Cement & Concrete Composites 41 (2013) 34–44

Contents lists available at SciVerse ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Durability and microstructure analysis of CO_2-cured cement-bonded wood particleboard

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ARTICLE INFO

Article history:
Received 10 August 2011
Received in revised form 17 April 2013
Accepted 25 April 2013
Available online 4 May 2013

Keywords:
Wood particles
Cement composites
Carbonation
Flexural strength
Durability
Microstructures

ABSTRACT

Dry-processed cement-based wood composites are reconstituted wood products with desirable longevity, fire resistance and life cycle cost. In this study, the effects of accelerated aging on the performance of CO_2-cured cement-bonded wood particleboards were investigated. The accelerated aging conditions considered simulated natural aging phenomena. Repeated wetting–drying and freezing–thawing cycles led to increased stiffness and somewhat reduced toughness. X-ray diffraction and thermogravimetric analyses indicated that aging effects led to increased $CaCO_3$ and decreased $Ca(OH)_2$ contents in CO_2-cured cementitious composites. Mercury intrusion porosimetry test results indicated that CO_2 curing reduced the capillary pore volume in both unaged and aged boards.

1. Introduction

Cement-bonded wood particleboards are reconstituted wood products with desirable longevity, fire resistance and life-cycle economy. The low productivity of cement-based wood composite production plants, resulting from the slow setting and hardening of cement, has led to relatively high initial cost of the product. Early exposure of cement binders to carbon dioxide (CO_2) can substantially reduce the setting and hardening times of cement based materials [1–3]. The processing and properties of wood–cement composites are sensitive, among other factors, to the specific wood species. Efforts towards substantially increasing the setting and hardening rate of cement should still provide sufficient "open time" during which the wood–cement–water furnish stays plastic to be mixed, formed and pressed.

The predominant chemical reaction occurring with carbonation of cement hydrates involves reaction of CO_2 with the $Ca(OH)_2$ resulting from hydration of cement, yielding $CaCO_3$. Rapid carbonation reactions may reduce the curing time of cement-based materials in the presence of carbon dioxide. This value-added use carbon dioxide also reduces greenhouse gas emissions due to the consumption of the polluting CO_2 [4–7].

The utilization of carbon dioxide gas in accelerated curing of cement-based materials transforms calcium hydroxide into calcium carbonate. Calcium carbonate is less soluble in water than calcium hydroxide, and there is a drop in porosity and an increase in hardness and impermeability associated with the formation of calcium carbonate [8]. These changes in composition and structure enhance the durability characteristics of cement-based materials. CO_2 curing also reduces the alkalinity of pore water in cementitious materials [9,10]. In the case of cement-bonded wood particleboard, the cementitious matrix of reduced alkalinity is more compatible with wood particles. The aim of this paper is to assess the long-term durability characteristics of CO_2 cured cement-bonded wood particleboard subjected to repeated cycles of freeze–thawing and wetting–drying, and to investigate the effects of accelerated aging on the flexural performance characteristics and structure of cement-bonded wood particleboard specimens. The experimental procedures followed BS 5669 [11] and ASTM C1185 [12].

2. Experimental program

2.1. Materials and specimens preparation

A softwood (Southern Pine, S.P.) and hardwood (Aspen) were used in this study. Particles have been made using a hummermill,

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http://dx.doi.org/10.1016/j.cemconcomp.2013.04.014

Fig. 1. Appearance of wood particles.

Table 1
Wood particle dimensions.

	Southern pine	Aspen
Length (mm) means (st. dev.)	5.32 (1.73)	4.97 (1.66)
Width (mm) means (st. dev.)	1.2 (0.67)	1.25 (0.47)
Thickness (mm) means (st. dev.)	0.4 (0.15)	0.5 (0.22)

Table 2
Chemical composition of the Type I Portland cement used.

Chemical composition	% By weight
Tricalcium silicate (C_3S)	43.3
Dicalcium silicate (C_2S)	26.3
Tricalcium aluminate (C_3A)	11.0
Tetracalciumaluminoferrite(C_4AF)	8.6
Insoluble residue	0.12

and then screened to a final of 0.65 to 3.35 mm particle size (passing through No. 6 sieve and staying on No. 20 sieve). The average moisture content of these particles was about 5% (oven dried basis). Fig. 1 shows a picture of wood particle. The average length, width and thickness of wood particles were measured using image analysis procedure and shown in Table 1. Type I Portland cement with chemical composition of Table 2 was used in the mixtures of this investigation. Agricultural grade of calcium hydroxide ($Ca(OH)_2$) at 7.5% by weight of cement was also used when curing was accomplished using CO_2 gas. In this case, calcium hydroxide replaced an equivalent weight of cement.

The manufacturing process of cement-bonded wood particleboard was similar to that used by Soroushian et al. [1]. It involved mixing of the constituents in a mortar mixer, and placing the blend into a 305 mm (12 in.) square wooden box. Wood/cement weight ratio of 0.28 or 0.35, and water/cement weight ratio of 0.25, were used in the process, targeting 12 mm thick boards. The mix was spread in the box between two fine screens, and was then placed in a press, and the wooden box was removed. Fig. 2 shows the cement-bonded wood particleboard processing system for CO_2 curing. To produce various concentrations of CO_2 gas in air, as seen in Fig. 2, two gas cylinders (one CO_2 and the other air) were used. Each cylinder was connected to a flow meter which controlled the gas flow level and thus the CO_2 concentration. A CO_2 gas heating element was used in the CO_2 pressure supply line to prevent the gas from freezing. Pressure was applied on boards using a 50-ton capacity press. In the unsealed press, the platens were perforated with 2.38 mm (3/32 in.) diameter holes drilled in a 13 × 51 mm (0:5 × 2 in.) spacing pattern which covered an area of 305 × 305 mm (12 × 12 in.). The top and bottom perforated plates were connected to the CO_2, air and vacuum lines. The set-up is capable of applying any combination of CO_2, air and vacuum on either side of the board. A constant carbon dioxide (CO_2) gas concentration in air, 25%, was used. A metal screen was used above the bottom plate. Moisture traps were used to prevent any potential damage to the vacuum pump by moisture. Fig. 3 shows the typical appearance of cement-bonded wood particleboard.

