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Original citation & hyperlink:
Khorami, M, Ganjian, E, Saidani, M, Mortazavi, A, Olubanwo, A & Gand, A 2017, 'Utilisation of waste cardboard and Nano silica fume in the production of fibre cement board reinforced by glass fibres' Construction and Building Materials, vol. 152, pp. 746-755.
https://dx.doi.org/10.1016/j.conbuildmat.2017.07.061

DOI 10.1016/j.conbuildmat.2017.07.061
ISSN 0950-0618
ESSN 1879-0526

Publisher: Elsevier

Utilisation of waste cardboard and Nano silica fume in the production of fibre cement board reinforced by glass fibres

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Highlight (each bullet point maximum 85 characters)

- Kraft fibre extracted from waste cardboard can be used in cement board production.
- Incorporating glass fibres with Kraft fibre can increase flexural strength of cement board.
- An appropriate amount of Nano Silica Fume can improve properties of cement board.

Abstract (100 word limit)

Acronyms

Glass Fibres (GL), Kraft pulp Fibres (KF), Nano Silica Fume (NSF), Cement Composite Board (CCB)

Water Absorption (WA), Moisture Movement (MM), Flexural Strength (FS) and Scanning Electron Microscopy (SEM)

In this research, Glass Fibres (GL), Kraft pulp Fibres (KF) and Nano Silica Fume (NSF) were used to produce Cement Composite Board (CCB).

Fifteen groups of mix proportions were produced to investigate the effects of fibre content and size along with the effect of NSF on interaction, bonding and mechanical properties of CCB. Density, Water Absorption, Moisture Movement, Flexural Strength test and Scanning Electron Microscopy observations were conducted. The results showed that some mixes could meet the standard requirements and also the inclusion of GL and NSF into the mix containing KF (extracted from waste cardboard) could enhance the characteristics of CCB.

Keywords: cement composite, Cement Board, Glass fibre, Nano Silica Fume, waste cardboard

1 Introduction

Cementitious composites are typically characterised as being brittle with low tensile strength and strain capacities. Fibres are introduced into the matrix to overcome these weaknesses [1,2]. The effectiveness of the fibre reinforcement depends on a number of factors, such as mix preparation process, size, type, geometry, volume and dispersion models of fibres [3,4].

Reinforcement fibres not only improve bearing capacity of CCB but also increase fracture toughness of specimens by decreasing the concentrated applied stresses acting on the tip of cracks [5,6].

Typical CCBs consist of two or more types of fibres. Some fibres are used to enhance the production process, other may have an important role in increasing the bearing capacity and fracture toughness [3-5]. Thus, the combination of fibres and materials can offer more benefits than the individual entities.

Different types of fibres from natural and synthetic sources have been investigated for their suitability in CCB. Cellulose fibres have increasing popularity over the past two decades, due to their renewability, accessibility, low-cost production process, mechanical property, and compatibility with hydrated cement media [1,6-8].

The interfacial bond between fibres and hydrated cementitious material has an important role in increasing the bearing capacity of CCB. It is essential to use appropriate fibres and accurate proportion of materials in the mix design to achieve the best efficiency of the product. For example, steel fibres have high tensile strength but are weak in interfacial bonding with hydrated cement products. So, they pull out from the matrix in the first stages of loading before reaching their maximum tensile strength. Several methods have been suggested to increase the interfacial bond such as applying additives, fibre treatment and decreasing the fibre-cement gaps using the vacuum/compressive pressure in the production of CCBs [3,6,9,10].

Concerns have been raised regarding the use of CCBs in roofing and cladding applications, mainly due to long-term exposure to aggressive environments. The studies showed that CCBs containing cellulose fibres solely are more sensitive to aggressive ambient conditions and may undergo a reduction in flexural strength and fracture toughness over time. This is associated with a decrease in bearing capacity of CCBs, due to a combination of deterioration of cellulose fibres in the high alkaline environment, fibre mineralisation and volume increasing due to their high water absorption. The high alkaline matrix could easily decompose the lignin and hemicellulose phase that linked individual filaments and resulting in an inhibitory effect on hydration of cement and weakness in fibre structure. In addition, cellulose volume variation could create cracks in the interfacial zone leading to a decrease in fibre pull out strength [1,5,11,12].

To provide a less aggressive environment for cellulose fibres, the concentration of hydroxyl in the pore solution needs to be reduced. This could be done by introducing Sulpho Aluminate, Metakaolin or micro silica into the mix [13,14]. The addition of pozzolans such as Nano Silica Fume (NSF) in the cement composite reinforced by natural and polymeric fibres could increase the flexural behaviour of samples [3,11,15].

Previous research carried out by the first and second author has shown that the negative effects of lignocellulosic particles cement interaction can be controlled by NSF to enhance durability and flexural strength of CCB [3,5].

Following concerns relating to asbestos hazard on human health, in the early 1970s, a global movement was established to remove asbestos fibres from a wide range of products such as asbestos-cement board. One of the most important synthetic fibres used as a replacement for asbestos fibres in cement board production is PVA (Poly Vinyl Alcohol) which can be relatively expensive. It should be taken into account that besides monopolising PVA production technology by several companies, difficulty in accessibility and the high cost of PVA are other adverse factors to using those fibres in some developing countries [1,4,16].

In this research, an attempt has been made to investigate the feasibility of producing CCB using a combination of GLs, KFs and NSF, which are relatively cost effective and also readily accessible in most countries.

GL has a number of advantageous such as low cost, high tensile strength, and high chemical resistance. It has already been used in cement mortar and demonstrated to have significantly improved the tensile strength and ductility characteristics.

The use of normal type GLs incorporated to CCB has shown poor durability due to the following;

i) Hydroxyl ions resulted from cement hydration can cause corrosion on the fibres.

ii) Precipitation of calcium hydroxide within the GLs, can change the microstructure of fibres from flexible to rigid. This may change the mode of failure from fibre pull out to fibre fracture.

iii) Densify the interfacial zone decrease fibres compliance, consequently the non-uniform tensile stress induced by flexural loading would disrupt fibre-bridging effect in cracks.

To overcome those above-mentioned problems, it has been suggested to apply "alkali resistant GLs" instead of normal GLs in production of CCB [4,17]. In other words, only alkali resistant GL should be used in cement composite, otherwise, due to chemical corrosion of normal GLs in alkaline media, CCBs can become brittle and weaker through the cement hydration process [18,19]. Currently, alkali resistant GL which is highly resistant to alkalis, acids and corrosion have been developed for reinforcing of cement composite.

The use of Nano silica fume in CCB has already been investigated by authors [3] and the results showed that NSF could increase the flexural strength, bending strength and fracture toughness of CCB reinforced by cellulose fibres.

In this research, the characteristics of CCB reinforced by Kraft pulp fibres extracted from waste cardboard and alkali resistant GL incorporating NSF have been investigated.

2 Experimental Methodology

2.1 Materials

The materials used in this research include:

- Portland cement: Ordinary Portland cement Type I, satisfying the requirement of BS EN 197-1: 2000.

- Kraft pulp fibres extracted from waste cardboard and refined by using laboratory refiner equipment.

- Nano Silica Fume (Grade 999): particle size distribution, physical and chemical properties of Nano silica have been illustrated in Table1

Table 1: Physical and chemical properties of Nano silica fume.

Colour	White
Melting Point (^oC)	Approx. 1650
Solubility (Water)	Poorly water soluble
Specific Gravity (Water = 1.0)	2.25
Bulk Density (kg/m^3)	95-105
Specific Surface (m^2/g)	50-60
pH Value	3.6-4.5

- Glass Fibres: GL has a three-dimensional structure and is considered as isotropic materials comprises of a long network of oxygen, silicon and other atoms arranged in a random fashion. The GLs provided for this research have the non-crystalline structure, that is, amorphous, with no distinct shape and classed as alkali resistant glass fibres. They were used in 3mm and 6 mm lengths. The properties of Kraft fibres extracted from waste cardboard and GL are given in Table 2.

Table 2: Physical properties of the fibres.

Glass fibres	
Fibre Length (mm)	3 & 6
Filament Diameter (µm)	14-16
Size Content (%) – *ISO 1887: 1980*	1.0
Moisture (%) – ISO 3344: 1977	0.35 max.
Modulus of Elasticity (GPa)	70-80
High dispersion (filaments per kg)	220 * 106
Kraft fibres	
*Average length (mm)	1.1
**Width (Micron)	20-35
Specific gravity (measured by helium pycnometer)	1.5
Tensile strength	Not specified
Freeness	580

* Average length obtained from 50 fibres.

**Pulp fibres have ribbon shapes (not cylindrical shapes) with 20-35 micron width.

It should be noted that due to the short length of fibres, measuring the tensile strength was not possible with existing equipment.

