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Aging mechanisms in cellulose fiber reinforced cement composites

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Abstract

This paper examines the effects of laboratory scale accelerated aging exposures on the changes in physical and mechanical properties of commercially produced cellulose fiber reinforced cement composites. Two different accelerated aging methods were used to simulate the possible aging mechanisms for which the material may experience under service conditions, both methods being compared to material naturally weathered for 5 yr in roofing. The first aging method consisted of different cycles of water immersion, carbonation, and heating exposures whereas in the second method, cycles of water immersion, heating and freeze–thaw exposures were used. The porosity, water absorption, permeability of nitrogen and compressive shear strength of the composites were examined before and after aging exposures. The surface morphologies of the composites fractured in compression shear tests were examined using scanning electron microscope. Experimental results showed that the compressive shear strength of the accelerated aged composites were related to the microstructures within the composites. Both natural weathering and accelerated aging in CO_2 environment reduced the porosity, water absorption, and nitrogen permeability in the cement matrix, and enhanced the durability of the cellulose fiber–cement composites. The aging test based on artificial carbonation was more effective in simulating natural aging performance of the composites, while the freeze–thaw cycling method failed to induce significant aging effects on the composites even after 21 cycles.

Keywords: Cellulose fiber; Cement; Carbonation; Freeze–thaw; Porosity; Permeability

1. Introduction

Over the past few years, lignocellulosic fibers have received considerable attention in the development of asbestos-free fiber reinforced cement composites [1–21]. Much effort has been devoted to wood fibers because they possess many advantages relative to asbestos in terms of availability, lower cost, simple production processes for making cementitious composites of various shapes, renewability and recyclability, non-hazardous nature, and biodegradability [2–7,16–21]. Coutts and coworkers [4,7] found that chemically pulped wood fibers could successfully replace asbestos fibers as the reinforcement in fiber-cement building products. The replacement of asbestos with comparable cellulosic fibers provided cost savings without reducing the mechanical properties of the composite. These fibers tended to increase the tensile and flexural strengths and toughness of the hardened composites, the extent of the improvement being dependent on the fiber content [17–21] and length [8]. However, problems may arise connected to fiber deterioration in a highly alkaline cement matrix and to the influence of high outdoor humidity [14,15,22–24]. Therefore, it is necessary to understand the long-term behavior of the cellulose fiber reinforced cement composites due to the alternations of wet and dry periods of climatic changes occurring in natural conditions.

The long-term behavior of materials due to environmental influences can be evaluated by the real time observations of the materials exposed to natural conditions for several years [25]. However, research programs lasting 10 or more years are rare for organizational and economic reasons. Accelerated aging tests seem to be useful for this purpose.

A great deal of research has been conducted over the past decade to understand the aging mechanisms involved in lignocellulosic fibers reinforced cement

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composites [10–16,21–24,26]. The durability of cellulose fiber–cement composites has been found to be directly related to the fiber types, fiber content, matrix types, and the aging methods [10–16,21,26].

The composites reinforced with fibers containing large amounts of lignin are more sensible to natural weathering than those with negligible amounts of lignin. This is because lignin and hemicelluloses existing in the middle lamellae of wood fiber are more susceptible to alkali attack and chemical degradation in the alkaline cement than cellulose [14–16,22–24]. However, the chemical degradation of lignocellulosic fibers in the alkaline cement matrix can be prevented by reducing the alkalinity of the matrix with partial substitution of cement with silica fume, by treating the fibers with water repellant agents, or using cellulose pulps obtained by chemical processes [10,12–16,22–24].

Natural weathering and accelerated aging in a CO_2 environment, on both naturally and autoclaved cure samples, have been reported to improve the flexural strength and rigidity of the cellulose fiber–cement composites. The enhancement in properties was attributed to the carbonation of the cement matrix [10–13,15]. Using statistical methods of analysis of variance, Marikunte and Soroushian [16] showed that composites containing softwood kraft pulp and hardwood kraft pulp produced comparable flexural strength and toughness. However, both properties increased as the fiber content increased in the composites, regardless of the fiber species. Accelerated wetting–drying and hot water-soaking for 25 days did not significantly (at 95% level of confidence) improve the flexural strength whereas the toughness was significantly reduced. Similar results were also reported by other investigators [11,26].

