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Damage assessment in cellulose–cement composites using dynamic mechanical characteristics

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Abstract

This paper reports on an experimental investigation of test methods that can detect damage in cement composites, incorporating cellulose macro-nodule fibers, subjected to freezing/thawing and immersion in hot water. Conventional methods of ascertaining damage, such as porosity and flexural strength testing, were carried out, along with dynamic mechanical testing for determination of elastic modulus and damping capacity. Increase in porosity and reduction in flexural strength (as compared to normally cured specimens) were observed for low fiber volume composites subjected to freezing and thawing, whereas no significant changes were observed for specimens with higher fiber volumes. Determination of dynamic elastic modulus and specific damping capacity at regular intervals of exposure showed that the high volume of porous macro-nodules in the mixture prevented damage due to freezing and thawing by acting as stress release sites. No appreciable change in porosity and flexural strength was observed for specimens continuously exposed to hot water; however, reduction in relative modulus and increase in damping capacity was observed. The changes in these system properties suggest that a certain degree of damage might be occurring under sustained hot water exposure, which the determination of porosity and flexural strength could not capture. Using a stiffness-loss map, it is shown in this paper that the damping characteristic is more sensitive than the stiffness in detecting damage as a result of continuous exposure to hot and wet conditions.
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Keywords: Cellulose–cement composites; Freeze–thaw; Hot water immersion; Relative modulus; Specific damping capacity; Loss tangent; Stiffness-loss map

1. Introduction

Various types of synthetic and natural fibers are used in cementitious matrices to enhance the mechanical properties of the composite like tensile and flexural strengths, toughness, impact resistance, and fracture energy [1–7]. The use of cellulose fibers in cementitious matrices have gained prominence because: (i) they make the composite lighter at high fiber contents, (ii) they have comparable cost-performance ratios to similar building materials [8], and (iii) they could be processed from waste paper, thus expanding the opportunities for waste utilization in cementitious materials [9]. The applications of cellulose fiber–cement composites include production of thin and flat corrugated sheets, residential wall boards, and highway noise barriers [1,2,8]. Fresh and hardened properties of cement composites reinforced with cellulose fibers have been reported in detail [10–16]. Recently, the use of morphologically altered cellulose fibers in cementitious matrix has been proposed to be beneficial in acoustic absorption and vibration damping [17].

Durability characteristics of cellulose fiber reinforced cement composites have received attention in the past. The deterioration mechanisms of such composites may be broadly classified as: (i) fiber degradation, (ii) fiber–matrix chemical interaction, (iii) fiber–matrix physical interaction at the interface, and (iv) volume stability and cracking sensitivity of the composite [18]. Accelerated aging tests like hot water exposure, cyclic wetting–drying, and carbonation

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on cellulose–cement composites have shown that the aging mechanisms could be directly related to the matrix and fiber types, porosity and the aging method used [19,20]. Cellulose–cement composites were found to be exceptionally moisture sensitive; increased moisture content results in reduction in flexural strength and increase in toughness [14,21]. Several studies have shown that accelerated wetting–drying and hot water immersion can lead to the alkaline pore water of concrete attacking the lignin in cellulose fibers, weakening the link between individual cells that constitute the fiber. Increase in strength and reduction in toughness caused due to the petrification of cellulose fibers are the consequences of alkaline pore water attack [18,22–25]. The changes in porosity and water absorption of cellulose–cement composites subjected to freezing and thawing cycles were reported to be statistically insignificant [23].

In all the above studies dealing with durability of cellulose–cement composites, the performance of the material after a period of aging (hot water soaking, cyclic wetting–drying, accelerated carbonation, cyclic freeze–thaw) was assessed using physical properties like porosity and/or mechanical properties like flexural strength and toughness. This mostly necessitates destructive testing of the samples, usually carried out at the end of the performance test duration. The drawbacks of these destructive tests are that they do not capture the changes in material behavior during the test period, and the mechanical tests may give misleading results (for example, continuous immersion of cellulose–cement composites in hot water does not show any sign of degradation in flexural strength, contrary to observations in natural conditions [18]). Hence there is a need to introduce parameters that can be obtained from non-destructive tests as reliable indicators of durability of cellulose–cement composites.

This paper examines the use of elastic modulus and damping determined by dynamic resonance techniques to quantify damage in cellulose–cement composites. Apart from quantitative measures like increase in porosity and decrease in flexural strength, damage, as referred to in this paper, is indicated by a reduction in dynamic modulus and an increase in specific damping capacity of the composite specimens subjected to a particular exposure regime for a certain duration as compared to that of a specimen normally moist cured for the same duration. Dynamic modulus is chosen in this study because the periodic monitoring of change in stiffness with duration of exposure to extreme conditions is believed to be a convenient and powerful method to detect and quantify damage. The fact that energy absorption in composites is generally due to damage, debonding, and defects lends support to the use of damping as a means of damage identification and quantification.

A brief introduction to the mechanisms influencing damping in composite materials is given in Section 1.1. Since dynamic modulus and damping can be measured non-destructively at desired intervals on specimens subjected to varying exposure conditions, it eliminates the drawbacks of destructive testing methods mentioned earlier.

