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Dede Hermawan · Toshimitsu Hata · Shuichi Kawai
Wakatsu Nagadomi · Yasuo Kuroki

Manufacturing oil palm fronds cement-bonded board cured by gaseous or supercritical carbon dioxide

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Abstract This study dealt with the effects of a curing method that uses gaseous and supercritical CO_2. Its effects on the properties of oil palm fronds cement-bonded board manufactured by the conventional cold-press setting method were recorded. The effect of $MgCl_2$ as an accelerator of cement setting was also investigated. The hydration of cement was examined using X-ray diffractometry, thermal gravimetry, and scanning electron microscopy. The results are as follows. (1) High-performance cement-bonded boards made from oil palm fronds were successfully manufactured using the CO_2 curing method. (2) The curing method using either gaseous or supercritical CO_2 resulted in accelerated curing of cement (within several minutes). Accelerated formation of the hydration products (e.g., calcium carbonate and calcium silicate) is the main reason for the high strength of CO_2-cured boards. (3) The CO_2 curing technology does not require setting accelerators, which cause a decrease in the dimensional stability of cement-bonded board.

Key words Oil palm fronds · Cement-bonded particleboard · Accelerator · Supercritical fluid

Introduction

There is increased awareness of the need for raw materials and sustainability of fiber supply for wood industries. At the same time, forestlands are decreasing with the increasing world population and human activities, natural resources are being used up, and our planet is being polluted. Therefore, new raw materials and technologies that are environmentally friendly are required.

In our previous study,[1] manufacture of oil palm fronds cement-bonded board by the conventional cold-pressing method was reported to be successful when 5.0%–7.5% $MgCl_2$ was added. The addition of $MgCl_2$ improved the compatibility of oil palm fronds with cement, and the properties of the boards improved significantly. However, the production process of cement-bonded board took a long time owing to the slow process of cement hydration; it required at least 14 days to achieve a high degree of curing. Efflorescence also appeared, and the dimensional stability decreased with the increasing amount of $MgCl_2$. This study investigates the utilization of CO_2 to accelerate the curing of cement and to reduce the amount of magnesium chloride used. As been reported previously,[2,3] CO_2 curing enhanced cement hydration, and the board strength properties improved.

The objective of this study was to develop the manufacturing technology of cement-bonded board using fronds of oil palm. The fundamental properties of oil palm fronds cement-bonded board manufactured by the conventional cold-pressing method for setting the cement and being cured with CO_2 at gaseous or supercritical phases were then investigated under various conditions. The degree of cement hydration relative to the mechanism of strength development using CO_2 addition was also investigated.

Materials and methods

Frond vascular bundles of oil palm (*Elaeis guineensis* Jacq) cut to an average length of 15 mm with ordinary portland cement (Osaka Sumitomo) as a binder and $MgCl_2$ as an accelerator of cement setting were used to manufacture cement-bonded board. Boards with a target density of 1.2 g/cm^3 were manufactured at a cement/oven-dried particle/water weight ratio of 2.2:1.0:1.32. The amounts of $MgCl_2$ added were 0%, 2.5%, and 5.0% based on cement weight. Two curing methods were applied: CO_2 gas curing

D. Hermawan (✉) · T. Hata · S. Kawai
Wood Research Institute, Kyoto University, Uji, Kyoto 611-0011, Japan
Tel. +81-774-38-3670; Fax +81-774-38-3678
e-mail: m54298@sakura.kudpc.kyoto-u.ac.jp

W. Nagadomi · Y. Kuroki
Nichiha Company, Nagoya, Japan

and supercritical CO_2 curing. The concentration of CO_2 used was about 95%. Hand-formed mats measuring 300 × 300mm were cold-pressed to a target thickness of 12mm and kept in an oven at 60°C for 24h. The specimens measuring 50 × 210mm cut from these boards were then used for each treatment condition.

For treatment with gaseous or supercritical CO_2, the specimens were placed in a reaction cell surrounded by a water jacket set at 60°C.[1] In this experiment the specimens were subjected to a CO_2 pressure of 1.0MPa in the gas phase and 7.5MPa in the supercritical phase at about 50°C. Following curing times of 10, 30, and 60min, they were placed in an oven set at 80°C for 10h. Later the specimens were conditioned at ambient temperature prior to property evaluation.