Much attention has been paid to study the long-term performance of cellulose fiber–cement composites from the point of view of durability related to mechanical properties [10–16,21–24,26]. Although physical properties such as permeability and porosity of cement matrix have been shown to provide a good measure of durability [25,27–32], relatively little in-depth examination of durability of cellulose fiber–cement composites in terms of physical properties has been performed. Specifically, research has not been extensively carried out to evaluate the extent to which the accelerated aging methods alter the physical properties of the cellulose fiber–cement composites, and is thus the focus of this study. The main objective of this paper is to evaluate the long-term performance of cellulose fiber reinforced cement composites through accelerated aging tests. Two different methods of accelerated aging are used to assess how the physical and mechanical performances of the material change, with both methods being compared to material that was removed from service after 5 yr of natural exposure.

2. Experimental

2.1. Materials

Commercially produced, autoclaved cellulose fiber reinforced cement sheets were obtained from a Canadian building material distributor. The sheets contained approximately 8% of bleached Kraft pulp, 54% Portland cement, and 38% silica fume. These sheets were estimated to be 5 months old at the beginning of the accelerated aging tests and were supplied as 2.44 m × 1.22 m × 9.53 mm with a flat dimension of 2.44 × 0.53 m and a corrugated dimension of 2.44 × 0.69 m. Samples were removed from the flat portion of the sheet for all experiments. The densities of the material were determined to be approximately 1.4 g/cm^3 for dry samples and 1.8 g/cm^3 for saturated samples (ASTM C1185). On the other hand, the sheets removed from service after 5 yr of use were supplied as 0.31 m × 0.15 m × 10 mm flat sheets [9].

2.2. Specimen preparation

Specimens were cut with a diamond saw to specified dimensions and stored in a controlled atmosphere of 50 ± 5% relative humidity and 21 ± 2°C for at least a week prior to testing. The dimensions of specimen used for compression shear test are illustrated in Fig. 1. The dimensions of the samples for the water absorption tests were 101.6 × 101.6 mm whereas those used for the pore size distribution were broken down into 6.35 × 19.1 mm to allow them to fit into the sample chamber. The

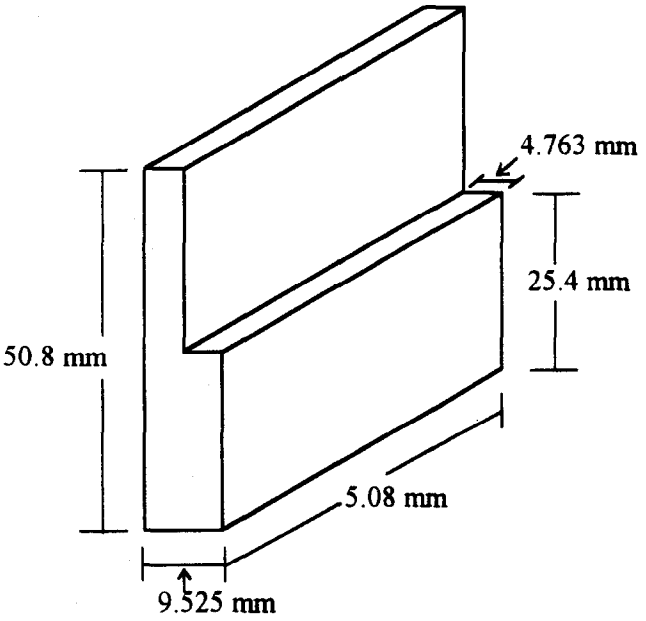

Fig. 1. Schematic and dimensions of samples used for compression shear test.