The freeness of pulp is designed to give a measure of the rate at which a dilute suspension of pulp (3 g of pulp in 1 L of water) may be drained. The freeness, or the drainage time of pulp is

related to the surface conditions and swelling of the fibres. Drainage of unrefined pulp, which is measured as freeness, can give an indication of:

- The fibre length. In which, the long fibres have more freeness compared to short fibres.
- Damage to fibres during pulping, bleaching or drying. In which, short fibres or fines that are produced during the pulping operation reduce pulp freeness.
- The refining energy required to achieve certain slowness during stock preparation.

In this study, the freeness test was carried out according to AS/NZS 1301.206s:2002 standard. Freeness is commonly called Canadian Standard Freeness (CSF) because it has been based on the test developed by the Canadian Pulp and Paper Research Institute. For the current study, CSF measured for KFs was around 560-600.

2.2 Production of the specimens

For each mix code, the slurry contains water, cementitious materials and fibres with a high water/cement ratio (i.e. around 3 by the weight of cement) was prepared in a mixer.

Then it is poured into a mould and subjected to vacuum and compressive stress of 7 kN/m^2 to form a flat sheet. After the slurry dewatering process, pad (dimensions 180 × 82 × 7 mm) was demoulded and cured for 21 days at 95% of relative humidity and air cured in the laboratory for 7 days at a temperature of 20 ± 2 °C. The cured specimens were subsequently tested.

2.3 Tests

The following subsections outline the tests undertaken in accordance with 1) BS EN 494:2004 +A3:2007 (Fibre-cement profiled sheets and fittings – Product specification and test methods) and 2) BS EN 12467:2004 (Fibre-cement flat sheets – Product specification and test methods).

2.3.1 Density

To measure the density of the specimens, a routine laboratory method was used. Initially the mass of the specimens placed in laboratory environment (20±2 °C and 40%-50% RH) for one day was measured. Then to find the volume of the specimens, they were submerged into the water and the displaced water was measured. The result of mass divided by volume was considered as density of the specimens.

2.3.2 Water Absorption

Water absorption was conducted in accordance with ASTM C1186-08 (2012) with reference to ASTM C1185-08 (2012). In this test, to meet the standard requirement, the specimens are dried in an oven at 90 ±2 °C until constant weight was achieved, then allowed to cool to room temperature and are weighted. The specimen is then submerged in potable water at 20 ± 4°C for 48 ± 8 hours. Then the specimen is removed and excess water wiped from the surfaces using a damp cloth and weighed again. The weight increased due to submerging the specimen in water is expressed in percentage as the water absorption.

2.3.3 Moisture Movement

Moisture movement was conducted in accordance with ASTM C1186-08 (2012) with reference to ASTM C1185-08 (2012). In this test, the linear deviation in length of the specimen due to moisture absorbed from the surrounding environment is determined. To find the moisture movement according to the standard procedure, the difference between the length of the

specimen at Relative Humidity (RH) of 90% and 30%, needs to be divided by the length at 30% RH and the result is expressed in percentage.

2.3.4 Flexural strength

Flexural behaviour of the specimens under a three-point load system according to BS EN12467:2004. The specimens were subjected to three-point loading flexural test to failure point which must occur between 30 and 60 seconds of loading time.

2.3.5 SEM

The JEOL JSM-6060 LV SEM machine was used for this study. The samples were mounted on stubs and were gold coated using the sputter coater to enhance conductivity. The samples were then placed in the vacuum chamber of the SEM machine and images were captured with different magnifications for the selected specimens. To obtain improved views of the fibres, some images were captured after tilting the samples 75 degrees in relation to the horizontal plane. In the observation, microstructure of fibres, fibre–cement interfacial areas and fibre bonding were studied.

2.4 Fibre preparation and mix design

The waste cardboard was shredded into strips 50×5 mm in size. They were then soaked in water and rinsed periodically for two days. The saturated pieces of cardboard were then refined using a laboratory refiner until a uniform Kraft pulp fibres were obtained.

The mix design plan is illustrated in Table 3. A broad range of fibre content, fibre size and NSF content has been investigated in this research. As shown, fifteen groups of mixes were developed to investigate;

1. The effect of the size (3 mm and 6 mm) and fibre content (1%, 2% and 3% by the weight of the cementitious materials).
2. The effect of NSF content (1%, 2% and 3%) as a replacement for cement on the specimens reinforced by 3 mm and 6 mm GLs.
3. The effect of the combination of 3 mm and 6 mm GL with and without NSF.

In all mixes, the amount of water was 300 grams which leads to water cementitious materials ratio of 3 for making the slurry. In Table 3, the amount of Kraft pulp fibres extracted from waste materials is kept constant (i.e. 4 grams) for all mixes which is 4% by the weight of cementitious materials. This amount of KF has already been studied by the authors as an optimum quantity when in combined with polymeric fibres [2,16].

In Table 3, the following symbols are defined:

K: Kraft pulp fibres extracted from waste cardboard GL: Glass fibres (alkali resistant type)

N: Nano silica fume

Table 3: Details of mix proportions for groups of mix design.

Mix	**Mix Code**	**N (g)**	**Cement (g)**	**GL 3-mm (g)**	**GL 6-mm (g)**
1	K4 (Control)	0	100	0	0
2	K4-GL1-3mm	0	100	1	0

3	K4-GL2-3mm	0	100	2	0
4	K4-GL3-3mm	0	100	3	0
5	K4-GL1-6mm	0	100	0	1
6	K4-GL2-6mm	0	100	0	2
7	K4-GL3-6mm	0	100	0	3
8	K4-GL2-3mm-N1	1	99	1	0
9	K4-GL2-3mm-N2	2	98	2	0
10	K4-GL2-3mm-N3	3	97	3	0
11	K4-GL1-6mm-N1	1	99	0	1
12	K4-GL1-6mm-N2	2	98	0	2
13	K4-GL1-6mm-N3	3	97	0	3
14	K4-GL1-3mm-GL1-6mm	0	100	1	1
15	K4-GL1-3mm-GL1-6mm-N2	2	98	1	1

The number after the symbol shows the percentage of the materials by the cement weight. For example, K4-GL1-3mm-GL1-6mm-N2 means all the specimens of this group contain 4% Kraft pulp fibre, 1% glass fibre of 3 mm length, 1% glass fibre of 6 mm length and 2% by weight Nano silica fume.

Six replicate specimens were manufactured for each group, allowing multiple tests to be done and averages taken. The first row of Table 3 (i.e. K4) belongs to the control mix that was produced only by Kraft pulp fibres, cement and water.

It should be noted that during the manufacturing procedures when the slurry was being mixed, it was observed that the specimens containing GL greater than 3% fibre content, the fibres were clustered and clumped hence disrupts the uniformity of fibres throughout the matrix. For this reason, the highest fibre content that was introduced during the slurry mixing was limited to 3% for glass fibres, as shown in Table 3.

The mix design shown in Table 3 is a part of the larger table including more than thirty mixes which have been made and tested in this research but after flexural testing of the specimens, it was revealed that flexural strength for many of mixes was less than 6 MPa. So, in this paper, only the results of some mixes which had a flexural strength greater than 6 MPa along with control group are analysed and discussed. For instance, the primary results showed that when the length of GL is 3 mm, the optimum fibre content to gain the highest flexural strength is 2% while for 6 mm length, the corresponding value is 1%. This will be discussed later in other sections. For this reason in Table 3, the effect of NSF on improving the flexural strength of the specimens reinforced by 2% and 1% of 3 mm and 6 mm GL, respectively was chosen to be analysed in this paper.

3 Results, Analysis and Discussion

Results have been obtained for density, water absorption, moisture movement, flexural behaviour and SEM based on the relevant standards. To show the test precision, error bars are shown on all bar charts (i.e Fig.1 to 4).

3.1 Density

Fig.1 illustrates the average density of the specimens of each group at age 28-day.