1.1. Damping mechanisms in composite materials

Damping defines the energy dissipation properties of a material or system. Viscoelastic materials can be used for damping mechanical vibrations and dissipating sound waves [26,27]. For a composite material, damping depends on the proportion, morphology and mechanical properties of the constituent phases, as well as the impedance mismatch at the interfaces. Air voids, presence of water in the voids, and microcracks are believed to be responsible for damping in plain concrete [28]. Opening and closing of microcracks during dynamic flexure, resulting in friction between the fiber and matrix is stated to be responsible for damping in stiff cement concrete matrices reinforced with viscoelastic fibers [29]. For polymer matrices modified with cellulose fibers, it was observed that good adhesion between fiber and matrix results in low damping values and high dynamic modulus [30]. Friction in the fiber–matrix interface and irreversible macro-cracks in the matrix caused increasing damping [31]. Energy dissipation in the area of matrix cracks and damaged fibers is very high as compared to undamaged regions [32].

The premise of this research, based on the foregoing discussion, is that it should be possible to detect and quantify damage in cellulose–cement composites with ''macro-nodule'' cellulose fibers as inclusions, exposed to either cycles of freezing and thawing, or wet conditions and elevated temperatures, from the dynamic mechanical characteristics of the material such as dynamic elastic modulus and damping. Macro-nodules are used in this study because the cementitious matrices incorporating them were found to be capable of absorbing sound and dissipating energy by vibration, opening avenues for application in quieter wall panels, noise barriers, and structural components [17].

2. Experimental program

2.1. Materials and mixture proportions

The matrix for the cellulose–cement composites used in this study was a cement–sand mortar with approximately 50% of paste (cement + water) by volume. Sand and cellulose fibers made up the remaining volume of the mixture. ASTM Type I cement was used along with river sand. Morphologically altered cellulose fibers, called macro-nodules, provided by Weyerhaeuser™ were used at levels of 2.5%, 5%, and 7.5% of total volume of the composite. Macro-nodules (Fig. 1) are fiber agglomerates, ranging from 1 to 8 mm in size, formed from bleached softwood by a flaking process that did not separate individual fibers during manufacture. Macro-nodules are porous, and because of their size, act as porous aggregates in the mixture.

Fig. 1. Macro-nodule fibers used in this study (scale shown in cm).

Table 1
Mixture proportions and flow characteristics of the mixtures

Fiber volume (%)	*w/c*	Volume percentage of mix constituents			Admixtures (% by wt. of cement)		Flow (±5%)
		Cement	Water	Sand	Water reducer	Accelerator	
0.0	0.45	19.86	28.16	51.98	0.0	0.0	70
2.5	0.50	18.79	29.54	49.17	1.0	0.0	65
5.0	0.56	17.70	31.02	46.28	1.5	1.0	55
7.5	0.62	16.80	32.26	43.44	2.5	1.0	50

With increasing fiber content, the water demand increases because of the water absorption of the fiber nodules as well as the reduced mobility of these compliant inclusions in the mixture. Since the addition of water reducer was not effective in maintaining constant water–cement ratio (*w/c*) for all mixtures, the *w/c* was adjusted to maintain fresh mixture workability at a reasonably practical level, represented by flow values determined in accordance with ASTM C 1437-01. An accelerator was added for mixtures with high volumes of fibers (5.0% and 7.5%) since a considerable retardation in setting time was noted otherwise, which can be attributed to the presence of lignin in the cellulose fibers. Table 1 summarizes the mixture proportions and flow characteristics of the fresh composites.

2.2. Specimen preparation

All the composite specimens were prepared in a Hobart mortar mixer having a capacity of 13.5 l. Cement and sand was first mixed at low speed for 1 min and then the fibers were added while mixing. Approximately three quarters of the mixing water was added and all ingredients were mixed at a medium speed for 2 min. The remaining water was then added along with the water reducer (and accelerator, when needed) and mixed for one more minute, until a uniform mixture was obtained. Sufficient care was taken to ensure that the mixer did not run for longer than required durations or at higher than required speeds so that the macro-nodules are not broken down in the mixture.

For each mixture, prismatic specimens (250 mm × 75 mm × 25 mm) were cast for determination of porosity, dynamic elastic modulus, specific damping capacity, and flexural strength. This specimen size was chosen in order to have a higher ratio of length to thickness (10 in this case) which produces a larger output in amplitude in the determination of specific damping capacity by the dynamic excitation method [32]. All the specimens were consolidated using external vibration and were kept damp inside the molds for 24 h, after which they were moist cured in a curing chamber (>98% RH, 23 °C) until the test age.

2.3. Methods to evaluate durability of cellulose–cement composites

2.3.1. Cyclic freezing and thawing

After 14 days of moist curing, prismatic cellulose–cement composite specimens were immersed in water in flexible containers and subjected to freezing and thawing in a controlled temperature chamber. The specimens were subjected to one freeze–thaw cycle per day. The temperature of the chamber was maintained at −16 °C for a period of 5 h before raising it at a rate of 6.3 °C/h to 22 °C where the temperature was maintained for a period of 5 h. At the end of 5 h the chamber was cooled at a rate of 7.5 °C/h until it reached −8 °C and then cooled to −16 °C at a slower rate of 1.5 °C/h. The fundamental flexural resonant frequencies of the specimens were determined prior to freezing and then periodically during the test, from which the dynamic elastic moduli were calculated as explained in Section 2.4.2. The specific damping capacities (Section 2.4.3) were also monitored at fixed intervals during the test. The specimens were subjected to 100 cycles of freezing and thawing after which the test was stopped. Flexural strength tests were carried out at the end of the test duration.