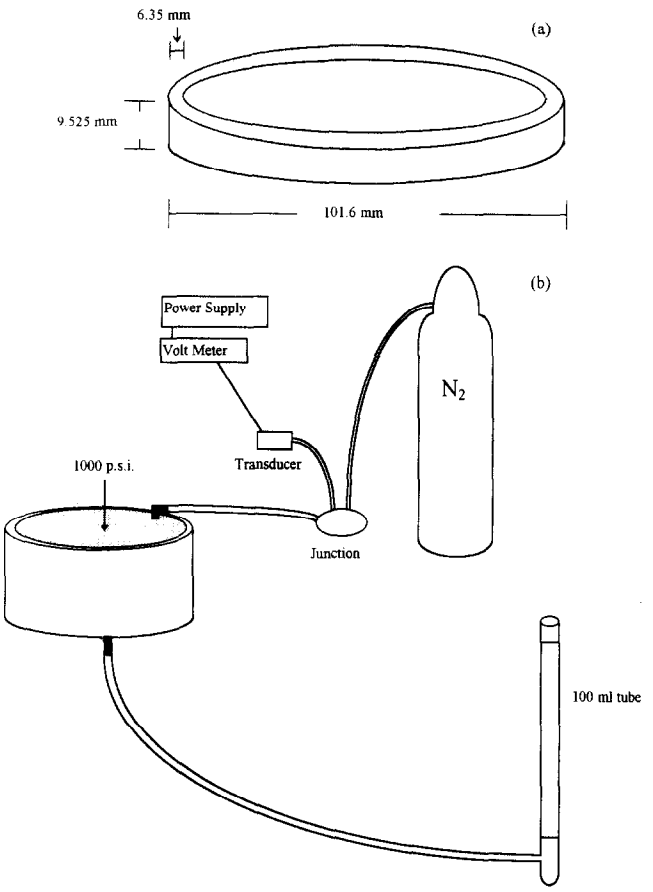

Fig. 2. Schematic of (a) the sample and (b) experimental set-up used for measurement of permeability of nitrogen in the composites.

samples for nitrogen permeability tests were cut into 101.6 mm diameter circles using a diamond-coring bit. The outside edge 6.35 mm thick was coated with an epoxy to prevent any nitrogen gas from permeating the samples from underneath the stabilizing rings [Fig. 2(a)].

2.3. Accelerated aging methods

Because the simulation of natural process is far from perfect and all these forecasts are uncertain, it is essential to combine accelerated tests with real time observation [25]. Since no single accelerated test can fully simulate the full range of aging processes, two different accelerated aging procedures were used to simulate the possible mechanisms which the materials may experience. For both methods, samples which were removed from service after 5 yr of use and those not exposed to any aging condition were used for comparisons.

2.3.1. Accelerated carbonation method

This method was designed to assess the changes in physical properties after exposure to cycles of heat aging, water soak, followed by a saturated carbon dioxide atmosphere. This test was taken and modified from the proposed ASTM standard 'Standard Test Method for the Accelerated Heat and Carbon Dioxide Weathering of Roofing Samples'. The modified accelerated aging consisted of 2-day cycles which included: 16 h immersion of the specimens in distilled water at 20°C, 1 h in a forced draft oven at 80°C, 5 min full vacuum, 7 h in a 0.124 MPa CO_2 environment at 20°C, 16 h immersed in distilled water at 20°C, and 8 h in a forced draft oven at 80°C. The samples were removed after 0, 1, 4, 9, 16, 25, 36 cycles and were subjected to the physical and mechanical property testing.

2.3.2. Cyclic freeze–thaw method

This accelerated aging method consisted of 3-day cycles which included: 24 h immersion of the specimens in distilled water at 20°C, 24 h freezing at −17°C, 2 h thawing at 20°C, and 22 h in a forced draft oven at 80°C [9]. The samples were removed after 0, 3, 7, 14, 21 cycles and subjected to physical and mechanical property testing.