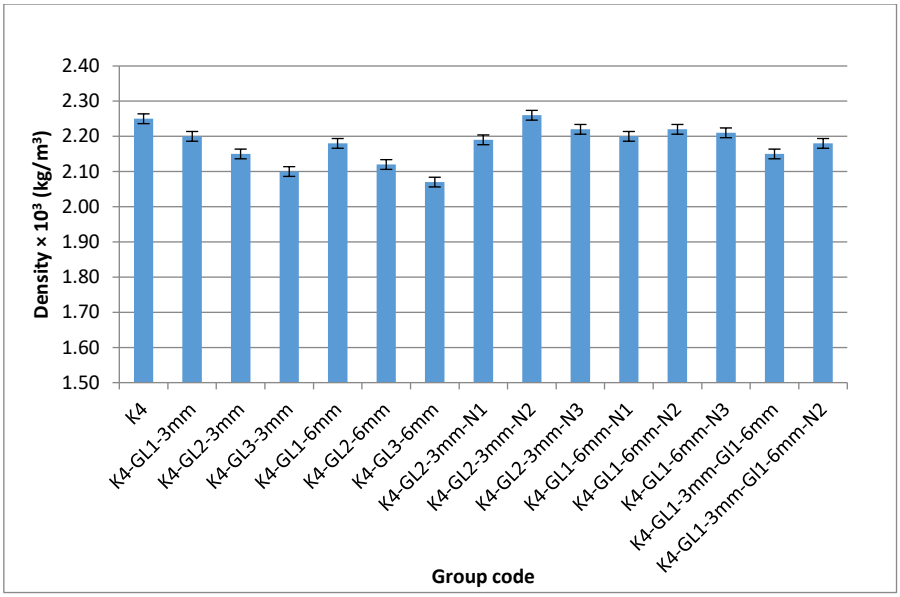

Fig.1: Average density of the specimens in each group.

As observed when GL is added to the mix, the density is relatively reduced comparing to the control group (K4). This may be associated with hydrophobic nature of the GLs. Whereas the GLs have little or no affinity for water, they cannot absorb water like Kraft pulp fibres which have hydrophilic nature.

Accurate observation of the specimens revealed that GLs are visible on the surface of the specimens reinforced by 3% GLs. This showed that incorporation of 3% GLs into the mix is too high to be embedded properly in the mix resulting in non-uniform dispersion GLs into the mix and higher concentrations being present at the surface that leads to further reduction in density.

As seen, embedding NSF into the mix causes an increase in density for all groups reinforced by 3 mm and 6 mm GL's. NSF comprises of amorphous (non-crystalline) silicon dioxide (SiO2),

with very small discrete particles. Large surface area due to very small size particles along with high SiO2 content makes NSF as a reactive pozzolan which enhances hydration kinetic by increasing the amount of beneficial cement hydration products. NSF reacts with calcium hydroxide crystals formed from the hydration of calcium silicates increasing C–S–H nucleation which leads to the denser microstructure of the matrix. It has been proved that NSF particles act as an activator to promote pozzolanic reactions and filler, thus using the appropriate proportion of NSF can fill the voids containing air and moisture resulting further density for the matrix.

As presented in Fig.1, in spite of initial prediction, with increasing the NSF content from 2% to 3%, density of the specimens decreases. This may associate with the optimum percentage of NSF. If NSF is used more than 2%, it could have different effects on the matrix, including; 1) the amount of small particles needed for filling the voids would be more than enough hence it may disrupt the appropriate grading in particle size distribution to reach the highest density. 2) The amount of NSF required for consuming the calcium hydroxide is more than enough hence the extra NSF will not participate in chemical reactions or filling the voids, so the density will not increase.

In this research, variations of density for CCB has been investigated to provide an additional and complementary parameter to determine any links between the density and other observations.

3.2 Water Absorption

Water Absorption (WA) is considered as a key feature for the long life durability of CCBs. Most applications of CCBs require as little WA as possible because it affects the rate of degradation severely.

The average WA resulted from each group is presented in Fig. 2.

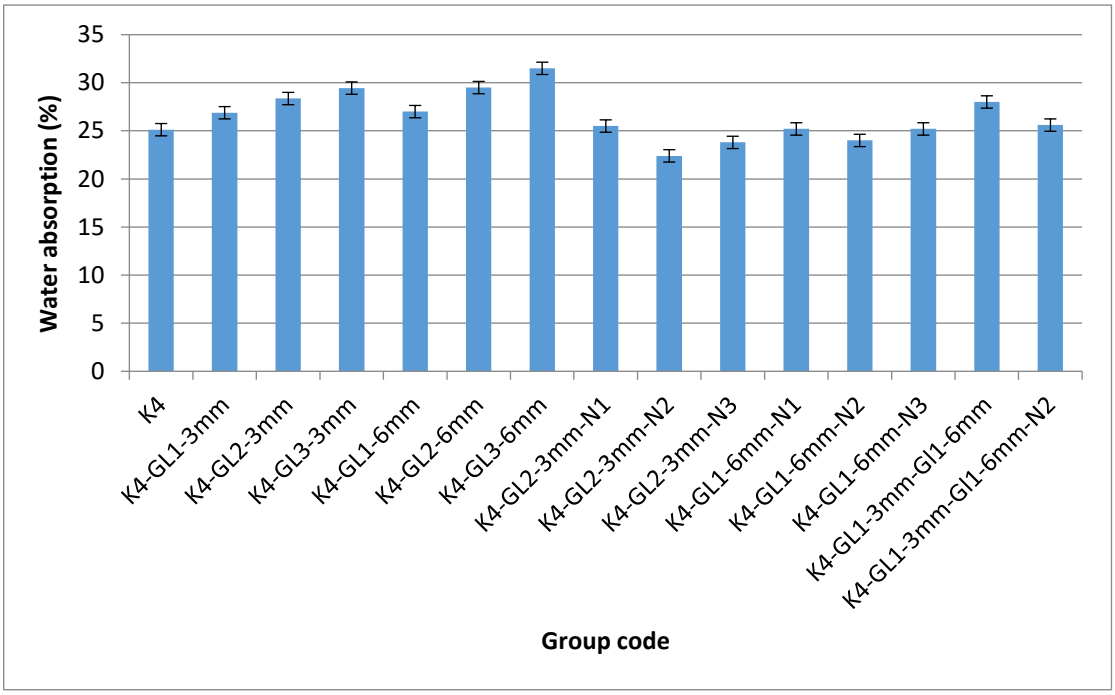

Fig.2: The results of average water absorption of the specimens in each group.

As illustrated in Fig. 2, with increasing the fibre content (3 mm or 6 mm lengths) into the mix WA increases. With the addition of GLs, the amount of pores within the specimen increases resulting in more space to enter the water to the specimen. It should be noted that the mechanism of moisture absorption is different with water absorption in CCB. Moisture absorption is largely based on capillary action in which the humidity and moisture of the environment could be sucked into the specimens through tiny pores while in water absorption, the molecules of water enter and penetrate into the larger voids due to the hydrostatic pressure of water, rather than sucked by smaller capillaries. In other words, water absorption could be more susceptible due to water pressure rather than absorbing water due to capillary action.

As presented in Fig. 2, the specimens containing 6 mm GL have a slightly greater affinity for water absorption in comparison to the specimens composed of 3 mm fibres. This could be associated to this fact that the larger 6 mm fibres could create larger pores resulting further porosity.

To investigate the effects of NSF on the characteristics of CCB both “fresh” and “hardened” cement composite should be studied considering the following:

1- NSF could accelerate the hydration of tricalcium silicate (C3S) and C-S-H gel formation. It also aids to remove the non-hydrogen bond OH groups [3,15].

2- NSF has important effects on the air content in fresh state and on the porosity in the hardened state of the cement composite. Incorporating NSF into the mix, the air content of fresh cement composite increases. This phenomenon affects the viscosity of mix so that with increasing NSF, the viscosity of mix significantly increases because the fine particles of NSF decrease or stop escaping the entrapped air from the fresh mix. In hardened cement composite, NSF decreases the porosity because the pores (i.e. entrapped air) have been surrounded and disjointed by NSF. This finding is consistent with other carried out research [20,21].

3- Each cement composite mix has a specific capacity of NSF to reach the minimum porosity. As outlined, only some of NSF reacts with hydrated cement products and the rest fills the pores. By filling the pores, both within hydrated cement products and the fibre-cement interfacial zone are filled, the grading of all mixed content is improved and hence the porosity decreases. If the amount of NSF is more than enough for filling the gaps, the extra NSF particles disrupt the grading of mix content resulting in porosity rising.

Considering those aforementioned effects, an optimal amount of NSF could provide the lowest porosity for the hardened composite [19,21,22].

As seen in Fig. 2, two different tendencies due to incorporating NSF can be observed in hardened cement composite; increasing the small amount of NSF (i.e. 1%) leads to a decrease in the porosity while the high amount of NSF (i.e. 3%) leads an increase in the porosity. As observed, 2% NSF could be considered as a critical value (optimum proportion) resulting in minimum porosity and minimum water absorption. Referring back to Fig.1, this statement is consistent with the observation that the highest density belongs to the group composed of 2% NSF.

3.3 Moisture Movement

This test is used to check the appropriateness of the CCB that are exposed to moisture and high humidity. The average moisture movement for all groups is shown in Fig. 3.