2.3.2. Immersion in hot water

The hot water immersion test method investigates the long term chemical interaction between the constituents in the composite. Wet conditions and elevated temperature are used to accelerate the deterioration. Prismatic specimens were saturated in water and maintained at 60 ± 2 °C. The test procedure was in accordance with ASTM C 1185-03 except for the duration which was 100 days in hot water while the standard specifies 56 ± 2 days. The standard suggests flexural strength testing at the end of the test duration and reporting the ratio of the strengths of the damaged composite to that of the control specimen. The resonant frequencies (and hence, the dynamic elastic modulus) and specific damping capacities of the specimens were also monitored in this study at fixed intervals to detect damage occurring during the test duration.

2.4. Test methods

2.4.1. Determination of porosity

Porosity was determined on slices (75 mm × 75 mm × 25 mm) cut from the prismatic specimens (250 mm × 75 mm × 25 mm). A procedure employing vacuum saturation as described in RILEM CPC 11.3 [33] was used to determine the porosity of the mortars with and with out macro-nodules. Details about porosity measurement could be found in [17].

2.4.2. Determination of dynamic modulus from resonance

The relative dynamic modulus (P) was calculated from the flexural resonant frequencies before damage (n) and after specified cycles of exposure to either freezing and thawing or hot water (n_1) as

$$P = \left(\frac{n_1}{n}\right)^2 \tag{1}$$

The specimens were supported on rollers at their fundamental nodal points (0.224 L from the supports) and excited to obtain the fundamental flexural resonant frequency (n_f).

The dynamic modulus of elasticity (E) was calculated from the fundamental flexural resonant frequency using impulse excitation of vibration in accordance with ASTM E 1876-01. The dynamic Young's modulus (E) in Pa can be calculated as

$$E = 0.9465(mn_f^2)\left(\frac{L^3}{t^3}\frac{1}{b}\right)T_1 \tag{2}$$

where m is the mass of the specimen in kg, L, b, and t are respectively the length, width and thickness of the specimen in metres, n_f is in Hz, and T_1 is a constant to account for finite thickness of the bar, and Poisson's ratio. ASTM E 1876-01 provides the criteria and the equations for the determination of T_1.

2.4.3. Determination of damping level of the system

The effect of damping in a system is evidenced by the diminishing amplitude of vibration with time. The measurements of either the rate of decay of free oscillation produced by suddenly removing an oscillating force, or the bandwidth of the frequency spectrum at resonance peak are common methods to evaluate damping [28,32]. The specific damping capacity (ξ) was determined in this study by the decaying sine wave method using Grindosonic™ equipment.

3. Analysis and discussion of the results

The two durability characteristics of cement composites incorporating macro-nodule fibers evaluated in this study are its resistance to cycles of freezing and thawing and response to immersion in hot water. Companion specimens that were moist cured for the entire duration of the durability tests were used in order to facilitate comparison. The variations in properties when subjected to both conditions of exposure are discussed in detail in the following sections.

3.1. Influence of exposure conditions on porosity

Fig. 2(a) depicts the relationship between fiber volume and porosity for cellulose–cement composites that are either moist cured for 100 days or subjected to 100 cycles of freezing and thawing after 14 days of moist curing. The porosity increases with increase in fiber volume for both the cases. It could be noticed from this figure that the increase in porosity of the plain mortar specimen (0% fiber volume) after 100 cycles of freezing and thawing is more than that of the specimens with fibers, indicating a higher degree of matrix cracking in the plain mortar. At a fiber volume of 7.5%, there is little statistical difference between the porosities of specimens that are moist cured for 100 days or subjected to 100 cycles of freezing and

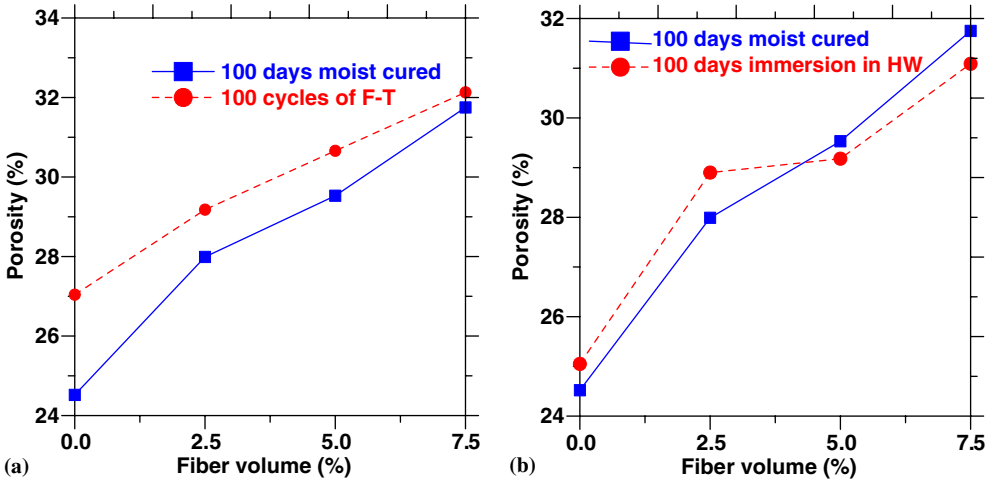

Fig. 2. Porosity versus fiber volume – comparison between specimens moist cured for 100 days and subjected to: (a) 100 cycles of freezing and thawing and (b) 100 days of immersion in hot water.

thawing. In the saturated fiber reinforced specimens, it is believed that the porous and complaint macro-nodule cellulose fibers act as escape sites for the freezing water in the capillaries of the cement paste, protecting them from dilating, and thus allowing for the release of stresses. The ultimate result is reduced damage, and consequently little or no change in porosity.