The experimental program followed in this paper is intended to provide further insight into effects of wood–cement ratio and wood species on various aspects of board performance

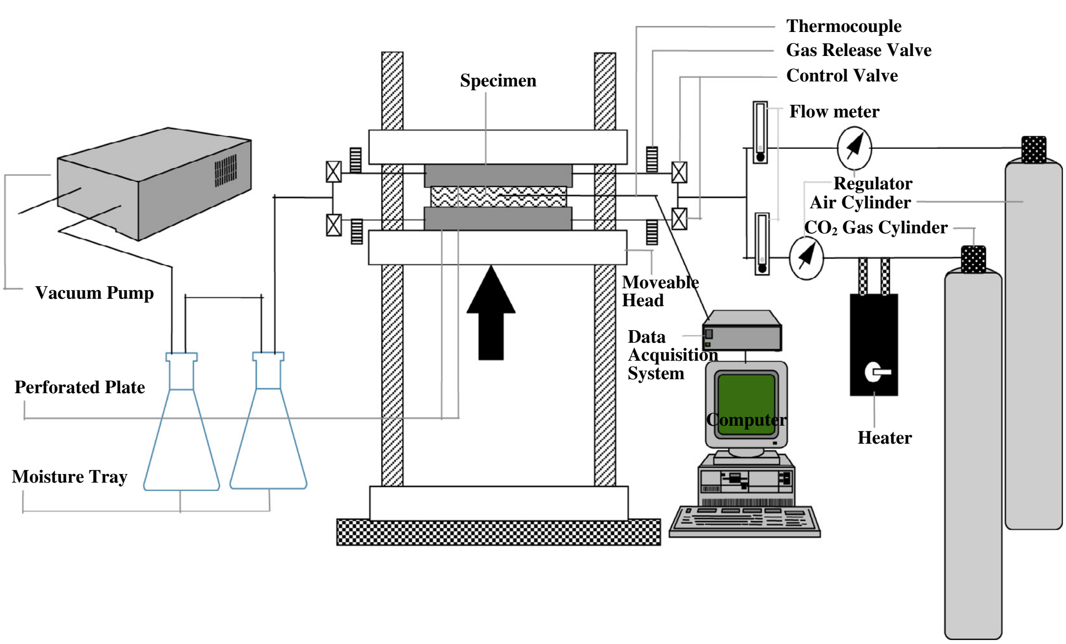

Fig. 2. Processing system incorporating CO_2 curing [1].

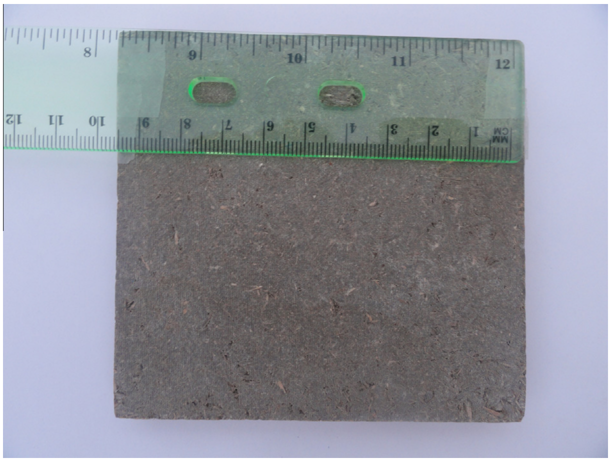

Fig. 3. Typical appearance of cement-bonded wood particleboard.

characteristics. It also concerned the comparison of curing process involving CO_2 with the conventional curing process, and for this purpose control specimens were prepared in the same way mentioned above, but without CO_2 curing.

2.2. Test procedures

2.2.1. Specimens and test procedures

Flexural tests were performed according to the ASTM C 1185. The flexural test samples have a clear span of 254 mm (10 in.), a width of 152.4 mm (6 in.), and a thickness of 12 mm (0.47 in.). A minimum of three replicated specimens were tested for each unaged and aged condition for all mix designs considered. A displacement rate of 2.8 mm/min was used in flexure tests (which were conducted in a displacement-controlled mode). A computer-controlled data acquisition system was used to record the test data. The load–deflection curves were characterized by flexural strength, toughness (total area underneath the load–deflection curve), and initial stiffness (defined here as the stiffness obtained through linear regression analysis of the load–deflection points for loads below 15% of maximum load). The flexural performance was evaluated in wet condition. Testing age period was started immediately after completion of the manufacturing process and extended until the moment of test. It was 28 days for unaged specimens. For aged specimens, testing age was extended to include the aging exposure cycles which will be described in the following Sections 2.2.2 and 2.2.3.

2.2.2. Repeated wetting and drying

Repeated wetting and drying cycles simulated the rain–heat cycles occurring in natural weathering. This aging condition

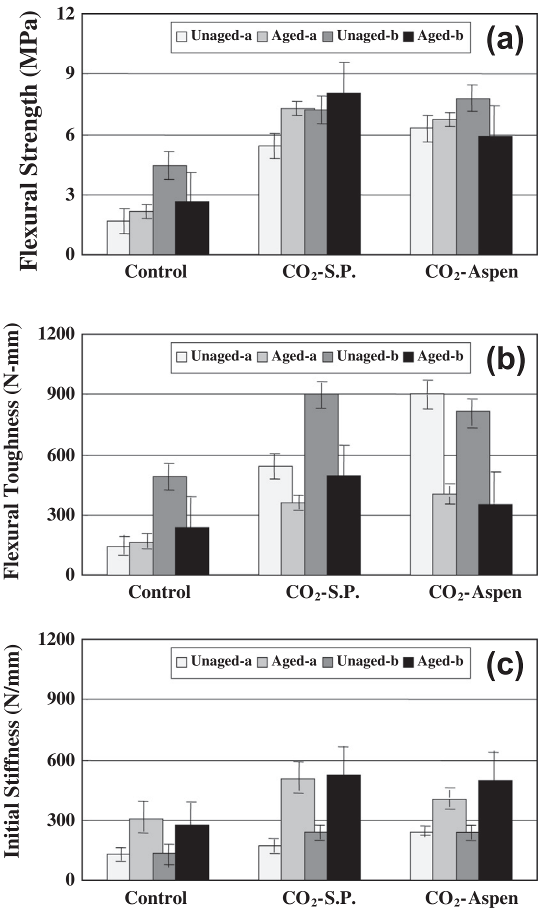

Fig. 5. Effects of repeated wetting–drying cycles on flexural behavior (means and 95% confidence intervals): (a) Flexural strength; (b) flexural toughness and (c) initial stiffness (a–wood/cement = 0.28; b–wood/cement = 0.35).

promotes some key chemical and physical mechanisms of deterioration in wood–cement composites. These conditions accelerate the alkaline pore water attack on wood particles; they also promote migration (through dissolution and re-precipitation) of some cement hydration products from the matrix into the wood fiber cores and their interface zones. A total of 25 wet–dry cycles were applied. These wetting–drying cycles followed the requirements of ASTM C1185.