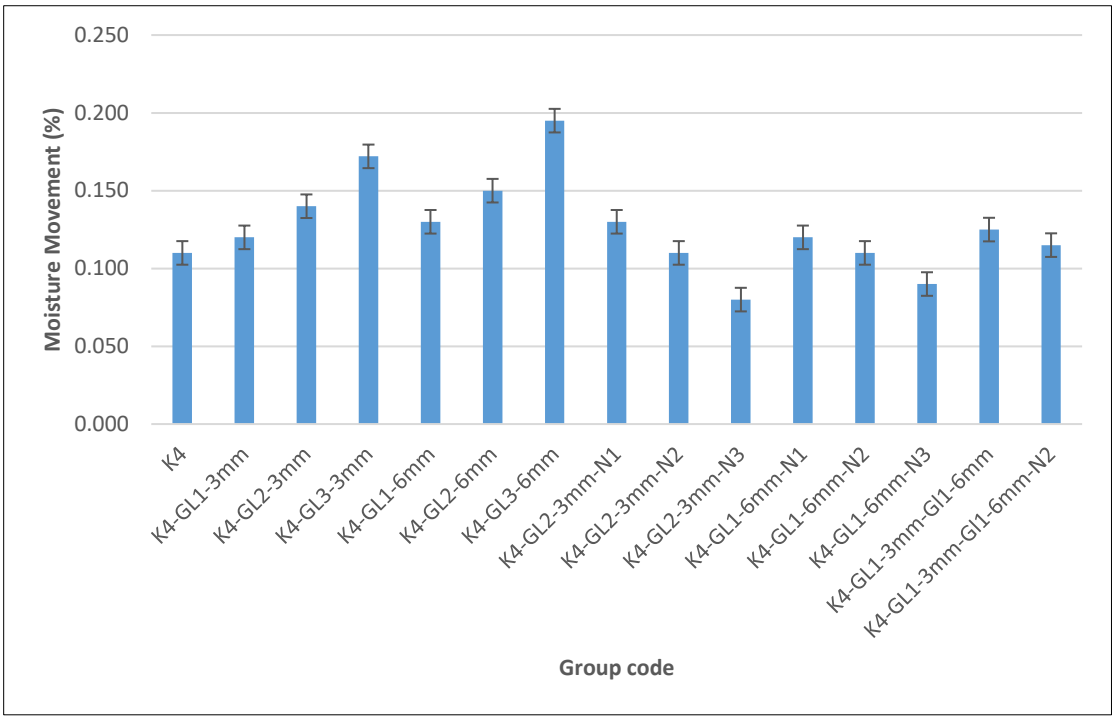

Fig.3: The results of moisture movement for all groups.

Fig. 3 shows that the amount of moisture movement for all groups is less than 0.20% which is the extremely small amount. CCB is a porous cementitious material containing cellulose fibres (Kraft pulp) that can absorb moisture and thus increase in size. This could be important for CCB depends on application purpose and should be in the permitted tolerance range. For most of the applications such as cladding and external walls, all groups fall in the permitted range of tolerances.

The inclusion of varying proportions and lengths of hydrophobic GLs in the mix can reduce the moisture movement for all groups compared to control group (K4). However, with increasing GL content, the moisture movement increases too. As outlined in water absorption, GL can increased porosity either in the form of small capillaries or creation of larger voids within the specimen. So, when the moisture is drawn in due to any reason such as capillary action, the

Kraft cellulose fibres absorb the moisture and swell leading to an increase in the size of the specimen.

The effect of NSL on moisture movement is consistent with the results already outlined in water absorption and density sections with minor differences. As NSF is introduced into the mix, the density and water absorption are affected but both of them show an optimum of 2% NSF to achieve the highest density and the lowest water absorption. As depicted in Fig. 3 with increasing NSF, MM decreases continuously. This may be attributed to the capillary action in which with increasing NSF, the capillary pores that could be considered as pathways for tiny molecules of humidity, are blocked by NSF, consequently, MM decreases. Similar behaviour has already been reported by other researchers [21,22]. The fewer voids constrict the fibres and inhibit their ability to absorb water. The least amount of moisture movement that can be achieved is generally considered as a significant advantage for CCB.

The mixes containing 3% of 6 mm GLs demonstrating higher water absorption rather than 3 mm GLs due to further porosity resulting from “balling phenomenon” which is described in the next section.

3.4 Flexural testing

Flexural test is the most important mechanical properties of CCBs which has been pointed by all relevant standards. The results of the flexural test on the specimens after 28 days curing are shown in Fig. 4.

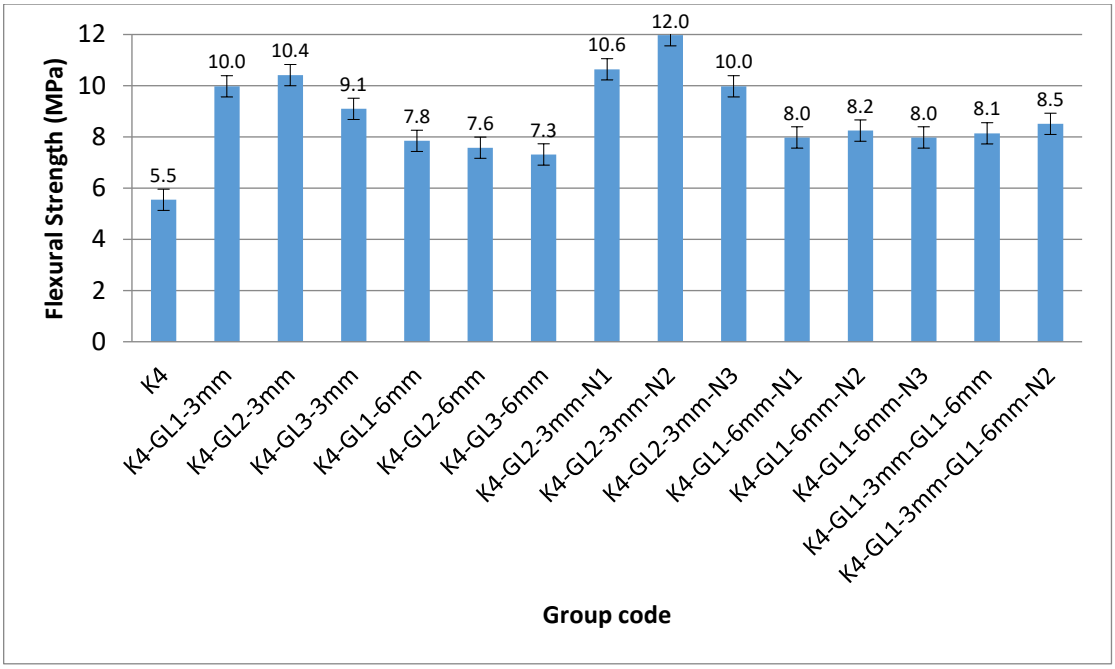

Fig.4: The average flexural strength of the specimens for each group.

The last experiment conducted on the specimens was the flexural test. It is believed that the specimens may have been interfered by the tests conducted previously (i.e. WA and MM). This could demonstrate the flexural strength of the specimens in the worst case by simulating the real environmental conditions such as cycles of high-low humidity and wet-dry exposure experienced by the specimens.

Fig. 4 shows;

- The incorporation of GLs (3 mm or 6 mm) into the mix increases the flexural strength in all groups comparing to control mix (K4). This is due to mechanical characteristics of GL such as tensile strength and modulus of elasticity which could improve bearing capacity of CCB.
- The effect of 3 mm length GLs in enhancing flexural strength is greater than 6 mm.
- The maximum flexural strength of the specimens reinforced by 6 mm length GLs solely belongs to 1% fibre content which is considerably lower than 3 mm. The dominant

reason to decrease the flexural strength is associated with balling (clumping) phenomenon; When the percentage of 6 mm length of GLs in the mix increases more than optimum, the fibres cannot disperse uniformly throughout the mix and clump together in the form of ball shape, forming bundles of accumulated individual fibres with low specific contact area, resulting in weak points in matrix. This phenomenon disrupts the crack-bridging effect to stop cracks, whereas the initiation of those cracks is made more likely by the greater extension of unreinforced areas found in the specimens. This will be discussed later in microstructure section.

- As seen, when the length of GL is 3 mm, the optimum fibre content to gain the highest flexural strength is 2% while for 6mm length, the corresponding value is 1%. For this reason, the effect of NSF on improving the flexural strength of the specimens was studied only on 2% fibre content for 3 mm GL and 1% fibre content for 6mm length.

- The NSF has a positive effect on rising the flexural strength of the specimens reinforced by GLs. The optimum amount of 2% NSF led to the maximum flexural strength of 12 MPa for group K4-GL2-3mm-N2. This is due to bonding improvement within fibre-cement interfacial zone, decreasing porosity and uniform dispersion of fibres into the mix. This will be illustrated by microstructural studies of the specimens in the next section.

- The simultaneous use of 3 mm and 6 mm length GLs couldn’t improve the flexural strength considerably comparing to the specimens reinforced by 3 mm or 6 mm solely.