2.4. Property testing

After each removal from accelerated aging tests, all samples were conditioned to 50% relative humidity and 20 ± 3°C until the difference between two consecutive weight readings was less than 0.1%.

Using a mercury intrusion porosimeter, the porosity of the cellulose fiber reinforced cement composites was examined. The pore volume and pore size analysis was performed on a Quantachrome Autoscan-60 Porosimeter. The samples were intruded with mercury and the quantity and pressure required for the intrusion was used to calculate the size and amount of pores within the sample according to the Washburn equation [33]:

$$r = \frac{-2\gamma\cos\theta}{P} \tag{1}$$

where r is the pore radius (μm), γ is the surface tension of mercury (480 mN/m), θ is the contact angle between mercury and the probe wall (usually 140° as recommended by the manufacturer of the porosimeter), and P is the applied pressure (MPa). At least three replications were used for each test.

The water absorption test was performed according to the ASTM standard C1185. At least eight samples were tested.

The apparatus used for the measurement of the permeability of nitrogen is illustrated schematically in Fig. 2(b). The specimens were placed in a pre-cast silicone rubber mold with metal supporting rings (outside diameter of 99 mm and thickness of 6.35 mm) placed on the top and bottom of the samples, with

silicone rubber gasket placed in between. Mechanical pressure of 6.9 MPa was applied to the top of the mold to produce an air tight seal for the sample. Nitrogen gas was introduced into the chamber at a pressure of approximately 0.25 MPa. Due to fluctuations with the regulator on the nitrogen source, the driving pressure of the nitrogen gas was taken to be the average of pressure at the start of the flow measurement and the pressure at the end of the flow measurement [9]. The flow of nitrogen (volume per unit time) through the specimen was measured by calculating the time taken for a soap bubble to travel up a calibrated 100 ml glass tube. From this information, the intrinsic permeability coefficient k of the specimens was calculated using a modified version of Darcy's equation [34]:

$$k = \frac{2Ql\mu P_2}{A(P_1^2 - P_2^2)} \quad (2)$$

where Q is the volume flow rate (m^3/s), l is the thickness of the specimen (m), μ is the fluid viscosity (17.8×10^{-6} Pa for N_2), P_1 is the driving pressure of N_2 (Pa), P_2 is the atmospheric pressure (1.0134×10^5 Pa), and A is the cross sectional area normal to the direction of flow (m^2). At least nine replicates were used for each test.

The compression shear test was carried out on a Tinius–Olsen testing machine in accordance to ASTM D1037. At least nine samples were tested at the crosshead displacement rate of 0.2 mm/min. Scanning electron microscopy (SEM Hitachi Model S-2500) analysis was performed after the specimens were subjected to compression shear tests to examine the quality of the fractured surface of the samples. Small samples were mounted on a stub and were gold coated using a sputter coater (Polatron) for enhanced conductivity.

Analysis of variance (ANOVA) was performed on the collected data with SAS package (SAS/STAT release 6.03). The SAS general linear model procedure was employed to distinguish the statistical differences associated with the aging of the cellulose fiber–cement composites. Mean, standard deviation, and coefficient of variation were computed and Tukey's studentized range test was used to compare means at a 0.05 level of significance.

3. Results and discussion

3.1. Effects of accelerated aging mechanisms on the physical properties of the composites

The porosity in cement and permeability of cement materials to gases are of direct importance to their durability when subjected to adverse environmental conditions: lower porosity and lower gas permeability are generally associated with a more durable product [25,27–32].

The changes in the pore volume (total porosity) and pore size associated with the accelerated carbonation, cyclic freeze–thaw, and naturally weathered cellulose fiber reinforced cement composites are listed in Table 1.