A comparison of the porosities of cellulose–cement composites either moist cured for 100 days, or subjected to 100 days of exposure to hot water is shown in Fig. 2(b). The porosities of the low fiber volume specimens (0% and 2.5%), when subjected to hot water immersion, are slightly higher than those of the 100 day moist cured specimens, while the trend is reversed for specimens with higher fiber volumes (5% and 7.5%). Considering the fact that there is only a less than 1% difference between porosities (average of 3 companion specimens) of specimens subjected to either 100 days moist curing or 100 days immersion in hot water, it could be safely stated that determination of porosities does not indicate any damage in cellulose–cement composites immersed in hot water for 100 days. The percentage change in mass of composite specimens subjected to hot water immersion is also in the order of less than 1%.

3.2. Influence of exposure conditions on flexural strength

The flexural strengths (f_r) of cellulose–cement composites were determined according to ASTM C 78-02. The variation of flexural strengths with porosity for specimens subjected to all three conditions of exposure is shown in Fig. 3(a). The flexural strengths can be adequately described by the expression

$$f_{r(V_f)} = f_{r(0)} - AV_f \quad (3)$$

where $f_{r(V_f)}$ is the flexural strength at any fiber volume (V_f) for a given exposure condition, $f_{r(0)}$ is the flexural strength at 0% fiber volume (plain mortar) for that exposure condition, and A is a constant.

The flexural strength of the plain mortar subjected to 100 cycles of freezing and thawing is significantly lower than that of similar specimens subjected to 100 day immersion in hot water or moist cured for 100 days, indicating that freezing and thawing creates stresses high enough to microcrack the matrix of the plain mortar. However, with addition of macro-nodule fibers, (i) the flexural strengths decrease because the macro-nodules are compliant inclusions in the matrix, and higher fiber content necessitates a higher w/c, and (ii) the difference between the flexural strengths of the composites under freezing and thawing, and the other two exposure conditions decreases. At a fiber volume of 7.5%, there is no discernable difference between the flexural strengths for composites exposed to any of the exposure regimes. This once again points to the ability of the porous, compliant macro-nodules to act as stress release sites, and thus to reduce the damage in composite specimens. The flexural strengths of the composite specimens undergoing hot water exposure remain fairly close to those of the normally cured specimens at all fiber volumes. This is consistent with results from previous studies [19,22,23], leading again to the conclusion that hot water immersion does not damage cellulose–cement composites. Same conclusion can be arrived at from Fig. 3(b) which shows the ratio of the strengths of the composite subjected to the two exposure conditions ($f_{r\text{-damaged}}$) to that of the specimens moist cured for 100 days ($f_{r\text{-undamaged}}$), as recommended by ASTM C 1185-03. With freezing and thawing, normalized strength increases with fiber volume, indicating that the porous macro-nodules are capable of mitigating damage. $f_{r\text{-damaged}}/f_{r\text{-undamaged}}$ remains fairly constant, and close to 1.0, irrespective of the fiber volume, for composites immersed in hot water, suggesting that hot water immersion does not damage the composite.

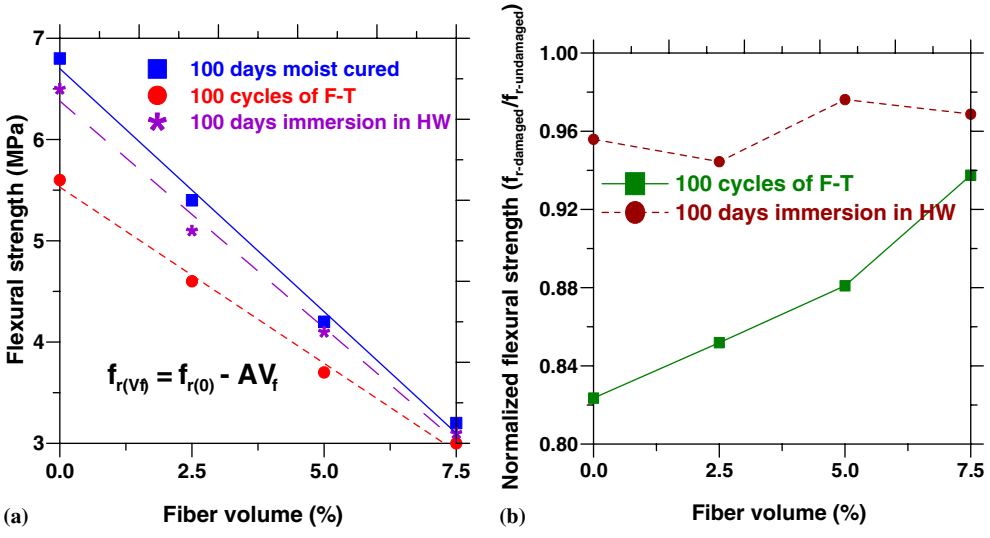

Fig. 3. Variation in: (a) flexural strength with fiber volume and (b) normalized flexural strength with fiber volume for composites subjected to different exposure conditions.