To study the flexural behaviour of the specimens during the loading procedure Figs. 5 to 7 illustrate the effects of the percentage (1%, 2% and 3%) of GL content, length (3 mm and 6 mm) of GL and NSF content (1%, 2% and 3%).

Fig. 5 compares the mid-span deflection against the flexural stress of the specimens reinforced by different percentage of 3 mm length of GLs.

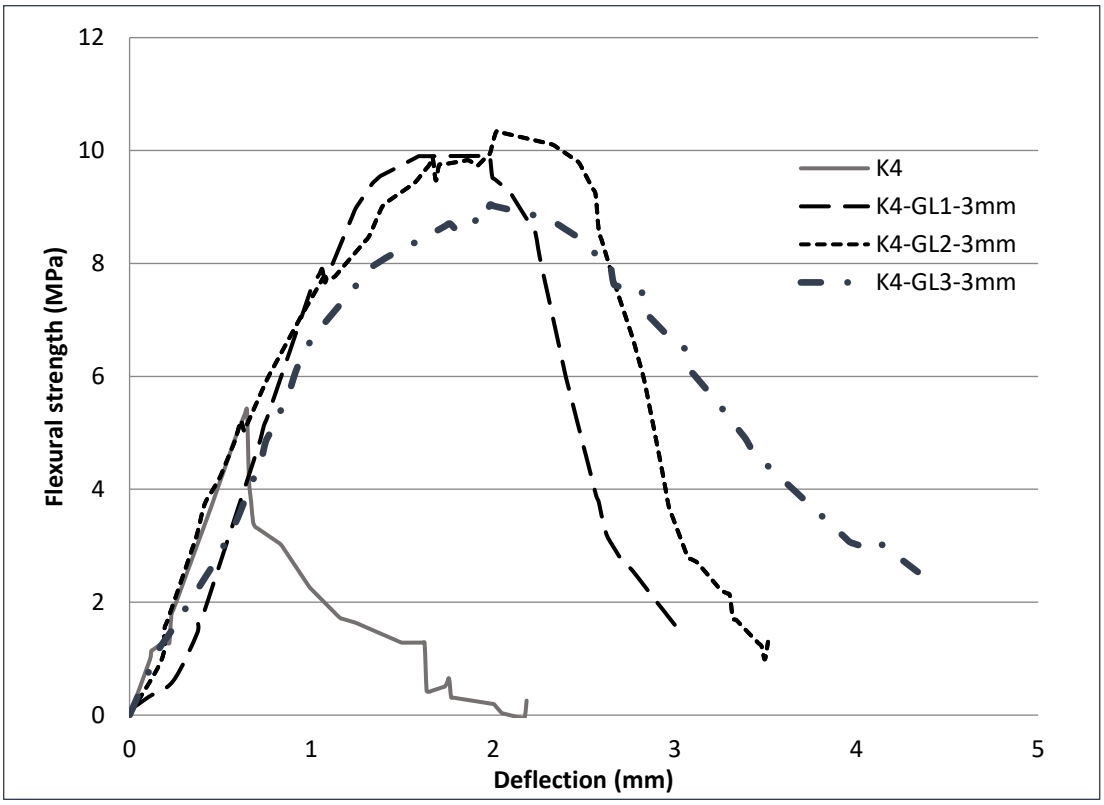

Fig.5: The effect of fibre content (1%, 2% and 3%) of GL-3mm on flexural behaviour.

As seen in Fig. 5, adding GL to the mix increases not only the area under the curve which is directly related to fracture toughness but also the flexural strength of the specimens. Although the energy absorption (which is identified by the area under the curve) for all groups reinforced by GL is approximately identical, the flexural strength of K4-GL2-3mm reaches to 10.4 MPa which is the greatest values in comparison to others. In other words, within the range of fibre content studied, the best proportion of GL for the specimens containing 4% of Kraft fibres is 2%, which provides the maximum of 10.4 MPa flexural strength. The mechanical

properties of GL, particularly, tensile strength plays an important role in bearing capacity of the specimens. This will be discussed later under microstructure study subsection.

As seen in fig.5, the plots for the specimens containing GLs indicate several steps or fluctuations (serrated line). This is associated with initiation of microcracks due to fibre pull out or fibre breaking. The applied stress is then redistributed and is taken by other fibres, allowing further increases in load bearing. In other words, when a micro-crack is arrested by fibre crack bridging mechanism, the stress at interfacial zone rises until either the fibre breaks or pull out, whichever reaches maximum bearing capacity earlier. Then by forming the cracks, the stress is released immediately (serrated line in the plots) and by redistribution of stress, new micro-cracks appear elsewhere. This mechanism continues until the micro-cracks join with each other, forming a visible crack. Previous research carried out by the first author showed that redistribution of stress after initial cracks could be observed in CCB reinforced by Kraft fibres and polymeric fibres [2,3,16]. The visible cracks are initiated at mid-span where maximum tensile stresses are initiated in the component fibres. This stress is called “Bend Over Point”. From this point, the stress keeps rising but with a different slope. As the crack width increases, the specimen elongation increases then the load is being taken by the fibres bridging the cracks until subsequent total failure occurs. In addition, the introduction of GLs within the mix could increase the ductility of the specimens in the threshold of failure about three times in comparison to the specimens wholly reinforced with KFs. This is also associated with the high tensile strength and longer anchorage length of GLs compared to KFs.

As outlined in Fig. 4, the highest flexural strength belongs to K4-GL2-3mm. So, this group was chosen to be investigated by inserting NSF in the matrix.

Fig. 6 presents the effect of various percentage of NSF on the flexural performance of the specimens reinforced by only 2% GL-3mm.

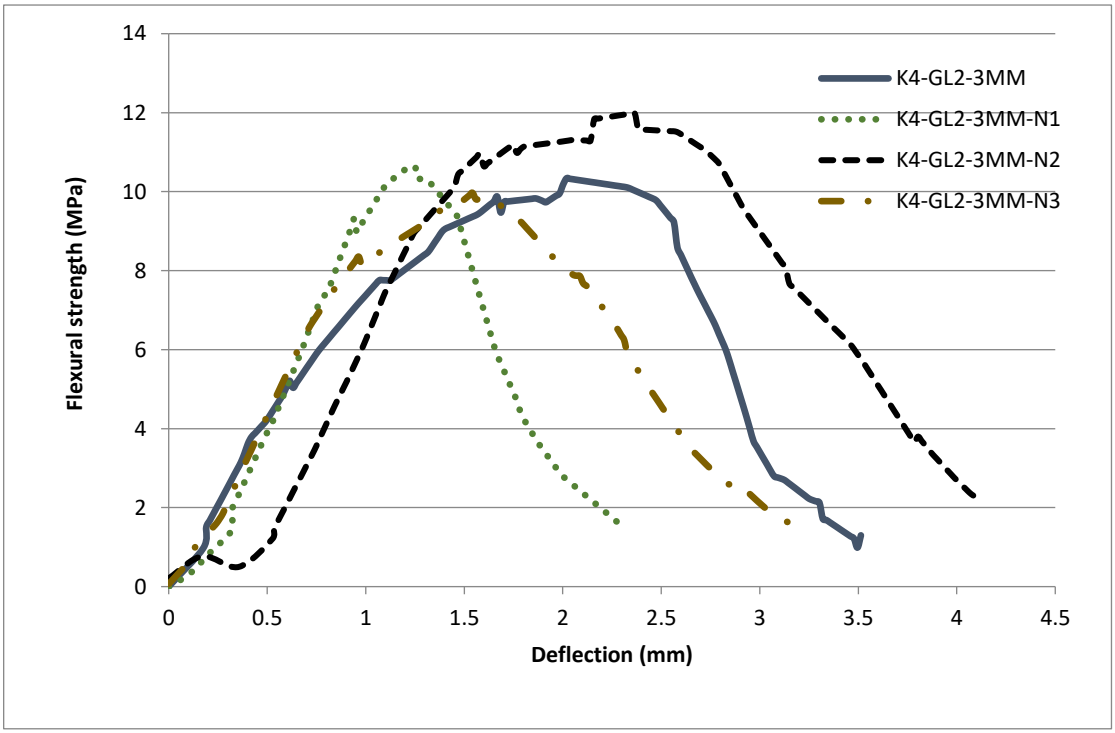

Fig.6: The effect of NSF on the flexural performance of the specimens reinforced by 2% GL-3mm.