A systematic decrease in porosity in the composites with increasing carbonation-aging cycles was observed, although any significant change was noticed only after the material was aged for at least 16 cycles. However, no significant decrease in porosity was observed above 16 carbonation aging cycles. The decreased porosity of the cellulose fibers reinforced cement composites was attributed to either the densification of the cement matrix resulting from shrinkage brought about by carbonation and/or densification due to continuation of the hydration process within the matrix [30]. Cyclic soaking and drying with exposure to an atmosphere with high CO_2 content used in this accelerated aging method is known to produce quicker carbonation [10–13,15,31]. As a result, the carbonation products fill the matrix structure, increase the density of cement matrix, and reduce the porosity. The typical densification of the cement matrix due to carbonation action is illustrated in Fig. 3(a–c). Detailed SEM analysis of fractured specimens in compression shear test shows a considerable difference in the fiber–cement transition zone. For unaged composites, the transition zone around the fibers was much more open, contained some gaps and some microcracks, and this was accompanied by a greater extent of fiber

Table 1
Porosity of cellulose fiber reinforced cement composites

Number of cycles	Mercury intrusion porosimetry			
	Total porosity		Pore size	
	Mean (%)[a]	TGrp[b]	Mean (μm)[a]	TGrp[b]
Accelerated carbonation method				
0	25.2 (3.5)	A	0.8 (0.2)	A
1	22.7 (2.3)	BA	0.7 (0.1)	BA
4	18.4 (2.7)	BA	0.6 (0.1)	BA
9	17.7 (1.1)	BA	0.6 (0.1)	BA
16	16.8 (0.7)	B	0.4 (0.1)	B
25	16.8 (0.8)	B	0.5 (0.1)	B
36	16.9 (1.8)	B	0.5 (0.1)	B
5-yr old	23.0 (5.6)	BA	0.6 (0.1)	BA
	P-value: 0.0002		*P*-value: 0.0006	
Accelerated freeze–thaw method				
0	25.2 (3.5)	A	0.8 (0.2)	A
3	21.0 (0.4)	A	0.8 (0.1)	A
7	21.6 (1.1)	A	0.7 (0.1)	A
14	22.2 (1.2)	A	0.8 (0.1)	A
21	22.4 (1.6)	A	0.7 (0.2)	A
5-yr old	23.0 (5.6)	A	0.6 (0.1)	A
	P-value: 0.1015		*P*-value: 0000	

[a]Values in brackets represent the standard deviation.
[b]TGrp stands for Tukey grouping and means with the same letter are not significantly different.

pull-out. By contrast, the samples exposed to carbonation (natural aging and accelerated aging in CO_2) showed a very dense and homogeneous cement matrix with intimate contact in the fiber–cement transition zone resulting in a less extent of fiber pull-out [Fig. 3(b) and (c)]. The extremely dense matrix seen in these