3.3. Limitations of porosity and flexural strength as parameters quantifying damage in cellulose–cement composites

It could be noticed from Fig. 2(a) that the porosity is higher for plain mortar and low fiber volume cellulose–cement composites undergoing freezing and thawing, than for the normally cured composites, indicating matrix cracking. The flexural strength results from Fig. 3 also point to a relatively higher loss of strength for low fiber volume composites subjected to freezing and thawing. From Fig. 2(b) it is seen that there is no considerable difference between porosities of composite specimens subjected to either 100 days of moist curing or 100 days of hot water immersion. Fig. 3(a) shows that the flexural strengths of composites undergoing hot water exposure are not significantly different from those moist cured for 100 days. From tests on both porosity and flexural strength, it is therefore observed that immersion in hot water does not produce damage in cellulose–cement composites as compared to cycles of freezing and thawing. But deterioration has been noticed in natural conditions for cellulose–cement composites exposed to wet conditions and elevated temperatures akin to hot water immersion test [18]. The inference therefore is that the conventional methods of damage identification and quantification like porosity and flexural strength determination at the end of the exposure duration might not be able to capture the material damage in cellulose–cement composites undergoing exposure to extreme conditions. More sensitive testing methods are needed to detect and quantify damage in cellulose–cement composites.

3.4. Dynamic elastic modulus to assess damage in cellulose–cement composites

The use of relative dynamic modulus calculated from Eq. (1) as an indicator of damage inflicted by freezing and thawing and immersion in hot water is explored in this section. Separate sets of prismatic specimens were subjected to the exposure conditions after 14 days of moist curing. Fig. 4(a) depicts the variation in relative dynamic modulus of the specimens with number of cycles of freezing and thawing. The drop in relative dynamic modulus is significant for specimens without fibers, indicating that freezing and thawing induces stresses in the matrix, and thus microcracking the plain mortar. With composites having a fiber volume of 2.5%, the magnitude of the modulus drop is reduced, indicating that the complaint fibers are acting as stress release sites. With higher fiber volumes, the relative modulus is practically unchanged with increasing number of freezing and thawing cycles, and it could be inferred that the cementitious matrix is undamaged due to freezing and thawing. However, it needs to be mentioned here that the effective elastic modulus of the macro-nodule fiber reinforced specimens reduces with increase in fiber volume; the reason being the same as that for the reduction in flexural strength.

Fig. 4(b) shows the variation in relative dynamic modulus with duration of immersion in hot water. The relative dynamic modulus shows a consistent drop with duration of immersion for the specimens with 0% and 2.5% fibers by volume for the entire test period. This could be attributed to the presence of microcracks in the matrix because of increased temperature. However, with higher fiber volumes, there is no appreciable change in the relative modulus after a certain duration of exposure to hot water. Some microcracking happens early on, resulting in a drop in relative modulus, but at longer durations of immersion in hot water, it is possible that petrification (densification of the fiber–matrix interface) occurs [18,25], negating the effects of cracking, thereby stabilizing the relative dynamic modulus. It is postulated here that this mechanism could be the reason for the porosity of the high fiber volume specimens immersed in hot water (ϕ_{HW}) being very close to or slightly lower than the porosities of the specimens moist cured for 100 days ($\phi_{undamaged}$), as seen from Fig. 2(b). More studies are needed to confirm this hypothesis.

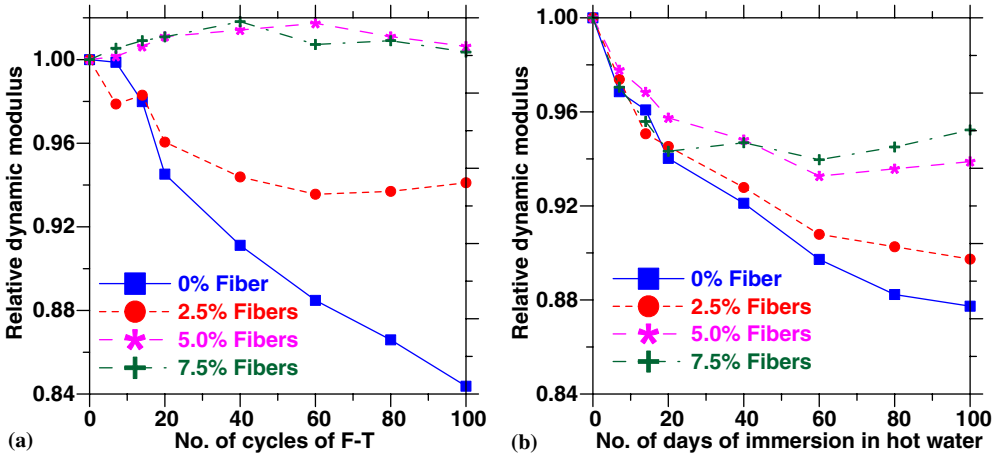

Fig. 4. Variation in relative dynamic modulus for specimens subjected to: (a) freezing-thawing cycles and (b) immersion in hot water.