As illustrated in Fig. 6, introduction of NSF to a mix containing 4% KF's and 2% GL's only offers positives strength characteristics (fracture toughness and flexural strength) when 2% NSF is used. This is associated with the optimum percentage of NSF. It seems that 1% NSF is not enough to fill the voids or acts as an activator to enhance the cement hydration process. Similarly, 3% NSF seems to be more than enough for the mix that can interfere with the cement. Excessive use of NSF results in poor-graded particle size distribution within the hydrated cement products, fillers and fibres, disrupts both the mechanical and physical

properties of CCB. As observed, 2% of NSF could meet the physical and mechanical requirements in terms of both filling the voids that lead to porosity reduction and activating calcium hydroxide to produce Calcium Silicate Hydrate (C-S-H). This statement is consistent with the results of density outlined in Fig1. In other words, the optimum percentage of NSF can enhance the grading of ingredients in terms of particle size distribution and pozzolanic activator to reach the highest density, lowest water absorption and appropriate moisture movement as outlined in the previous sections.

Fig. 7 illustrates the effects of GL fibre's length (i.e. 3 mm and 6 mm) on the flexural performance of CCBs.

As seen in Fig. 7, the flexural strength of specimens reinforced by longer GLs (i.e. 6 mm) are weaker than the specimens reinforced by 3 mm lengths of GLs. The maximum flexural strength experienced by the specimens reinforced by 3 mm GL's are 10.3 MPa and 9 MPa for 2 % and 3% fibre content respectively while the corresponding values for 6mm GL's are 8.9 MPa and 7.7 MPa respectively. As observed in Fig. 7, fracture toughness which relates to the area under the curve for the specimens reinforced by 3mm GL's are relatively larger than the counterpart specimens reinforced by 6 mm GLs. This behaviour is attributed to the interaction of the relatively longer fibres with each other resulting in "balling phenomenon" which in turn reduces the bearing capacity of the specimens.

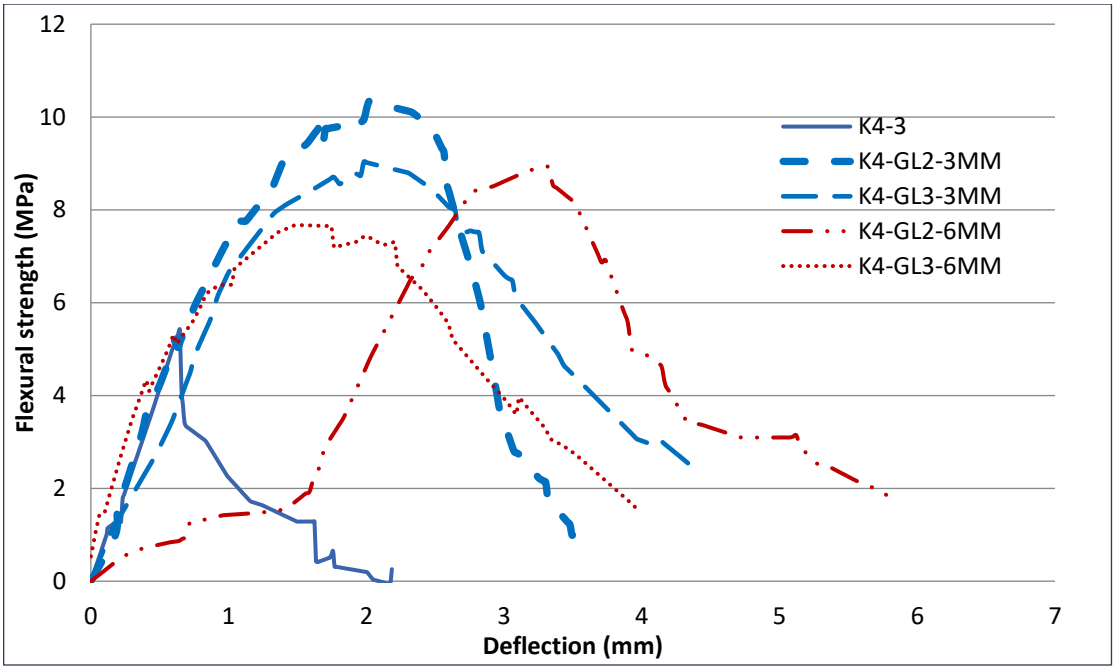

Fig.7: The effect of GL's length on flexural performance of the specimens.

Surrounding fibres by hydrated cement products could provide enough anchorage length for transferring the load by the bridge-cracking effect. In spite of this fact, during manufacturing process of the specimens, the long GLs (i.e. 6 mm) could not be distributed uniformly in the mix and clumped with each other or with cellulose fibres forming a ball shape twisted/tied fibres (i.e. balling phenomenon) that could be considered as the voids in the matrix. In other words, uniform fibre dispersion is essential in the matrix because the cracks initiate and advance from sections of a composite that has either fibre clumping or larger fibre free areas.

4 Microstructure study (SEM)

Microstructure studies of three samples including K4, K4-GL2-3mm and K4-Gl-3mm-N2 were carried out using SEM. The fibres and materials which have been focused on this study include; Kraft fibres, Glass fibres and Nano silica fume.

Fig. 8 indicates the fracture surface of KFs reinforced CCB in group K4, displaying Kraft fibres dispersion and the positions of some Kraft fibres after pulling out across the section. As seen, both types of failure including fibre breaking and fibre pull out are visible in Fig. 8. This confirms that there is a relatively strong bond in the interfacial zone within Kraft fibres and hydrated cement products. If the anchorage length of Kraft fibres falls in appropriate ranges, the mechanism of failure tends to pull out rather than breaking the fibres [1,16]. The voids shown in Fig. 8 have the same size as the fibres diameter confirming that the failure occurs due to a pull out mechanism. Apart from the voids created by pulling the fibres, there are other voids visible in Fig. 8 which have been formed in some part of unreinforced matrix due to partial compaction

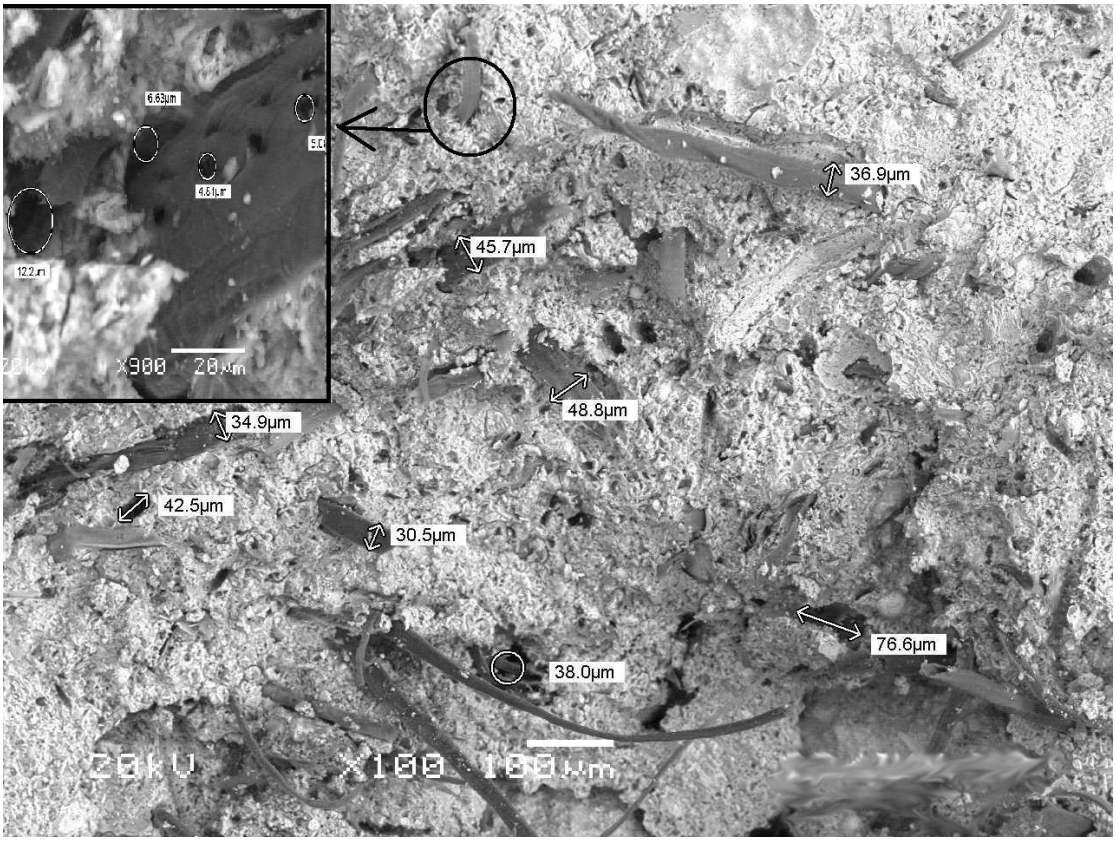

Fig.8: Interfacial zone within fibres and hydrated cement products in K4 (X100 & X900 magnifications).