2.2.3. Repeated freezing and thawing

This test investigates possible degradation of the product due to exposure to repeated freeze–thaw cycles. Freezing of water in the cement paste capillary pores, due to the volume increase of water

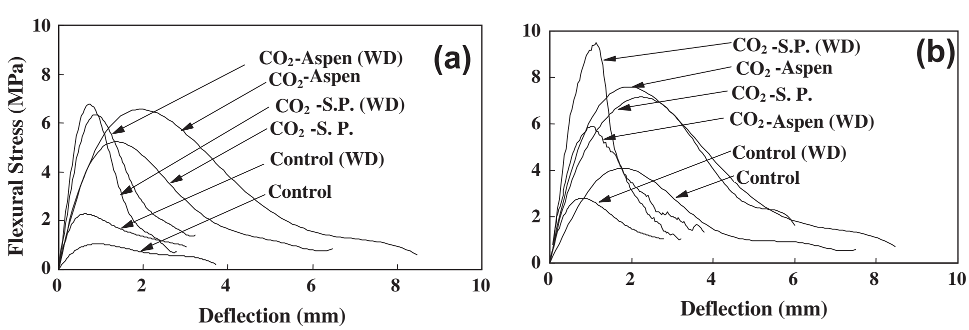

Fig. 4. Effects of repeated wetting–drying on the flexural curve characteristics of cement-bonded particleboard (SP = Southern pine; WD = After wetting–drying); (a) wood/cement = 0.28, and (b) wood/cement = 0.35.

Table 3
Results of analysis of variance after wetting–drying.

	Flexural strength			Flexural toughness			Initial stiffness		
	A	B	C	A	B	C	A	B	C
A	–			–			–		
B	**	–		*	–		**	–	
C	–	**	–	*	–	–	–	**	–

–: Statistically insignificant difference.
A: Wood species; B Aging (wetting–drying) conditions; C: Wood/cement ratio.
* Statistically significant difference at 95% level of confidence.
** Statistically significant difference at 99% level of confidence.

upon freezing, would produce internal pressures which can lead to cracking and deterioration of cement based materials. A total of 25 freeze–thaw cycles were applied to cement-bonded wood particleboard specimens, according to BS 4624 [13]. Before the test cycle, the sample is immersed in water (not less than 5 °C, 41 °F) for 48 h. The specimen is then subjected to alternate freezing and thawing between temperature of 20 ± 3 °C (68 ± 5 °F) and −20 ± 3 °C (−68 ± 5 °F) (each cycle consisting of 12 h of freezing and 12 h of thawing).

A 3 × 3 × 3 factorial design of experiments was implemented, in this phase of study, to investigate the effects of accelerated aging on the flexural performance of CO_2-cured cement-bonded particleboards.

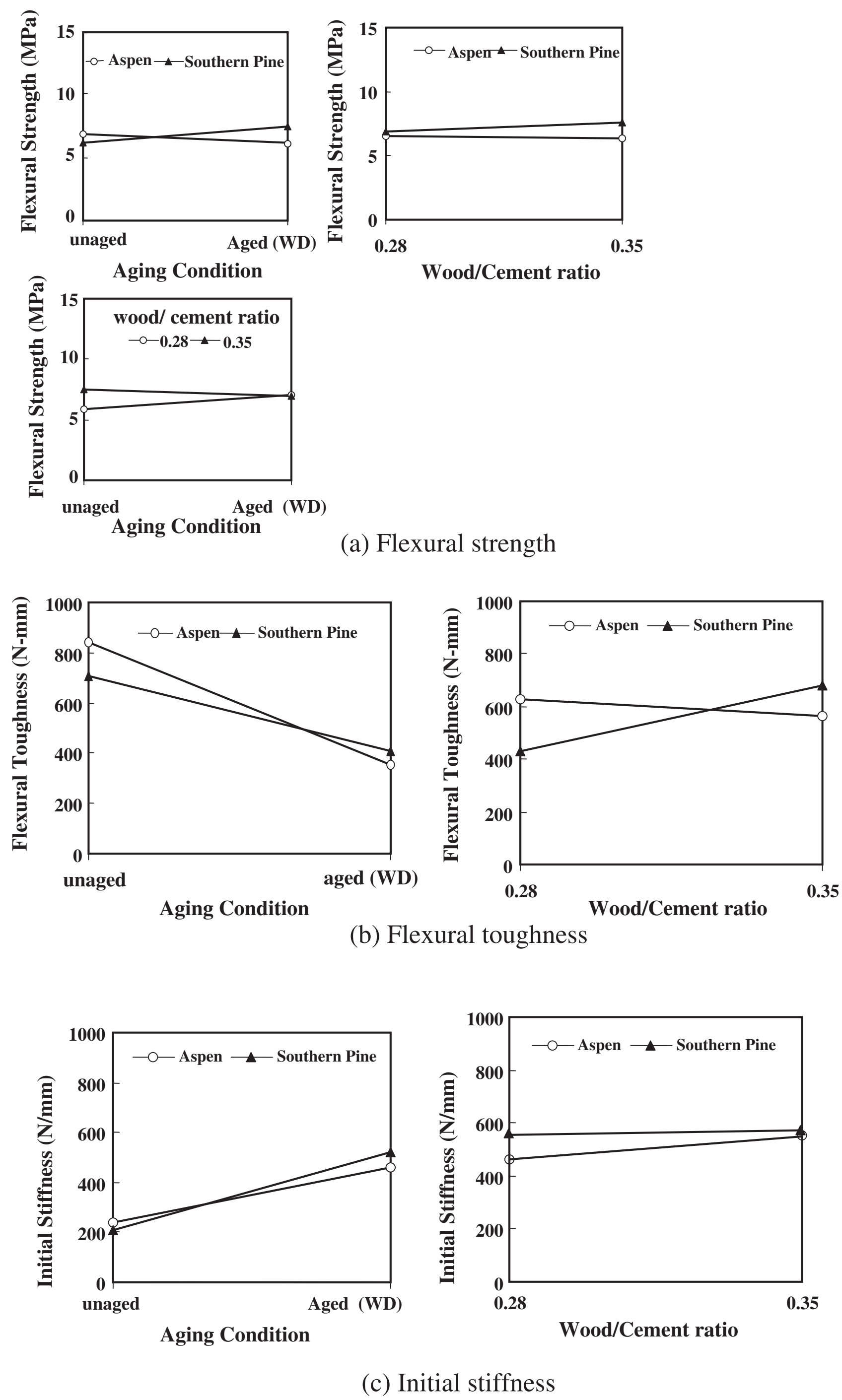

Fig. 6. The trends in flexural performance: CO_2 cured boards (WD = After wetting–drying).