The mechanical and dimensional properties of the boards were tested in accordance with the Japan Industrial Standard (JIS) A 5908 (1994). The boards were cut into 50 × 210mm samples for the static bending test and 50 × 50mm for the internal bond (IB) strength, thickness swelling (TS), and water absorption (WA) tests. Three test samples were prepared from each treatment group for the above tests. The static bending tests were conducted using a three-point bending test over an effective span of 180mm (15 times the board thickness) at a loading speed of 10mm/min. The degree of hydration of the composites was examined using X-ray diffractometry (XRD), thermal gravimetry (TG-DTG), and scanning electron microscopy (SEM).

XRD analysis

Powdered samples (passing through 120 mesh size) obtained from the IB test specimen were examined by XRD analysis. Step scan measurements were done using X-rays (Cu-Kα) at 40kV and 40mA, with 2θ ranging from 3.0° to 80.0°, corresponding to scanning speeds of 0.02°/min and 2°/min. The amount of unreacted clinkers taken at 2θ = 32.2° and 32.6°, calcium hydroxide at 2θ = 18.0°, and calcium carbonate at 2θ = 36.0°, 39.4°, 43.2°, and 48.5° were determined and compared with those of the samples.[2–5]

Thermal analysis

Powdered samples passing through 200 mesh size were examined by a thermogravimetric analyzer (TGA 2050; TA Instruments). Thermal degradation of the specimens ranging from room temperature to 1000°C at 10°C/min heating rate and nitrogen flow of 100ml/min was observed. The amounts of calcium hydroxide and calcium carbonate generated were determined.[5–8]

SEM observations

The test specimens for SEM observations were prepared by cutting small sections from the fractured surfaces of the IB test samples. The small samples were mounted on specimen stubs and then coated with gold for examination in a JSM-5310 (Jeol).

Results and discussion

Properties of oil palm fronds cement-bonded boards

Figure 1 shows the effect of $MgCl_2$ content and CO_2 curing conditions on the moduli of rupture (MOR) and elasticity (MOE). The MOR and MOE values of the CO_2-cured boards increased with increasing curing time, and they reached a value similar to that of supercritical CO_2-cured boards after 60min of curing time.

The MOR and MOE values increased to about 25.0MPa and 5.5GPa, respectively, when supercritical CO_2 was used for curing, which showed saturated values over curing times of 10, 30, and 60min. The addition of up to 5% $MgCl_2$ did not affect the bending properties when board was cured with either gaseous or supercritical CO_2. However, with the conventional 2-week curing method the bending properties of boards improved significantly by addition of $MgCl_2$; the MOR and MOE values increased to about 23MPa and 4GPa, respectively, when 5.0%–7.5% $MgCl_2$ was added.[1] In addition, compared to the conventional method, without any accelerators, the MOR and MOE of CO_2-cured boards were three- to fivefold stronger. As reported previously,[1] the compatibility of oil palm fronds with cement is less than 31%, and cement hydration is retarded when fronds are present. Oil palm fronds could not be used solely as raw materials for cement-bonded board owing to their inhibitory effect on cement hydration caused by the inherent extractives of the materials.[9–15]

The average values of IB strength of the boards cured at gaseous and supercritical phases are presented in Fig. 2. As well as bending properties, the IB strength of CO_2 gas-cured boards increased when the curing time increased, reaching high IB values, similar to those of supercritical CO_2-cured board, after 60min of curing.

The high bondability of supercritical CO_2-cured boards was obtained when CO_2 was introduced for 30min, and they were similar to those obtained by the conventional 2-week curing method. However, addition of 7.5% $MgCl_2$ was needed to accelerate the hydration of cement when the board was cured with the conventional curing method.[1]

Figure 3 shows the TS values of the boards after 24h of water soaking. It was revealed that the dimensional stability improved significantly when board was cured with either gaseous or supercritical CO_2. At the same level of $MgCl_2$ addition, the TS values decreased with the longer curing time. However, the dimensional satiability decreased with an increasing amount of $MgCl_2$, especially with gaseous curing.