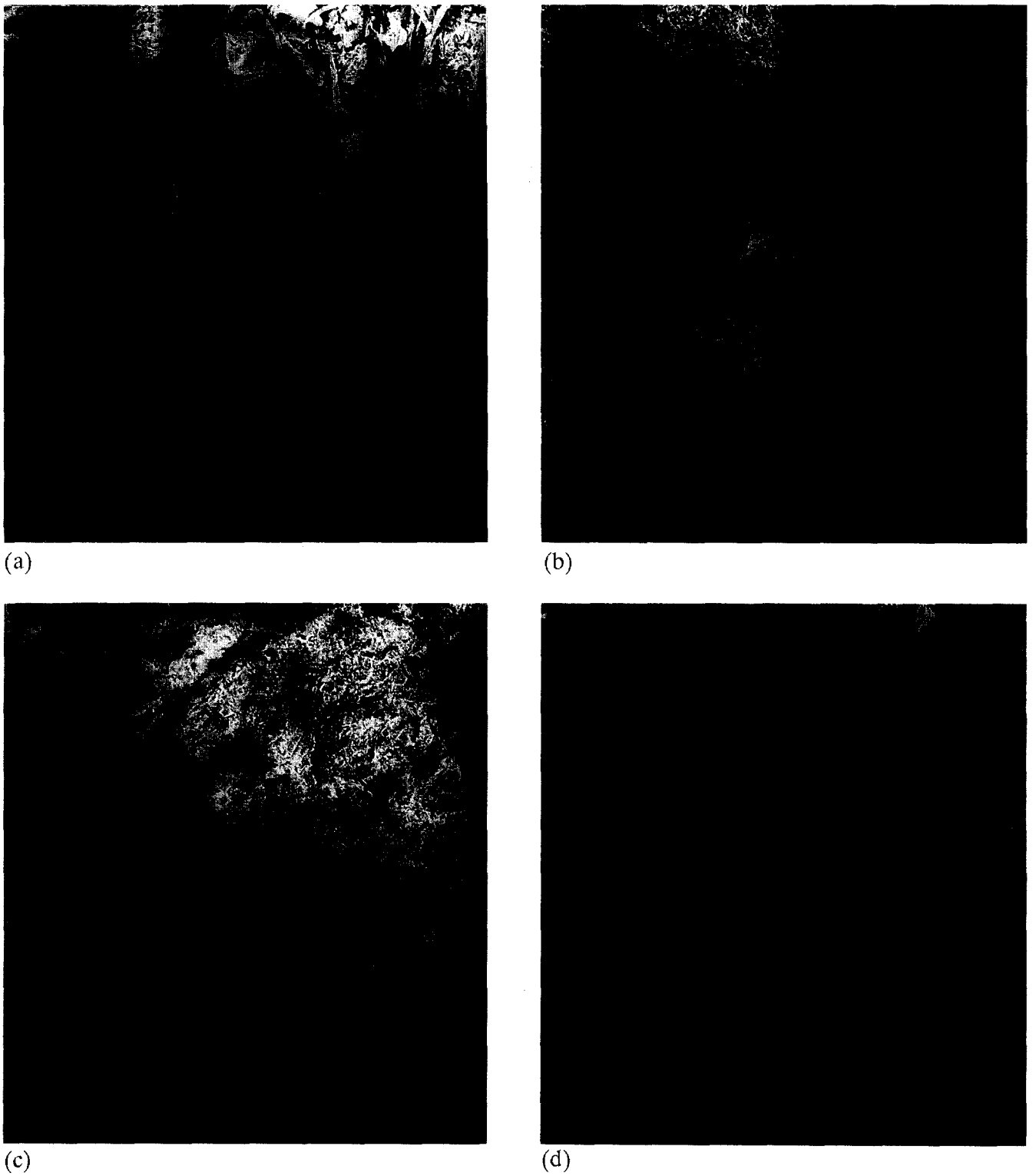

Fig. 3. SEM micrographs of fractured composites in compression shear tests: (a) unaged; (b) 36 cycles of accelerated carbonation; (c) naturally weathered for 5 yr; and (d) 21 cycles of accelerated freeze–thaw.

micrographs suggests that carbonation is an effective means for reducing porosity. It was believed that the porosity of the composites naturally weathered for 5 yr should have been reduced further because of the continuation of carbonation and hydration processes experienced by these specimens throughout their life time. Contrarily to this belief, it appeared from the experimental results that their porosity did not differ from the samples exposed to at least nine carbonation aging cycles, but was significantly greater than the porosity of the samples exposed to at least 16 cycles of carbonation. The high standard deviation exhibited by these specimens may be the cause of the observed results.

Unlike the results of the accelerated carbonation, the changes in the porosity values due to cyclic freeze–thaw aging were not significant at the 5% level (Table 1). It was thought that the freeze–thaw action of this accelerated aging procedure would result in an increase in the porosity due to the formation of cracks that may be brought on by the expansion of the freezing water. However, there actually appeared to be a drop in the total porosity after only three cycles of accelerated freeze–thaw, although not statistically significant. Since this shift was only significant at the 10% level, the confidence placed in the results becomes somewhat diminished.