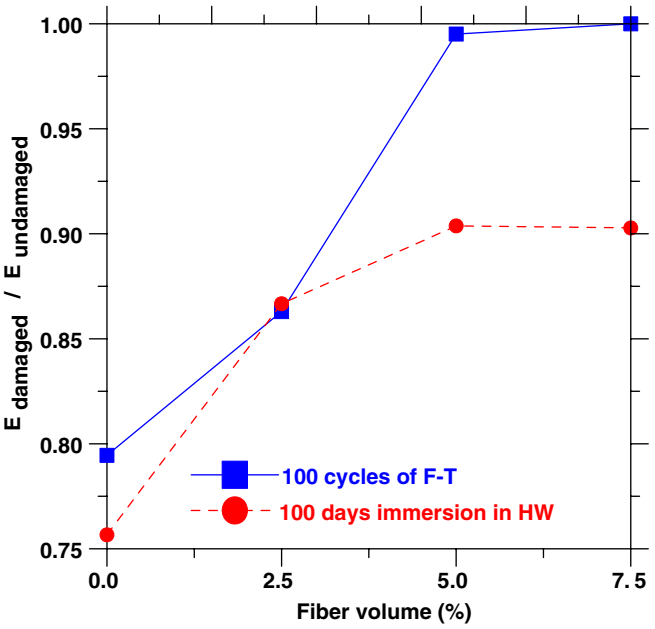

Fig. 5. Variation in ratio of damaged to undamaged modulus ($E_{damaged}$/$E_{undamaged}$) with fiber volume.

Fig. 5 represents the relative damage expressed as the ratio of the modulus after the damage ($E_{damaged}$) to that of the specimens moist cured for 100 days ($E_{undamaged}$) for both freezing and thawing and immersion in hot water. The modulus values were calculated using Eq. (2). For all fiber volumes, the normalized moduli for the specimens subjected to freezing and thawing are observed to be higher than or equal to those of composites subjected to immersion in hot water. The relative damage, therefore, is higher when the specimens are immersed in hot water, which is contrary to the deduction from the flexural strength results (Fig. 3) which showed higher apparent damage for the composites subjected to freezing and thawing. Conventional testing procedures like porosity and flexural strength predicted little or no damage in the composites subjected to hot water immersion as compared to the specimens moist cured for the entire test duration (as seen from Figs. 2 and 3) while the dynamic modulus could better detect and quantify the relative damage in these materials. Also, the non-destructive nature of this test provides a means to detect the variations during the test period, as is evidenced from the determination of the change in trend of relative modulus after 60 days of hot water immersion (Fig. 4(b)).

3.5. Damping characteristics to assess damage in cellulose–cement composites

Specific damping capacity (ξ) is commonly used as a measure of the capacity of the material to dissipate energy. Compliant materials tend to show a higher value of ξ than stiffer materials. The damping capacities were determined in a water saturated state for all the specimens. Special care was taken to make sure that there was no water loss during testing since drying was found to reduce the damping capacities drastically [17].

3.5.1. Variation in specific damping capacities

The variation in specific damping capacity (ξ) with number of cycles of freezing and thawing for the composite specimens is given in Fig. 6(a). ξ increases with increase in number of freezing and thawing cycles for specimens without fibers, which can be attributed to an increase in matrix cracking. For specimens with 2.5% fiber volume, ξ increases initially and then stays constant. This behavior is in agreement with the observed drop in relative dynamic modulus shown in Fig. 4(a). Damage is manifested as a reduction in relative modulus and an increase in damping capacity. For higher fiber volumes, there is an initial increase in damping, followed by a reduction, and then ξ plateaus off. The ultimate values of ξ for the high fiber volume specimens after 100 cycles are essentially the same as when the freezing and thawing process was started, indi-

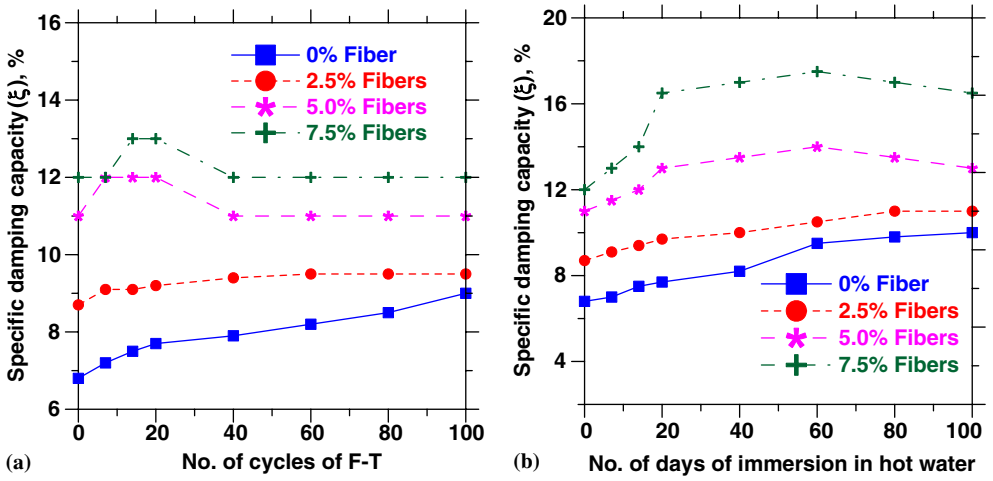

Fig. 6. Variation in specific damping capacity (ξ) for specimens subjected to: (a) freezing-thawing cycles and (b) immersion in hot water.

cating that freezing and thawing induces no significant damage in high fiber volume specimens, a deduction that has been made from the observation of relative modulus values also. After about 40 cycles of freezing and thawing, the difference between the ξ values of specimens with 2.5%, 5%, and 7.5% fibers essentially remains the same, as can be seen from the portions of the curves that are parallel to the X-axis. This difference is primarily due to the presence of compliant macro-nodules rather than the damage.