Addition up to 5% $MgCl_2$ did not affect the dimensional satiability when board was cured with supercritical CO_2. The improvement effected by $MgCl_2$ addition was pronounced when board was cured with the conventional 2-week curing method; the TS values decreased gradually when 7.5% $MgCl_2$ was added.[1]

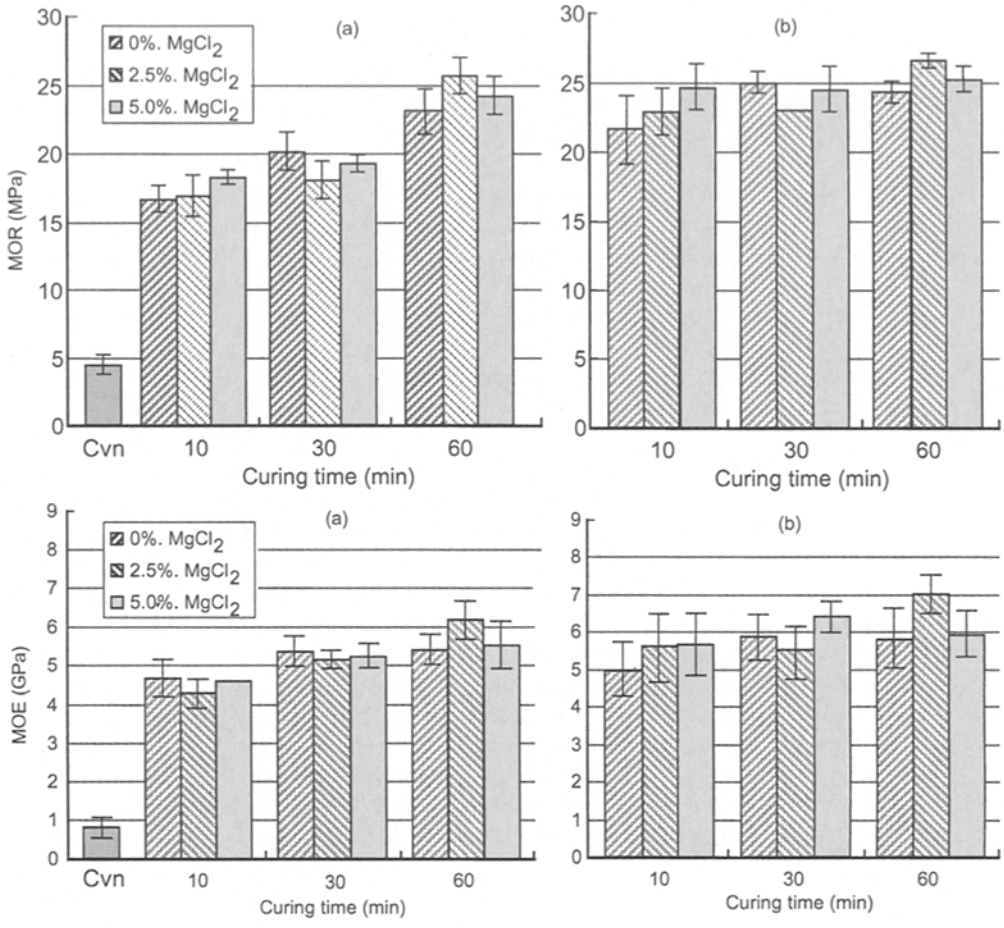

Fig. 1. Effect of $MgCl_2$ and curing time on the moduli of rupture (MOR) and elasticity (MOE). **a** Gaseous CO_2. **b** Supercritical CO_2

