with 0.005% CNFs. The index value then

started to reduce as the CNFs content was increased up to 0.01 and 0.015% (see Fig.10 and Table 3). On the other hand, the toughness index of control UHP mortars is equal to 1 since the mortar specimen failed right after the first crack occurred. The toughness index is in line with the energy absorption capacity results where UHP mortar with 0.005% exhibited the highest correlation compared to CNFs 0.01% and 0.015% UHP mortars indicating that even a low volume fraction i.e 0.005% of CNFs is very effective in increasing the ductility of ultra-high performance mortar.

TABLE III
ULTIMATE LOAD, RESIDUAL FLEXURAL STRENGTH, CMOD AND ENERGY ABSORPTION CAPACITY OF UHP-CNFS MORTAR

Type of Mixes	Peak load (N)	Residual flexural strength (MPa)	CMOD (mm)	Energy absorption capacity (kJ/m ³)	Toughness Index
Control	1131	10.52	0.022	11.92	1
CNFs-0.005	1540	14.44	0.020	14.42	1.21
CNFs-0.01	1330	12.47	0.020	12.23	1.03
CNFs-0.015	1143	10.72	0.022	11.51	0.97

E. Scanning Electron Microscopy of UHP mortars containing cellulose nano-fibers

Figures 11a-d show the images of fractured mortar specimens with and without CNFs obtained from Scanning Electron Microscopy analysis. This analysis was conducted in order to investigate how the CNFs influences the matrix binder of cement mortar. It can be seen in Figures 11b-d, the presence of CNFs impeded in the UHP cement mortar matrix surrounding the hydration products indicating the strong interfacial bonding between the CNFs and cement matrix. In addition, some areas in the images show the phenomenon of bridging effect of CNFs (see Fig. 11b). However, the significant role of CNFs to act as reinforcement could not be found clearly due to the short length of CNFs since the short fiber might be fractured under loading due to less bonding. Additionally, the cracks in UHP mortar with CNFs are observed do not propagate around the interfacial zone but cut through the aggregate due to the very dense packing of UHP mortar matrix. Reference [25] also commented the limitation of CNFs in acting as reinforcement on crack-bridging system due to its very fine size compared to macro- and micro-sizes fiber. The same observation was reported in [27] and [28] where the short length of fibers can be pulled out as the crack increases thus not have enough ability in preventing the growth of macrocracks matrix.

Despite of that, the fineness of CNFs can alter the hydration reactions kinetics and enhances the mechanical properties. In this study, the inclusion of CNFs is suggested enhances the fibre-matrix interaction and improves the microstructure of UHP mortar thus increased the mechanical properties of UHP mortar. Nevertheless, further increasing the CNFs content up to 0.015% leads to initiate the bundle formation of CNFs as seen in Figures 11c and d. This bundle formation is assumed to be the unreacted defects due to the

agglomeration of CNFs that induces higher stress concentration under loading thus reduced the mechanical strength of CNFs mortars as discussed in the previous sections.

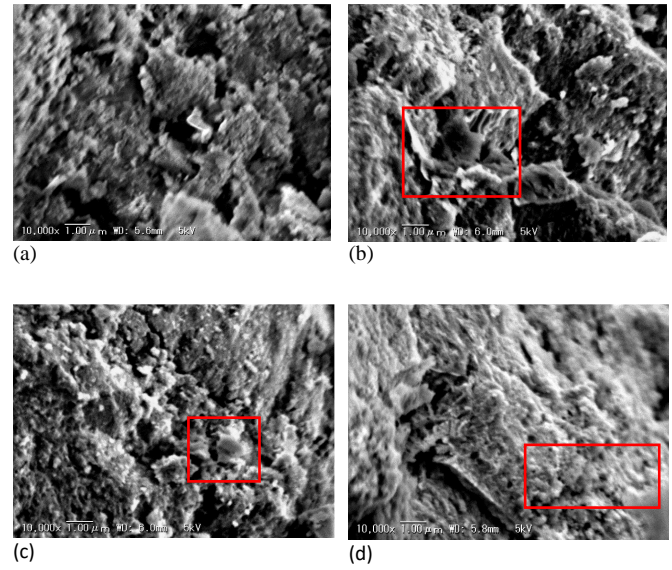


Fig. 11 SEM images of (a) control mortar, (b) 0.005% CNFs mortar, (c) 0.01% CNFs mortar, (d) 0.015% CNFs mortar

IV. CONCLUSIONS

From the limited experimental results obtained on this experimental works, some conclusions can be drawn as follows: Dispersion method of CNFs using ultrasonic homogenizer can be applied to achieve uniform dispersion of suspended CNFs when compared to other dispersion methods such as omni mixer homogenizer and laboratory hand mixer. However, the mixing time should be properly considered since the longer time of dispersion can re-agglomerate the CNFs which can hinder the potential effect of CNFs in cement mortar matrix.

Compared to plain UHP mortar, the addition of 0.005% CNFs by wt. resulted in an improvement of compressive strength of ultra-high performance mortar after 7 days of steam curing. The compressive strength is 184 MPa, about 8% higher than control mortar and approximately 4-8% higher than 0.01% and 0.015% CNFs mortars, respectively. However, increasing the volume fraction percentage of CNFs tends to lower the compressive strength. This can be due to reactions between the hydroxyl and carboxyl groups in cellulose molecules with Ca²⁺ that can delay the induction period of hydration and setting time, also the porosity and agglomeration of CNFs that weaken the bonding interfaces and promoted stress concentration.

Based on load-CMOD curves, UHP mortar reinforced with 0.005% CNFs is most effective and enhances the energy absorption capacity up to 14.42 kJ/m³ with flexural strength at peak load is 14.44 MPa (36% higher than control UHP mortar). This result supports the analysis on toughness index of UHP mortar containing CNFs mortar. The toughness index of UHP mortar containing CNFs (0.005% and 0.01% by wt.) is higher than UHP mortar with low-heat cement only. The results indicate the well-post crack behavior of CNFs mortars in comparison with control

cement and conclude that even a low volume fraction i.e 0.005% of CNFs is very sufficient in increasing the ductility of ultra-high performance mortar.

SEM analysis showed the phenomenon of bridging effect of CNFs in UHP mortar. However, it is difficult to identify the significant bridging effect of CNFs due to the short length of CNFs. Extensive research on UHP mortar reinforced with a combination between cellulose micro- and nano fibers can be considerably established.

NOMENCLATURE

g	energy absorption capacity	kJ/m^3
σ	stress	MPa
ϵ	strain	-
f	residual flexural strength	MPa
P	load	N
l	length of span	mm
b	width of specimen	mm
h	distance between the tip and the notch of specimen	mm

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