Associated with the changes in pore volume and pore size are the changes in the amount of water absorbed by the composites (Table 2) and the permeability of nitrogen in the composites (Table 3). Because of the reduced porosity, the ability to absorb water for the composites also decreased with the accelerated carbonation aging cycles, although no significant decrease was noticed until the ninth cycle and thereafter. Similarly, the permeability of nitrogen in the composites decreased with the carbonation aging cycles. It appeared that the composites experienced an immediate reduction in the nitrogen permeability after one cycle of carbonation. However, this reduction was not significantly different from the unaged composites. As in the case of porosity, a significant difference in the nitrogen permeability between the unaged and aged composites was observed at 16 cycles and more of accelerated carbonation. However, the permeation of nitrogen in the composites naturally weathered for 5 yr did not differ from any of the accelerated and unaged samples.

The water uptake results (Table 2) showed that changes associated with both the accelerated cyclic freeze–thaw aging and naturally weathered composites also were not significantly different at the 5% level. However, a different trend was observed for the nitrogen permeability for the composites exposed to the accelerated freeze–thaw method. Almost immediately after the composites were exposed to the accelerated aging cycles, their nitrogen permeability significantly increased (Table 3). This increased nitrogen permeability remained significantly higher for the duration of the aging cycles. It might be possible that the material experienced internal cracking early in the experiment attributed to the freeze–thaw action. However, if this was the case, it would also have been reflected in the mercury intrusion porosimetry results, for which it was not [Fig. 3(d)]. The permeation of nitrogen in the composites naturally weathered for 5 yr was not significantly different from

Table 2
Amount of water absorbed by the cellulose fiber reinforced cement composites due to aging mechanisms

Water absorption (%)					
Accelerated carbonation method			Accelerated freeze–thaw method		
# Cycles	Mean	SD[a]	# Cycles	Mean	SD[a]
0	22.2	(2.5)[c]	0	22.2	(2.5)
1	23.3	(3.0)	3	23.6	(0.7)
4	19.2	(2.7)[b]	7	23.4	(2.6)
9	15.0	(2.1)[bcd]	14	24.9	(2.3)
16	13.9	(2.2)[cd]	21	24.1	(1.7)
25	15.1	(2.0)[cd]	5-yr old	19.2	(1.3)[d]
36	16.7	(2.3)[cd]			
5-yr old	19.2	(1.3)[d]			
P-value: 0.0001			*P*-value: 0.0891		

[a] SD = standard deviation.
[b]Denotes a significant change between the recorded value and the value directly above, $\alpha = 0.05$.
[c]Denotes a significant change between the unaged samples (0 cycle) and the others, $\alpha = 0.05$.
[d]Denotes a significant difference between the 5-yr old samples and the others, $\alpha = 0.05$.

Table 3
Intrinsic permeability of nitrogen in cellulose fiber reinforced cement composites

Intrinsic permeability of nitrogen (10^{-17} m^2)			
Accelerated carbonation method		Accelerated freeze–thaw method	
Number of cycles	Mean[a]	Number of cycles	Mean[a]
0	12.6(1.8)[c]	0	12.6(1.8)[c]
1	10.2(2.5)	3	28.7(13.9)[bcd]
4	14.5(4.6)	7	26.1(7.1)[cd]
9	12.4(6.1)	14	27.7(8.0)[cd]
16	6.9(3.2)[bc]	21	37.4(1.3)[cd]
25	7.1(1.8)[c]	5-yr old	9.0(1.3)[d]
36	7.2(1.1)[c]		
5-yr old	9.0(1.3)		
P-value: 0.0161		*P*-value: 0.0005	

[a]Values in brackets represent the standard deviation.
[b]Denotes a significant change between the recorded value and the value directly above, $\alpha = 0.05$.
[c]Denotes a significant change between the unaged samples (0 cycle) and the others, $\alpha = 0.05$.
[d]Denotes a significant difference between the 5-yr old samples and the others, $\alpha = 0.05$.

that of the unaged material but significantly lower than all those artificially aged. Therefore, the moisture stressing of the material brought about by this accelerated aging method was not effective in altering the porosity of cellulose fiber reinforced cement composites.