3. Test results

3.1. Repeated wetting–drying cycles

Accelerated wetting–drying tests were performed to study the aging behavior of cement-bonded particleboard. Flexural tests were conducted on composites subjected to 25 cycles of accelerated wetting–drying. The effects of repeated wetting–drying on flexural behavior and flexural test results (strength, toughness, and stiffness), for control and carbonated composites, are shown in Figs. 4 and 5 respectively. In general, repeated wetting–drying cycles lead to increased stiffness and reduced toughness values. The effects on flexural strength are mixed. The flexural performance of CO_2 cured cement-bonded particleboard versus control specimen was generally improved for both aspen and Southern pine. The results were better with wood/cement ratio 0.35 when compared with 0.28. Some researches [14–16] have also shown that the strength of fiber cement composites may experience no change or actually improves with wet–dry cycles.

Statistical analyses of the flexural strength test results for CO_2 cured boards (see Table 3) suggested that wood species (aspen versus Southern pine) and wood–cement ratios had statistically significant interactions with the aging effects on the flexural strength of cement-bonded wood particleboard. The trends in the effects of various factors on flexural strength (Fig. 6a), however suggest that, from a practical point of view, the effects and interactions of wood species, wood–cement ratio and aging on flexural strength are relatively small.

In the case of toughness (Fig. 6b), aging has a definite adverse effect. Other effects and interactions in relation to toughness seem to be of minor practical significance.

In the case of initial stiffness (Fig. 6c), wood–cement ratio had a relatively small but statistically significant effect on stiffness. Aging leads to significant gains in stiffness; the interaction of aging with wood species in relation to stiffness, while statistically significant, seems to be rather small from a practical point of view.

Statistical analysis of the flexural strength test results suggests that curing conditions (CO_2 versus conventional) and wood–

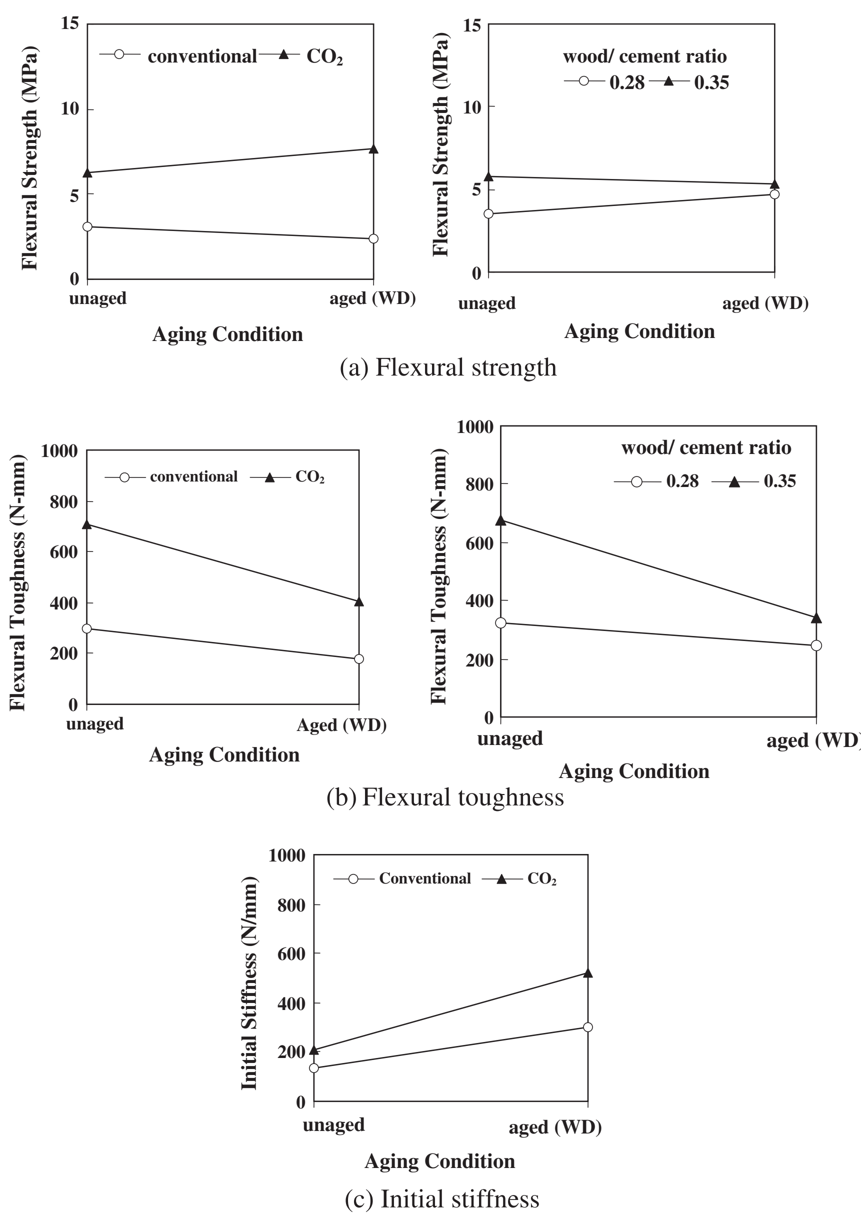

Fig. 7. The trends in flexural performance: CO_2 versus conventionally cured boards (WD = After wetting–drying).

cement ratio (0.28 versus 0.35) had statistically significant interactions with the aging effect on flexural strength. The trends in the effects of various factors on flexural strength (Fig. 7a) suggest that, CO_2 curing not only enhanced the initial flexural strength of cement-bonded particleboard, but also improved the resistance to aging effects on the flexural strength. The effects and interaction of wood–cement ratio and aging in regard to flexural strength were relatively small. In the case of flexural toughness (Fig. 7b), the effects and interactions of curing condition and aging were statistically significant. Flexural toughness was higher with CO_2-curing; aging caused embrittlement in both CO_2 and conventionally cured boards but flexural toughness after aging remained higher in CO_2 cured boards. The adverse effects of aging on toughness were more pronounced at the higher wood–cement ratio of 0.35. In the case of initial stiffness (Fig. 7c), aging leads to significant gains in stiffness; the interaction of aging with curing condition in relation to stiffness was statistically significant; the CO_2-cured boards showed more gain in stiffness upon aging than the conventionally cured boards.

3.2. Repeated freezing–thawing cycles

The boards were tested for flexural performance before and after exposure to freeze–thaw cycles. The effects of repeated freeze–thaw cycles on flexural behavior and flexural test results (strength, toughness, and stiffness), for carbonated and non-carbonated composites, are presented in Figs. 8 and 9, respectively. No cracks were observed in the specimens subjected to repeated freeze–thaw cycles. In general, repeated freezing–thawing cycles led to increased stiffness and reduced toughness of cement-bonded wood particleboard. The effects on flexural strength were mixed but generally negative. Results were better with wood/cement ratio 0.35 when compared with 0.28, particularly for southern pin.