Fig. 6(b) depicts the variation in ξ with number of days of immersion in hot water for all the composite specimens. The trend for specimens with 0% and 2.5% fiber volume is the same as that observed for specimens subjected to freezing and thawing – increasing damping capacity with exposure duration, indicating the presence of damage. This is in agreement with the relative modulus variation for these specimens shown in Fig. 4(b). It could be inferred that the observed changes in damping capacity is due to microcracking rather than an increase in water uptake since no appreciable mass change was noticed for the specimens. For composites with 5% and 7.5% fiber volume, ξ increases up to a certain duration of immersion in hot water, and then shows a marginal drop, which could possibly be attributed to petrification. The eventual damping capacity at 100 days of immersion in hot water is higher than the damping capacity at the start of hot water immersion test for all the samples, indicating that immersion in hot water damages the composites.

3.5.2. Comparison of loss tangents

Loss tangent (the tangent of phase angle between stress and strain in sinusoidal loading – $\tan\delta$), derivable from ξ is another useful measure of the material damping characteristic, and is proportional to the energy loss per cycle [34]. It is the ratio of the imaginary and real parts of the complex dynamic modulus E^* which is given as

$$E^* = E' + iE'' \tag{4}$$

where E' is the storage modulus given as $E\cos\delta$ and E'' is the loss modulus given as $E\sin\delta$. The loss tangent then becomes

$$\tan\delta = \frac{E''}{E'} \tag{5}$$

which can also be represented in terms of specific damping capacity as

$$\tan\delta = \frac{\xi}{2\pi} \tag{6}$$

Most structural materials have very low loss tangents ($\approx$0.005–0.01). In general, materials with high loss tangents tend to be more compliant [27,34].

Fig. 7 shows the loss tangent ($\tan\delta$) plotted against fiber volume for the cellulose–cement composites moist cured for 100 days, subjected to 100 cycles of freezing and thawing, and 100 days of immersion in hot water. It could be noticed from this figure that the loss tangent is highest for the specimens immersed in hot water for all fiber volumes, indicating that the relative damage is higher for cellulose–cement composites under such a durability testing regime. Comparing the loss tangents of normally cured specimens, and specimens subjected to freezing and thawing, it is seen that the difference between loss tangents decrease with increase in fiber volume. For specimens with 7.5% fiber volume, the loss tangents for both these regimes are same. This, coupled with the practical invariance of relative elastic modulus (Fig. 4(a)), reinforces the conclusion that composites with high volumes of macro-nodules do not undergo damage due to freezing and thawing. The difference between loss tangents at 7.5% fiber volume for specimens subjected to freezing-thawing and hot water immersion is high, pointing to the ability of loss tangent (or specific damping capacity) to detect damage in cellulose–cement composites. The variation in loss tangents (Fig. 7) are more striking than the variation in relative modulus (Fig. 5), showing that the damping characteristic is more sensitive to damage than the modulus.

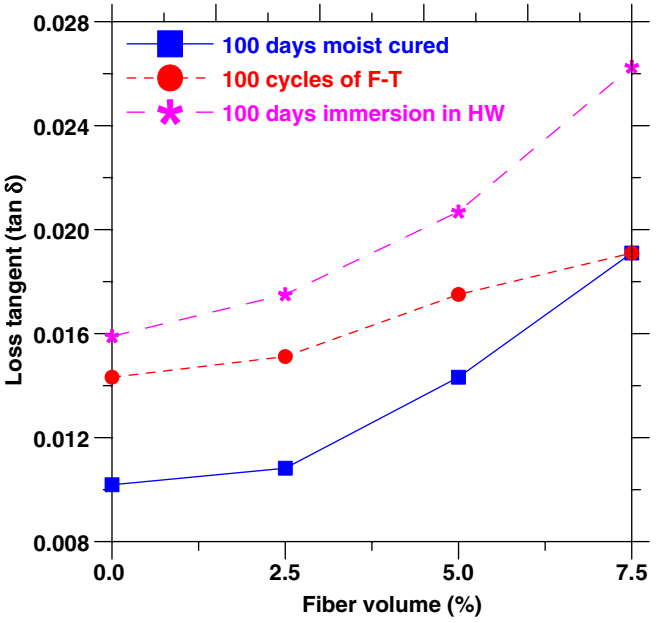

Fig. 7. Variation in loss tangent ($\tan\delta$) with fiber volume for all the three regimes.

The analysis of both porosity and flexural strength results (Figs. 2(b) and 3) indicated that the damage in composites subjected to hot water immersion is negligible or non-existent, but the analysis of the damping characteristics of the material shows that composites subjected to hot water immersion undergo damage, which can be detected and quantified using specific damping capacity or loss tangent.