3.2. Effects of accelerated aging mechanisms on the compressive shear property of the composites

The changes in compressive strength due to the accelerated and natural aging of the composites are listed in Table 4. The compressive shear strength of the composites increased significantly as the composites were exposed to a greater number of accelerated carbonation aging cycles. This increase was not significant until at least nine aging cycles were completed. However, after the ninth aging cycle, all subsequent accelerated carbonation of the composites exhibited significantly greater compressive strengths than unaged composites. The increased compressive strength of the cellulose fibers reinforced cement composites was attributed to the reduction in porosity of the composites combined with the increased density of the matrix at the interface due to continuing hydration and carbonation [25,30,32]. A denser matrix in the fiber matrix transition zone leads to lower porosity and a higher bond, resulting in higher strength. The effect of lower porosity on increasing the compressive strength of reinforced cement composites can be evaluated from simple application of the rule of mixture. By assuming that the compressive strength is proportional to the cross-section of the solid material, the compressive strength of the fiber reinforced cement composites can be related to the porosity of the composite by [35]:

$$f_c = f_o\left(1 - 1.2P^{2/3}\right) \tag{3}$$

Table 4
Compression shear strength of cellulose fiber reinforced cement composites

Compression shear strength (MPa)					
Accelerated carbonation method			Accelerated freeze–thaw method		
# Cycles	Mean[a]	TGrp[b]	# Cycles	Mean[a]	TGrp[b]
0	2.1(0.7)	B	0	2.1(0.7)	B
1	2.6(1.1)	BA	3	2.0(0.5)	B
4	2.8(1.0)	BA	7	2.0(0.6)	B
9	3.7(1.0)	A	14	2.4(0.3)	B
16	3.7(1.3)	A	21	2.6(1.2)	B
25	4.0(1.3)	A	5-yr old	3.9(1.1)	A
36	3.4(0.5)	BA			
5-yr old	3.9(1.1)	A			
P-value: 0.0004			*P*-value: 0.0858		

[a]Values in brackets represent the standard deviation.
[b]TGrp stands for Tukey grouping and means with the same letter are not significantly different.

where f_c is the composite strength of a material with a system of pores, f_o is the strength of a solid material, and P is the porosity. The above relationship suggests that the total porosity is of greater importance in controlling compressive strength of the composites: a lower porosity being consistent with higher strength. Therefore, natural weathering and accelerated aging with CO_2, which reduce the porosity and nitrogen permeability of the cement matrix, make the cellulose fiber–cement composites better than unaged samples.

On the other hand, a different trend was obtained for the specimens exposed to the accelerated freeze–thaw aging method. Except for the specimens naturally aged for 5 yr, no significant difference was observed between the unaged and artificially aged samples (Table 4). The higher compressive strength property of naturally weathered composites compared to those aged by freeze–thaw action could be attributed to CO_2 exposure for an extended period of time which caused carbonation shrinkage leading to a denser product that is capable of handling a greater compressive load. It appeared that exposure to carbon dioxide was more effective than freeze and thawing in simulating natural aging conditions.

4. Conclusions

On the basis of the data collected in the series of experiments, it can be concluded that substantial reduction in porosity, water absorption, and nitrogen permeability in cellulose fiber reinforced cement composites can be achieved by accelerated carbonation aging tests. This results in a denser matrix, and a higher bond at the fiber-matrix transition zone, thereby contributing to increased compressive strength. Both natural weathering and accelerated aging in a CO_2 environment enhanced the durability of the cellulose fiber–cement composites. Unlike the accelerated carbonation cycles, the testing of the cellulose fiber reinforced cement composites subjected to freeze–thaw cycles did not produce any aging effects similar to those caused by natural weather. Therefore, the aging test based on artificial carbonation seems to be more suitable for simulating the aging performance of the cellulose fiber reinforced cement composites than the freeze–thaw cycling.

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