Statistical analysis of the flexural strength test results for CO_2 cured boards (see Table 4) suggests that the effects and interactions of various variables, although statistically significant, are relatively small and of little practical significance. Flexural strength tends to be rather stable under repeated freeze–thaw cycles (Fig. 10a). As shown in Fig. 10b, flexural toughness seriously drops with aging under freeze–thaw effect. This adverse effect of repeated freeze–thaw cycles was more pronounced at the higher wood–cement ratio of 0.35. Fig. 10c indicates that repeated freeze–thaw cycles substantially increase the stiffness of cement-bonded wood particleboard.

Statistical analysis of the flexural strength test results for CO_2 versus conventional curing (Fig. 11a) confirms that flexural strength was rather stable under repeated freeze–thaw cycles. CO_2-curing produces substantially improved flexural strengths before and after the application of freeze–thaw cycles. As shown in Fig. 11b, flexural toughness seriously drops with aging. There was a strong interaction of curing condition and wood–cement ratio with aging. The freeze–thaw effects on toughness were more pronounced at the higher wood–cement ratio and in the case of

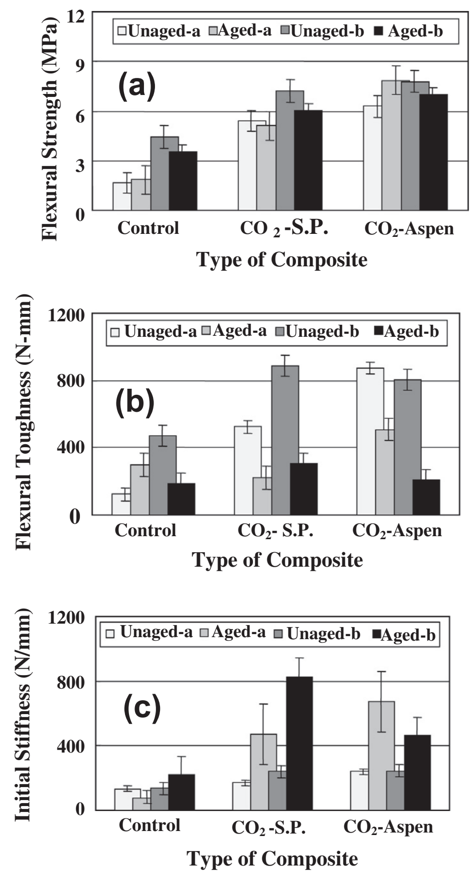

Fig. 9. Effects of repeated freezing–thawing cycles on flexural behavior (means and 95% confidence intervals): (a) Flexural strength; (b) flexural toughness and (c) initial stiffness; (a–wood/cement = 0.28, b–wood/cement = 0.35).

Table 4
Results of analysis of variance after freezing–thawing.

	Flexural strength			Flexural toughness			Initial stiffness		
	A	B	C	A	B	C	A	B	C
A	–			–			–		
B	–	–		*	–		–	–	
C	–	*	–	–	–	–	**	–	–

–: Statistically insignificant difference.
A: Wood species; B: Aging (freezing–thawing) conditions; C: Wood/cement ratio.
* Statistically significant difference at 95% level of confidence.
** Statistically significant difference at 99% level of confidence.

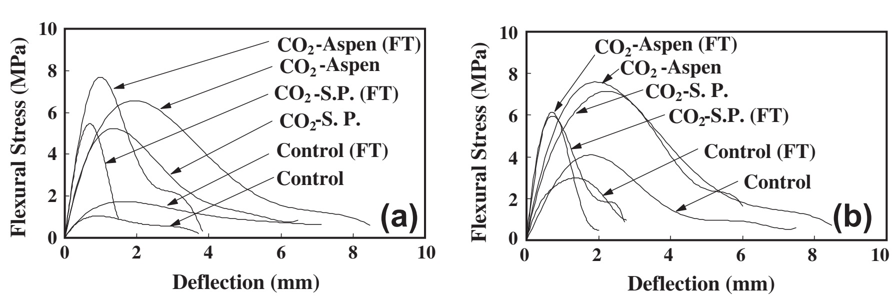

Fig. 8. Effects of repeated freezing–thawing cycles on the flexural curve characteristics of cement-bonded particleboard (SP = Southern pine; FT = After freezing–thawing); (a)wood/Cement = 0.28, and (b)wood/Cement = 0.35.