3.6. Stiffness-loss map for cellulose–cement composites undergoing damage

Stiffness-loss plots (E' versus $\tan\delta$) for cellulose–cement composites moist cured for 100 days, subjected to 100

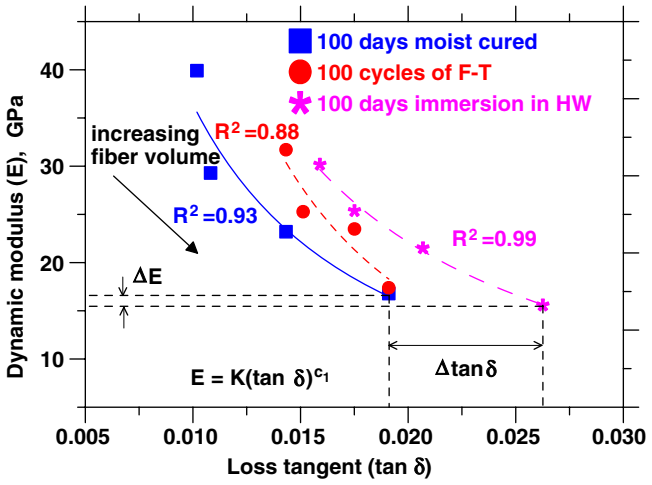

Fig. 8. Stiffness-loss map for composites subjected to all the three regimes.

cycles of freezing and thawing, and 100 days of immersion in hot water are given in Fig. 8. The dynamic modulus values obtained experimentally (E) is plotted in the Y-axis instead of E' since for small values of δ, E' (which is equal to $E\cos\delta$) $\approx E$. The relationship between stiffness and loss tangent could adequately be expressed by the relation [17]

$$E = K(\tan\delta)^{c_1} \tag{7}$$

where K and c_1 are constants depending on the magnitude of damage in the specimens. These values are the highest for undamaged specimens. The coefficients of determination (R^2) for the fit are 0.93, 0.88, and 0.99 for moist cured specimens, specimens subjected to freezing and thawing, and hot water immersion respectively.

It could be observed from Fig. 8 that the specimens moist cured for 100 days show the smallest values of tan δ. For both the exposure conditions investigated in this study, the loss tangents increase with fiber volume, indicating that the materials have become more compliant with damage. The only exception to this is the case with the composite containing 7.5% fiber volume subjected to freezing and thawing, which shows the same stiffness and loss tangent as that of the undamaged composite, for reasons stated previously. The stiffness-loss curve of composites subjected to hot water immersion has the maximum shift to the right; another representation that hot water immersion induces damage in cellulose–cement composites. The stiffness-loss map is thus a useful aid to visualize the reduction in elastic modulus and increase in damping characteristic together for the composites subjected to durability tests, in relation with the undamaged composite.

Comparing the E and tan δ values for moist cured and hot water immersed composites with 7.5% fiber volume in Fig. 8 (the bottom most points on the E-tan δ plot), it could be noticed that the change in E values (ΔE) is small compared to the difference in their loss tangents ($\Delta\tan\delta$), with tan δ being higher for the hot water immersed specimen. This goes on to prove an earlier mentioned point that, for cellulose–cement composites subjected to hot water immersion, the damping characteristic (either ξ or tan δ) is more sensitive to damage than the stiffness.

4. Conclusions

This paper has dealt with the durability of macro-nodule cellulose fiber–cement composites subjected to freezing and thawing and immersion in hot water, and test methods that are capable of detecting damage in the specimens. The following are the conclusions drawn from this investigation:

(i) Determination of porosity and flexural strength of cellulose–cement composites at the end of exposure durations resulted in the conclusion that (a) an increase in fiber volume results in a reduction in relative damage induced by freezing and thawing, and (b) there is negligible or no damage in specimens subjected to hot water immersion.

(ii) As determined from relative elastic modulus and specific damping capacity results, freezing and thawing damage was the highest for plain mortar specimens. Composites with higher volumes of macro-nodule cellulose fibers were able to resist damage due to freezing and thawing because the porous, compliant fiber nodules act as stress release sites during freezing and thawing.

(iii) The relative modulus decreases with exposure to hot water, the largest reduction being for the plain mortar specimens. High fiber volume composites also experience a reduction in relative modulus on continuous immersion in hot water. The ultimate values of specific damping capacity, ξ for the high fiber volume specimens after 100 cycles are essentially the same as when the freezing and thawing process was started, indicating that freezing and thawing induces no significant damage in high fiber volume specimens. The ultimate values of ξ for specimens immersed in hot water is higher than the ξ at the start for all the samples. These also indicate that immersion in hot water damages the composites.

(iv) For all fiber volumes, the normalized moduli ($E_{\text{damaged}}/E_{\text{undamaged}}$) at the end of the exposure periods for the specimens subjected to freezing and thawing were seen to be higher than those subjected to immersion in hot water indicating higher degree of damage for the latter case, contrary to the results from the flexural strength test results.

(v) The variation in loss tangent is more conspicuous than the variation in elastic modulus as seen from their respective plots against fiber volume and the stiffness-loss map for composites. This shows that the damping characteristic (tan δ or ξ) is more sensitive to damage than stiffness.

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