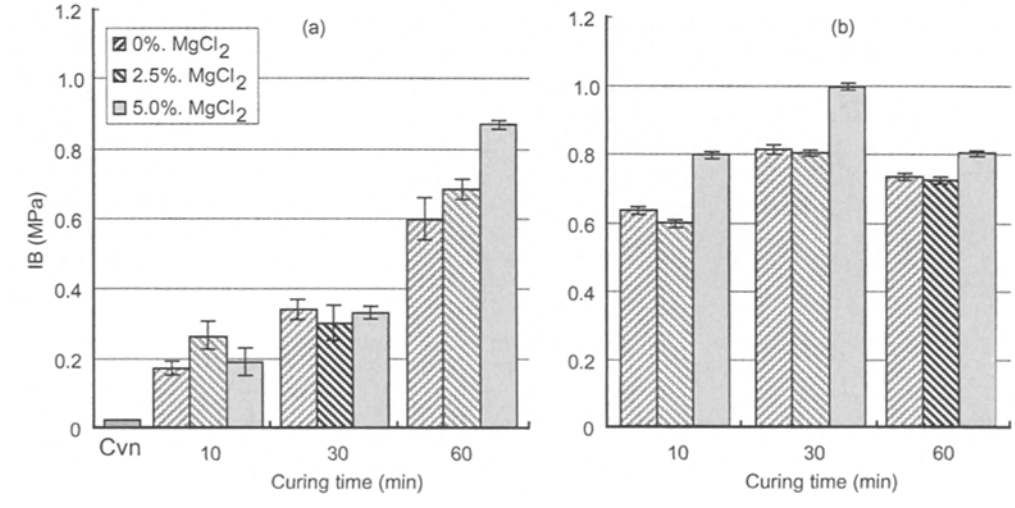

Fig. 2. Effect of $MgCl_2$ and curing time on internal bond strength (IB). **a** Gaseous CO_2. **b** Supercritical CO_2

Improved mechanism for CO_2 curing

The high alkalinity of the cement hydration products, especially calcium hydroxide [$Ca(OH)_2$], can trigger dissolution of the inhibitory extractives of wood. This unsuitable alkalinity could also weaken the strength of natural fibers in cement composites and finally board strength properties.[16]

The CO_2 curing method imparts significantly higher properties and faster curing of cement-bonded boards compared to the conventional curing method. The improved effects of the CO_2 curing method may be due to the suitable alkalinity of cement mixtures caused by the formation of carbonic acid. When CO_2 dissolves in water, hydrogen carbonate (H_2CO_3) is formed, and the pH can drop to about 5.9. Under the influence of such

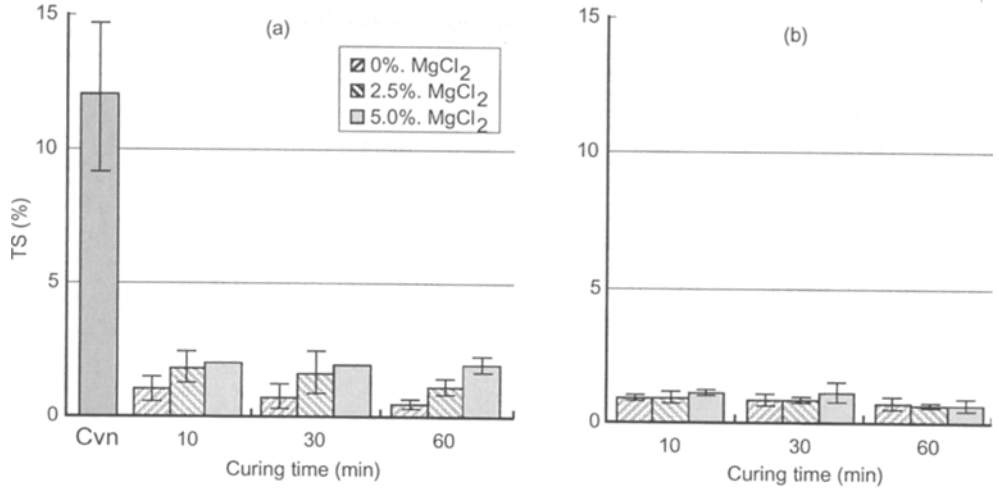

Fig. 3. Effect of $MgCl_2$ and curing time on thickness swelling (TS). **a** Gaseous CO_2. **b** Supercritical CO_2

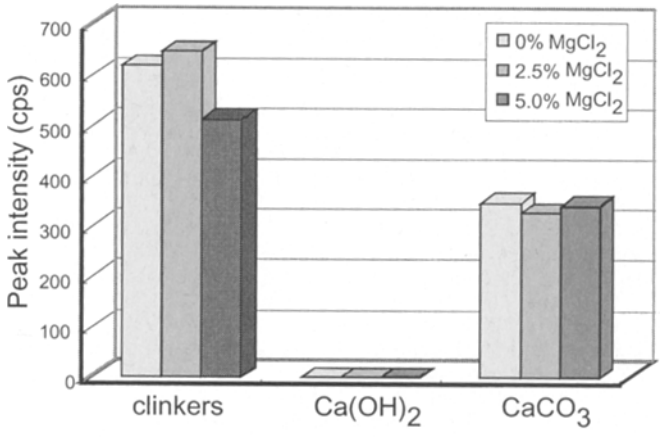

Fig. 4. X-ray diffractometry analysis of supercritical CO_2-cured boards after 60 min of curing