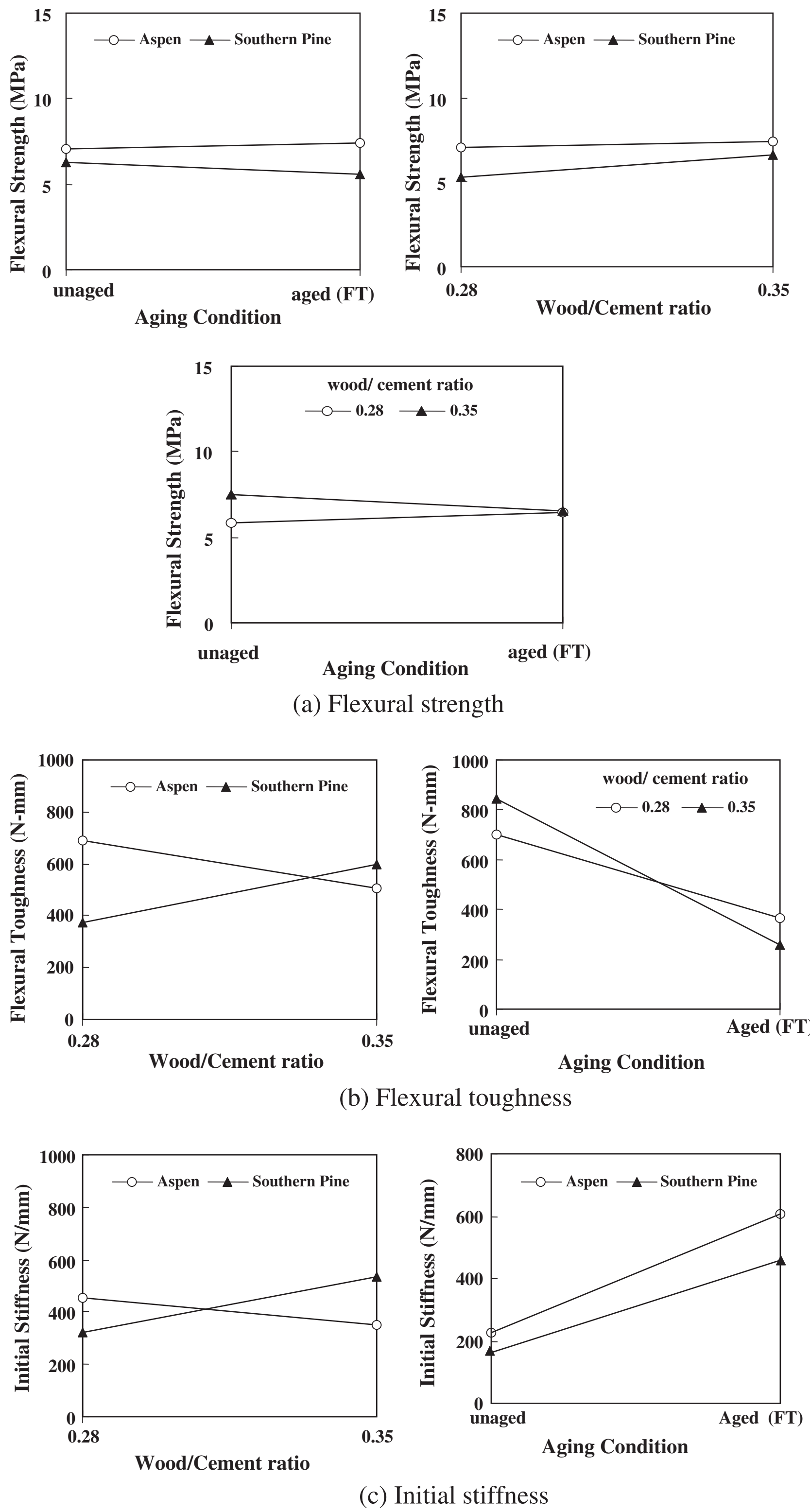

Fig. 10. The trends in flexural performance: CO_2 cured boards (FT = After freezing–thawing).

CO_2-cured boards. Fig. 11c confirm that repeated freeze–thaw cycles substantially increased the stiffness of cement-bonded wood particleboard; the effects and interactions of curing conditions and wood–cement ratio with aging were statistically significant. The increase in the stiffness of cement-bonded wood particleboard was more pronounced when the boards were subjected to CO_2-curing.

4. Microstructural analysis

The aging effects on the microstructure of cement-bonded particleboard which could describe the corresponding effects on material properties have been investigated in three ways: X-ray diffraction, Thermogravimetric analysis (TGA) and Mercury intrusion porosimetry.

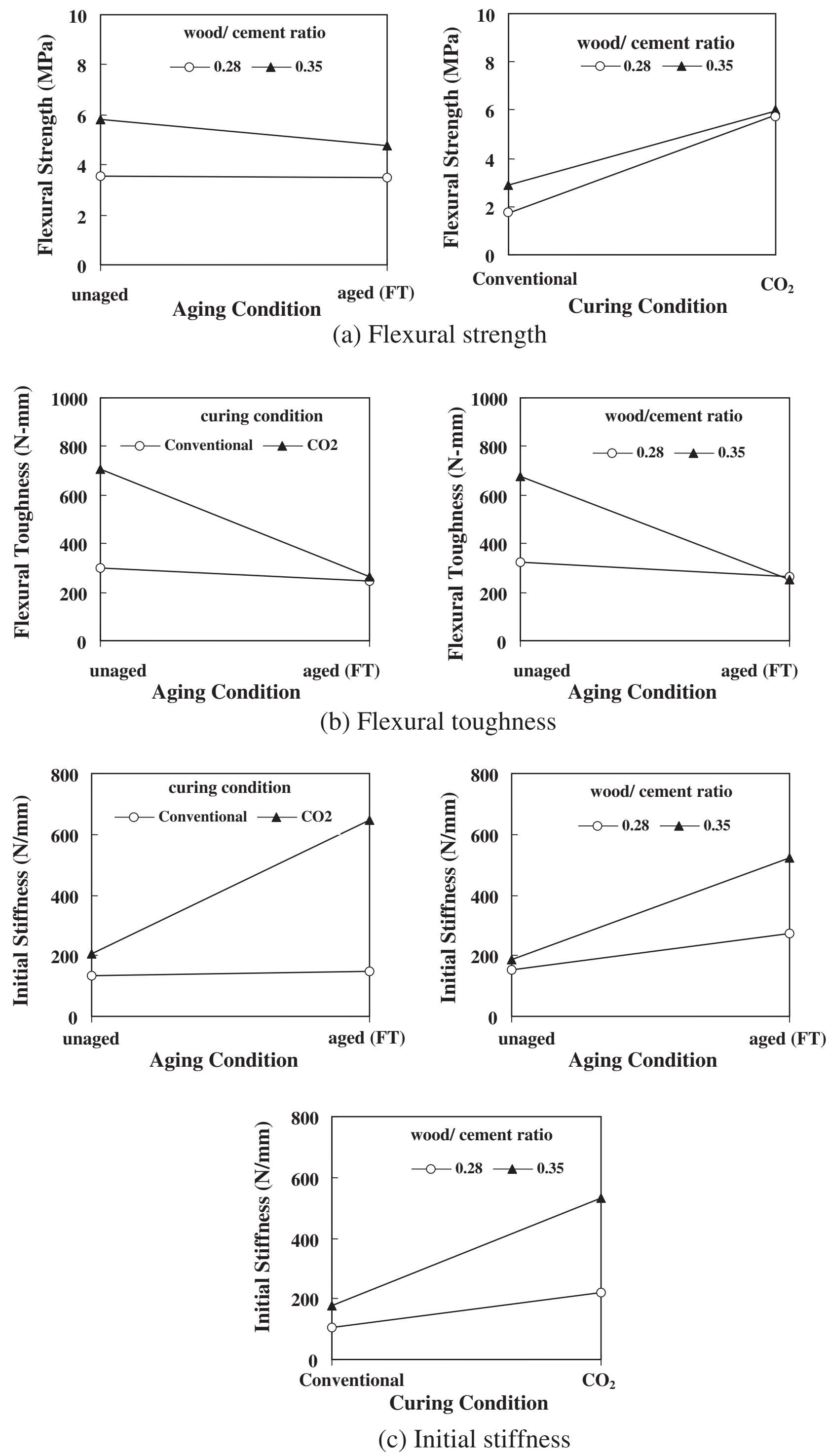

Fig. 11. The trends in flexural performance: CO_2 versus conventionally cured boards (FT = After freezing–thawing).