As seen in Fig. 8, the entire section has been dominated by calcium silicate hydrate (C-S-H) which are connected and surrounded by numerous needle shaped hydrates [20,21]. C-S-H is considered as the principle binding product within hydrated cement product.

Fig. 9 depicts a cross section of sample K4-GL2-3mm in which the cylindrical GL's have been covered by the small amount of hydrated cement products (magnified at top left inset of Fig. 9) while ribbon shape KFs (magnified at the bottom right inset of Fig. 9) have been almost fully covered. Primarily, the most important cement hydrated products which are formed around GLs are ettringite needle shaped crystals that are formed from the reaction between calcium aluminate and calcium sulphate of cement. The formation of ettringite needle shaped crystals in the vicinity of polymeric fibres has already been reported by other researchers [4,7].

Several phenomena occur at early stages of hydration process. In addition to the plateau of the gel pore water intensity, the rate of consumption of capillary water is changed. As C-S-H grows, the capillary pores in the vicinity of GLs increase because of the hydrophobic property of GLs. The excess water that is not consumed in the hydration process will evaporate and leave the space within the matrix and thus increase the porosity. The porosity can cause a reduction in bearing capacity of the specimen and also increase water absorption as already discussed.

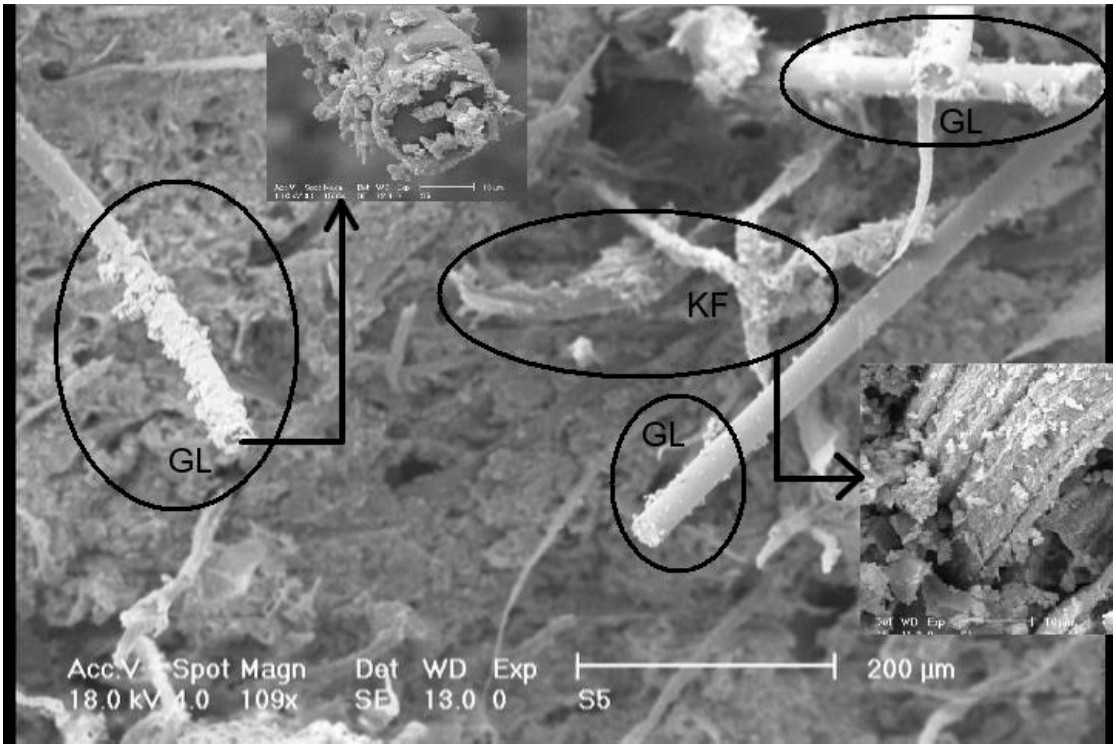

Fig.9: Hydrated cement products distribution on GL's and KF's (X100 and X1600 magnifications).

As illustrated in Fig. 9, fibres are distributed randomly through the cross section. Some fibres are protruding outwards from the composite and some of them lay lengthways along the fractured face of the composite. Obviously, all distributed fibres can contribute in controlling the shrinkage cracks while only some of them which are distributed on the longitudinal axis of CCB could contribute in the flexural bearing capacity of CCB.

As seen, in spite of the KFs, the lateral surface of the GLs is smooth and contains only some randomly distributed minute (i.e. 1.2 - 2µm) imperfections or debris. It seems that due to hydrophobic characteristics coupled with the smooth surface of GLs, the tendency of hydrated cement products for placing on the GLs is lower than KFs. Although signs of cement hydration products on the glass fibre are observable, interfacial bonding within GLs and hydrated cement cannot be as strong as KFs.

572 It is clear that GLs are not as hydrophilic (wettable) as the KFs. Wetting occurs due to
573 intermolecular interactions when the two molecules are brought together [4]. As seen in Fig.
574 9, although rough (irregular) surface and hydrophilic property of KFs could create a relatively
575 strong bonding in interfacial zone, the slight adhesive bonding within GLs and the hydrated
576 cement products are still observed.

577 Generally, two types of bonding occur simultaneously at the fibre-cement interfacial zone
578 described as, a) Mechanical interlocking resulted from friction within fibres and hydrated
579 cement products and b) Chemical bonding which largely includes strong covalent O-H bonds
580 and weaker Hydrogen bond.

581 It should be noted that polymeric fibres with hydrophobic properties cannot establish any
582 strong chemical bonding with hydrated cement products unless the structure of the polymeric
583 fibres has metal ions to form an ionic bond in the interfacial zone.

584 Since the mechanical characteristics of glass fibres in terms of elasticity modulus and tensile
585 strength are substantially greater than KFs, it can increase flexural strength and fracture
586 toughness of CCBs when an appropriate proportion of GLs is embedded into the mix. It should
587 be noted that depending on the anchorage length, both fibre-breaking and pull out
588 mechanisms have been observed in KFs while the high tensile strength of GLs justifies fibre
589 pull out predominance.

590 Fibres have an important role in transferring the load. As cracks progress, while the fibres
591 carrying the maximum stress, fibre pull out is being occurred gradually. During this loading
592 procedure, the main factor that ultimately controls this compromise is interfacial bond, not
593 the properties of individual materials working alone [1,4,5].

594 Both Figs. 8 and 9, illustrate that the intensity of porosity in the fibre-cement interfacial zone
595 is greater than other zones. This porosity could be considered as tiny cracks in the vicinity of

fibres and could be attributed to fibre properties specifically water absorption. Water absorption is one of the intrinsic properties of Kraft pulp fibres which causes considerable volumetric changes in the structure of the cellulose fibres. This increases the water-cement ratio in the interfacial zone which has a negative effect on fibre-cement bonding due to differential drying shrinkage of the matrix. As a consequence, it can generate the cracks and cause a reduction in flexural strength.

As already mentioned, in this research, to reduce porosity in the vicinity of the fibres, NSF was used as shown in Fig. 10.

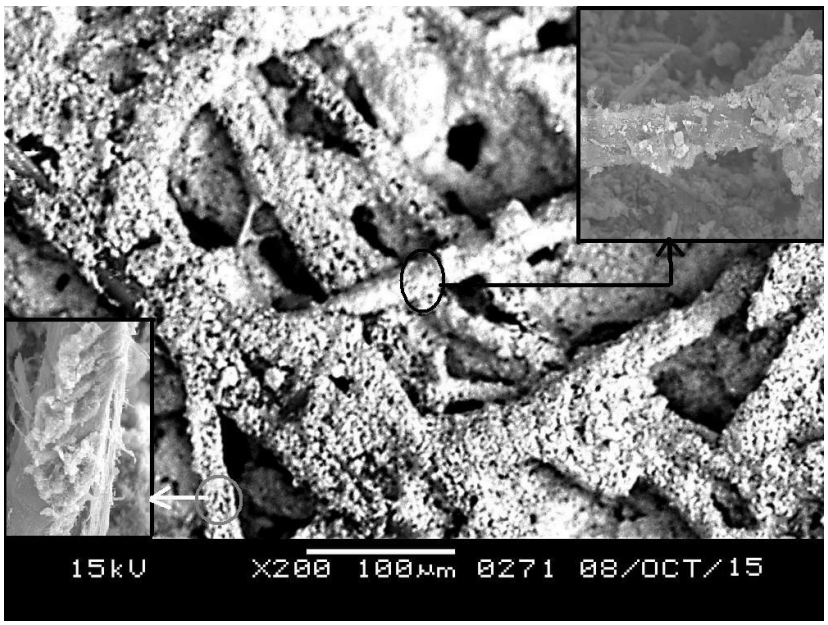

Fig.10. Effect of NSF on the microstructure of CCBs containing KF's and GL's. (x200 and x1000 magnifications).