4.1. X-ray diffraction

Fig. 12 shows the X-ray patterns of unaged composites after 28 days of curing.CO_2-cured composites in the unaged condition had higher $CaCO_3$ contents and lower $Ca(OH)_2$ contents than conventionally cured composites. Composite with different wood–cement ratios performed similarly. The conversion of $Ca(OH)_2$ to $CaCO_3$ during CO_2-curing illustrates the increase in $CaCO_3$ content of CO_2 cured composites. This is consistent with observation of Maail et al. [17], who observed that the application of CO_2-curing could promote the reaction of carbon dioxide to form calcium carbonate ($CaCO_3$), which leads to increased final strength.

After repeated wetting–drying cycles (Fig. 13), conventionally cured specimens exhibited an increase in $CaCO_3$ content and a drop in $Ca(OH)_2$ content, which could be attributed to carbonation in the atmosphere during the aging process. The X-ray patterns for CO_2 cured composites were rather comparable before and after the application of repeated wetting–drying cycles. There was a general drop in $Ca(OH)_2$ content for all composites upon aging. Similar behavior has been observed after repeated freezing–thawing

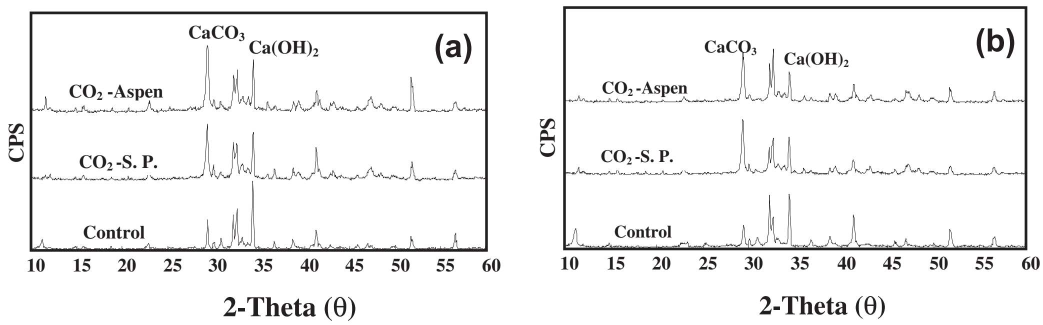

Fig. 12. X-ray patterns for unaged specimens of cement-bonded particleboard after 28-day of curing; (a) wood/cement = 0.28, and (b) wood/cement = 0.35.

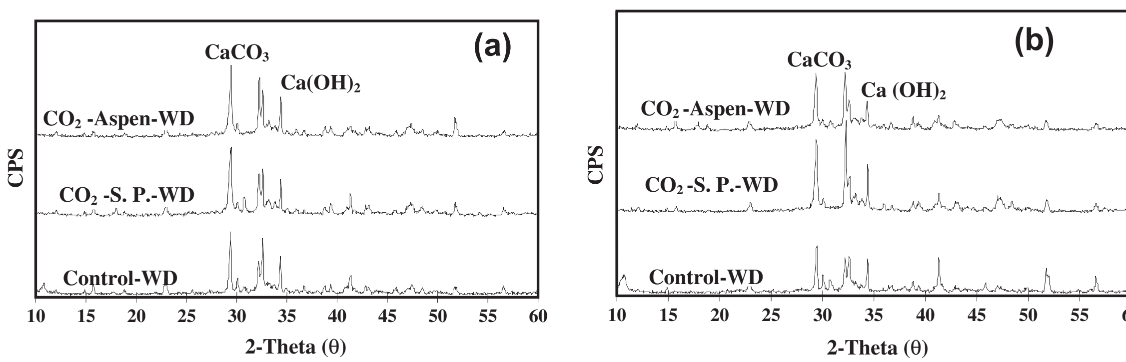

Fig. 13. X-ray patterns after repeated wetting–drying cycles for cement-bonded particleboard; (a) wood/cement = 0.28, and (b) wood/cement = 0.35.

cycles, indicating that such cycles also promoted the carbonation of conventionally cured specimens. CO_2-cured specimens did not go through as much mineral changes associated with carbonation. Aging led to reduced $Ca(OH)_2$ content in all composites. The increased calcium carbonates and decreased calcium hydroxide intensities due to the application of wet–dry cycles observed by Ardanuy et al. [18] support this explanation.

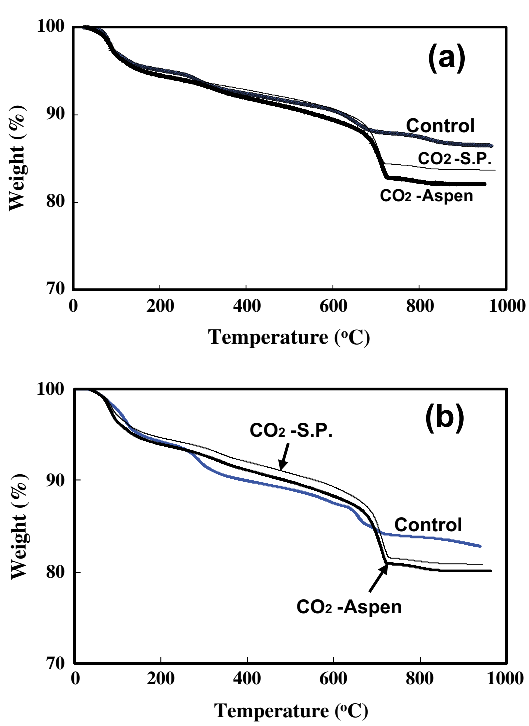

Fig. 14. Thermogravimetric analysis of cement-bonded particleboard after 28 days of curing; (a) wood/cement = 0.28, and (b) wood/cement = 0.35.

4.2. Thermogravimetric analysis (TGA)

The rate of heating was used 208 °C (68 °F)/min and the weight loss with temperature was recorded. Typical weight loss curves of cement bonded particleboard after 28-day of curing are presented in Fig. 14; the $CaCO_3$ and $Ca(OH)_2$ contents may be calculated from these curves. The amount of free $Ca(OH)_2$ and $CaCO_3$ for unaged and various aged particleboards derived from the TGA test results are presented in Table 5. The results suggest that control board, when compared with carbonated boards, show more changes in calcium carbonate content upon aging. The carbonated boards, on the other hand, have high calcium carbonate content prior to aging, which is subject to less change with aging. The additive of

Table 5
Thermogravimetric compositional analysis.

		Control		CO_2-curing			
		Wood/cement ratio					
		0.28	0.35	0.28		0.35	
		Wood species		Wood species			
		SP*	SP*	SP*	Aspen	SP*	Aspen
Unaged	$Ca(OH)_2$	5.10	6.51	12.63	13.12	6.38	7.92
	$CaCO_3$	4.37	5.33	7.646	8.56	7.27	9.11
After W–D	$Ca(OH)_2$	12.23	7.90	13.57	12.19	7.08	3.26
	$CaCO_3$	7.78	6.35	9.050	8.49	8.18	7.77
After F–T	$Ca(OH)_2$	10.98	12.03	13.96	9.99	15.10	8.33
	$CaCO_3$	7.08	7.61	8.36	7.74	11.29	8.95

* Southern pine.

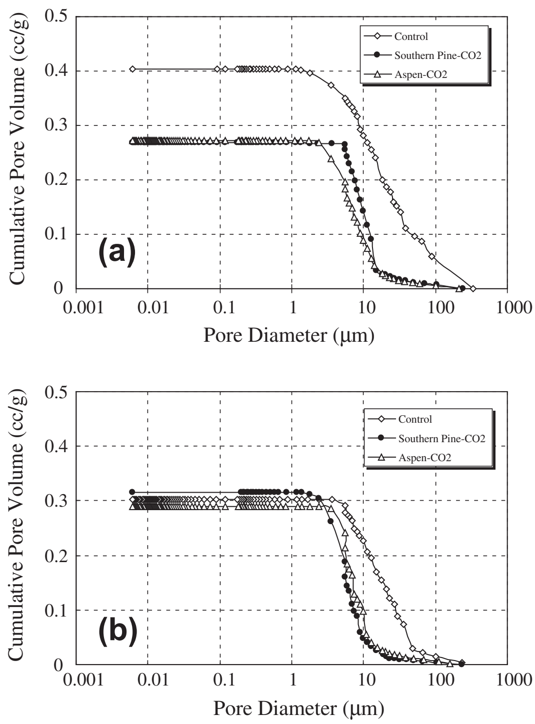

Fig. 15. Pore size distribution of unaged cement-bonded particleboard after 28 days of curing; (a) wood/cement = 0.28, and (b) wood/cement = 0.35.

lime to the carbonated mixes led to increased $Ca(OH)_2$ content of unaged carbonated boards. In the control boards, the gradual release of lime associated with the hydration of cement seems to increase $Ca(OH)_2$ content upon aging.

4.3. Mercury intrusion porosimetry

The pore size distribution curves (in logarithmic scale) after 28 days of curing and after different aging processes were recorded. Typical curves are presented in Fig. 15. It indicates that the general distribution curves shifted to the left (towards lower-diameter pores) in the case of CO_2-cured boards; that is, the pore structure became increasing finer when using CO_2-curing. This is expected because $CaCO_3$ particles produced during carbonation tend to fill some capillary pores. Aging also produces finer pores, which could again be illustrated by carbonation during aging with atmosphere. Table 6 summarizes the measurements on total capillary pore volume of cement-bonded particleboard. Unaged carbonated boards possessed a smaller total pore volume, which could result from the filling of pores with $CaCO_3$. After aging, however, control (uncarbonated) boards show a large reduction in total pore volume and these aged carbonated and uncarbonated boards have more comparable total capillary porosities.

It is interesting to notice that, the detected capillary pores are related to the intruded volume. Young and Diamond, suggest that intrusion of the pore space between the C–S–H gel become less marked in aged specimens, or in case of rich content of cement and low water cement ratio, due to the continued formation of contact points and the growth of calcium hydroxide in the capillary voids occluding the C–S–H gel [19,20]. This suggestion, probably, is the reason behind the undetected pores below about one micrometer (Fig. 15). Mercury porosimetry, however, gives a better appreciation of the large capillary pore system, which has an important influence on permeability and shrinkage at high humidity conditions [21].

4.4. Observation of fracture surfaces

Visual inspection for fracture surfaces provided some indications of wood particle petrifaction in unaged composites subjected to CO_2 curing. Fracture surface of control unaged boards (without CO_2 curing) tailed mainly pulling out the wood fibers. In CO_2 cured boards, however, a combination of wood fiber fracture and pull-out occurrence. This is indicative of stronger fiber-to-matrix bonding in CO_2 cured boards. Accelerated aging (wetting–drying) cycles led to incurred fiber fracture in both control and CO_2 cured boards: This is indicative of improved bonding after exposure to repeated wetting–drying cycles. Freezing–Thawing cycles seem to also improve bonding but the matrix seems to have received some damage after this accelerated aging process.

5. Summary and conclusions

The long-term durability characteristics of cement-bonded wood particleboards were assessed. The effects of accelerated aging on the flexural performance of the CO_2-cured and control composites were investigated. The impact of using CO_2-curing on cement binder phase was evaluated. In general, carbonation curing modifies the matrix phase of cementitious composites. The test results indicated that:

- Repeated wetting–drying and freezing–thawing cycles lead to increased stiffness and reduced toughness values. The effects on flexural strength were mixed.

Table 6
Comparison of pore volumes of unaged and aged cement-bonded particleboards.

	Wood species		Control		CO_2-curing			
			Wood/cement ratio					
			0.28	0.35	0.28		0.35	
			Wood species		Wood species			
	SP*	Aspen	SP*	SP*	SP*	Aspen	SP*	Aspen
Unaged								
Total intrusion volume (cc/g)	0.732	0.971	0.403	0.303	0.267	0.273	0.315	0.290
After repeated wetting–drying cycles								
Total intrusion volume (cc/g)			0.214	0.387	0.180	0.267	0.170	0.289
After repeated freezing–thawing cycles								
Total intrusion volume (cc/g)			0.226	0.197	0.240	0.290	0.295	0.271

* Southern pine.

- The CO_2-cured composites generally have higher $CaCO_3$ and lower $Ca(OH)_2$ contents when compared with the conventionally cured boards. Higher $CaCO_3$ contents usually correlate with higher flexural strength and stiffness, and lower toughness values.
- Aging effects lead to further rise in $CaCO_3$content and drop in $Ca(OH)_2$ contents. Higher $CaCO_3$ contents enhanced the fiber matrix interfaces, and the matrix structure was densified. This effect was more pronounced at the lower wood cement ratio of 0.28.
- CO_2 curing reduces the capillary pore volume in both unaged and aged boards.
- Aging effects generally reduce the pore volume in the case of repeated wetting–drying cycles, but had relatively small effects in the case of freezing–thawing cycles.
- From a practical point of view, the interaction effects of wood species, wood–cement ratio with aging on flexural strength are relatively small.
- Compared with Southern pine, aspen has a lower specific gravity and provides a more porous structure for gas penetration into the product during curing.
- The more porous nature of aspen leads to more pronounced petrifaction of wood particles than Southern pine.

This study demonstrates the beneficial effect of accelerated CO_2 curing on the long-term structure and durability characteristics of cement-bonded wood particleboards composite.

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