As seen in Fig. 10, both pozzolanic reactions and filler action cause GLs (highlighted at the right side of Fig. 10) and KFs (left side of Fig. 10) are covered by hydrated cement products particularly Calcium Silicate Hydrate (C-S-H).

611 Pozzolanic reactions of NSF ensures that calcium hydroxide (CH, also called “portlandite”
612 which is considered as less-beneficial hydration products) is consumed rapidly to increase C–
613 S–H nucleation which has the main role in providing the strength in a cement matrix. The
614 reduction of CH could also improve the durability of cellulose fibres in alkaline media [3].

615 Pozzolanic reaction of NSF is also caused a reduction in the Ca/(Si + Al) ratio which can improve
616 hydration products. It has been demonstrated that less than one-third of the calcium
617 hydroxide (portlandite) reacts with NSF to form C-S-H. The rest of the calcium comes from the
618 C-S-H that has already been produced in the early stages of hydration process by the reaction
619 of Alite (C_3S) before NSF promote pozzolanic reactions [20].

620 Muller *et al*. [20] showed that NSF has an important effect on reducing the porosity by
621 consuming the water in both interlayer and gel pore spaces of C–S–H. C–S–H can be classified
622 as “solid C–S–H” and “bulk C–S–H”. The “solid C–S–H” includes the Ca–O backbone layers with
623 SiO2 tetrahedral and the interlayer water in between but excludes the water and any
624 hydroxyls on the outer surface of the stacked layers. The “bulk C–S–H” is the C–S–H inclusive
625 of the gel water [20]. Both solid and bulk resulting from NSF can decrease the porosity and
626 consequently, lead to increasing fibre-cement interface bonding.

627 It is well known that by reducing the porosity, water absorption and moisture movement
628 decrease, whereas density increases. The more hydrated products deposit on glass and Kraft
629 fibres, the more interfacial bonding is created, leading to the greater flexural strength and
630 fracture toughness.

631 The capillary pores (also called “interhydrate pores”) that are formed with growing C–S–H
632 needles (hydration process) plateau rapidly with no further decrease. The reservoir of capillary
633 water which has already been absorbed by cellulose fibres (i.e. KFs) reaches the interhydrate

pores over the hydration process and reacts with the silica fume that has already been placed in capillary pores [20,21].

As seen in Fig. 10, many of capillary voids or even large pores have not been filled by water that needed to be discussed from a viewpoint of porosity. Porosity would have a negative effect on durability and strength if the tiny voids are linked and joined together resulting in increasing penetrability of CCB. In other words, the capillary pores which are not connected or joined with each other cannot affect the durability of CCB. They can even increase fracture toughness and also the durability of composite against environmental condition such as freeze-thaw cycling.

5 Concluding remarks

The results of this research showed that discrete alkali resistant glass fibres have an appropriate potential to be used in combination with Kraft pulp fibres, cement and Nano-silica fume to produce CCB's. The following can be deduced from the study:

1. Flexural strength of CCBs increases substantially as GLs are introduced into the mix containing only KFs .
2. GLs with a length of 3 mm showed better consistency with other ingredients in the mix and could improve flexural strength, water absorption and moisture movement.
3. The most impressive flexural strength results (12 MPa) belong to the group "K4-GL2-3mm-N2" which comprises of 4% KF's, 2% of 3 mm length GL's, and 2% NSF. The corresponding values for water absorption, moisture movement and density of that group are 22.5%, 0.11% and 2260 kg/m^3, respectively.

4. There is a particular limit for adding GLs into the mix depending on fibre size, manufacturing process and the amount of other ingredients. Extra fibres cause negative effects, such as non-uniform fibre dispersion or clumping fibres which could weaken CCB's properties. In this study, laboratory observation indicates that 3% of cement weight is the maximum limit for incorporating of glass fibre into the mix.

5. Microstructural studies showed that the pores in the vicinity of GLs are more than the counterpart KFs.

6. Incorporating an appropriate percentage of NSF into the mix can improve the flexural strength and reduce the porosity of the specimens. Pozzolanic reaction along with filler action of NSF form a stronger bonding at fibre-cement interfacial zone and also improve the durability of CCB by decreasing the permeability of the matrix.

6 Acknowledgements

The authors would like to thank Coventry University for providing the required facilities for this research.

7 References

[1] Khorami M, Sobhani J. An experimental study on the flexural performance of agro-waste cement composite boards. Int J Civ Eng 2013;11:207-16.

[2] Khorami M, Ganjian E, Srivastav A. Feasibility Study on Production of Fiber Cement Board Using Waste Kraft Pulp in Corporation with Polypropylene and Acrylic Fibers. Materials Today: Proceedings 2016;3:376-80.

[3] Khorami M, Ganjian E. The effect of limestone powder, silica fume and fibre content on the flexural behaviour of cement composite reinforced by waste Kraft pulp. Constr Build Mater 2013;46:142-9.

[4] Ardanuy M, Claramunt J, Toledo Filho RD. Cellulosic fibre reinforced cement-based composites: a review of recent research. Constr Build Mater 2015;79:115-28.

[5] Khorami M, Ganjian E. Comparing the flexural behaviour of fibre–cement composites reinforced bagasse: Wheat and eucalyptus. Constr Build Mater 2011;25:3661-7.

[6] Pacheco-Torgal F, Jalali S. Cementitious building materials reinforced with vegetable fibres: A review. Constr Build Mater 2011;25:575-81.

[7] Xie X, Zhou Z, Jiang M, Xu X, Wang Z, Hui D. Cellulosic fibres from rice straw and bamboo used as reinforcement of cement-based composites for remarkably improving mechanical properties. Composites Part B: Engineering 2015;78:153-61.

[8] Mohammad Kazemi F, Doosthoseini K, Ganjian E, Azin M. Manufacturing of bacterial nano-cellulose reinforced fibre− cement composites. Constr Build Mater 2015;101:958-64.

[9] Ashori A, Tabarsa T, Valizadeh I. Fiber reinforced cement boards made from recycled newsprint paper. Materials Science and Engineering: A 2011;528:7801-4.

[10] Torkaman J, Ashori A, Momtazi AS. Using wood fiber waste, rice husk ash, and limestone powder waste as cement replacement materials for lightweight concrete blocks. Constr Build Mater 2014;50:432-6.

[11] Sonphuak W, Rojanarowan N. Strength improvement of fibre cement product. International Journal of Industrial Engineering Computations 2013;4:505-16.

[12] Hakamy A, Shaikh F, Low IM. Effect of calcined nano clay on microstructural and mechanical properties of chemically treated hemp fabric-reinforced cement nanocomposites. Constr Build Mater 2015;95:882-91.

[13] Marikunte S, Aldea C, Shah SP. Durability of glass fibre reinforced cement composites:: Effect of silica fume and metakaolin. Adv Cem Based Mater 1997;5:100-8.

[14] Asokan P, Osmani M, Price AD. Assessing the recycling potential of glass fibre reinforced plastic waste in concrete and cement composites. J Clean Prod 2009;17:821-9.

[15] Aly M, Hashmi MSJ, Olabi AG, Messeiry M, Abadir EF, Hussain AI. Effect of colloidal nano-silica on the mechanical and physical behaviour of waste-glass cement mortar. Mater Des 2012;33:127-35.

[16] Ganjian E, Khorami M. Application of Kraft and Acrylic Fibres to Replace Asbestos in Composite Cement Board. 2008:52-61.

[17] Ali M, Majumdar AJ, Singh B. Properties of glass fibre cement—the effect of fibre length and content. J Mater Sci 1975;10:1732-40.

[18] D.D. T. Creep characteristics of glass reinforced cement under flexural loading. Cement and Concrete Composites 1995;17:267-79.

[19] Enfedaque A, Gálvez J, Suárez F. Analysis of fracture tests of glass fibre reinforced cement (GRC) using digital image correlation. Constr Build Mater 2015;75:472-87.

[20] Muller A, Scrivener K, Skibsted J, Gajewicz A, McDonald P. Influence of silica fume on the microstructure of cement pastes: New insights from 1 H NMR relaxometry. Cem Concr Res 2015;74:116-25.

[21] Vinayag MB, Aleem MA, Thaarrini J, Roja SY. REVIEW ON STRENGTH AND DURABILITY CHARACTERISTIC OF GEOPOLYMER CONCRETE WITH MACRO SILICA, NANO SILICA. 2015.

[22] Pourjavadi A, Fakoorpoor SM, Khaloo A, Hosseini P. Improving the performance of cement-based composites containing superabsorbent polymers by utilisation of nano-SiO2 particles. Mater Des 2012;42:94-101.