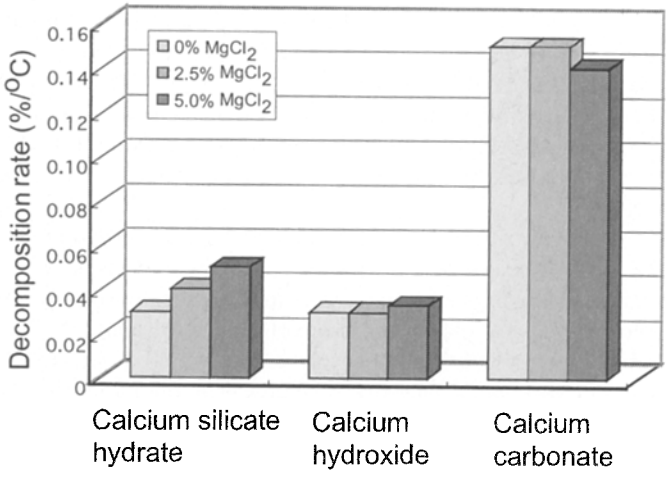

Fig. 5. Effect of $MgCl_2$ on the thermal property of supercritical CO_2-cured boards after 60 min of curing

water, the pH of the hardened cement can be reduced to about 8.[17]

Reduced pH of the hardened cement is also caused by the deformation of $Ca(OH)_2$. In the latter case of cement hydration, under the influence of CO_2 the formed $Ca(OH)_2$ is carbonized to calcium carbonate ($CaCO_3$). Calcium silicate hydrate is also formed during this hydration reaction.[3,17] These phases are mainly responsible for the strength development of cement. Therefore, the superior strength of CO_2-cured boards was promoted by the high strength of both cement and undisturbed fibers.

In general, the addition up to 5.0% $MgCl_2$ for setting the cement did not result in any significant differences in the board properties when the board was cured with CO_2 at either the gaseous or the supercritical phase. The degree of cement hydration was similar at any of the $MgCl_2$ concentrations added, as shown in Figs. 4 and 5. Figure 4 shows the intensities of hydration products of supercritical CO_2-cured boards by XRD analysis. After 60 min of curing, the formed $Ca(OH)_2$ was almost completely carbonized to $CaCO_3$ at all levels of $MgCl_2$ addition.

Figure 5 shows DTG analysis of supercritical CO_2-cured boards. The decomposition rate of $Ca(OH)_2$ was about 0.03%/°C over $MgCl_2$ additions of 0%, 2.5%, and 5.0%. As with $Ca(OH)_2$, the accelerated formation of $CaCO_3$ was similar at all levels of $MgCl_2$ addition, and its decomposition rate was in the range of 0.14%–0.16%/°C.

Figure 6 shows an SEM observation of the fractured surface of the CO_2-cured boards without the $MgCl_2$ accelerator. Calcium carbonate was formed as a result of the hydration of all basic cement compounds. It was mentioned in our previous study and other research work[3,18,19] that these formations are believed to interlock with the calcium silicate hydrate and wood surfaces, resulting in high-strength cement-bonded board.

Conclusions

The mechanical and dimensional properties of oil palm fronds cement-bonded board manufactured by the conven-

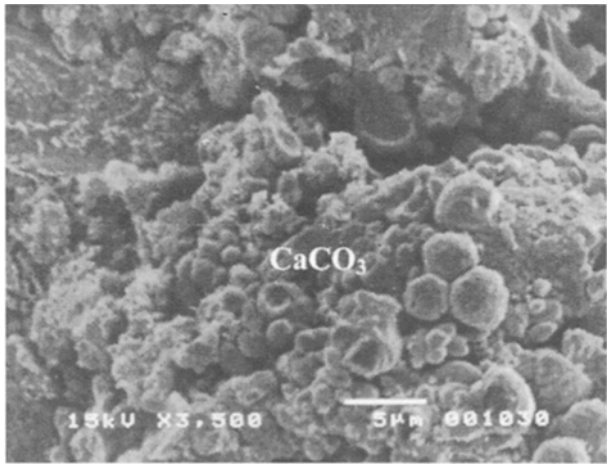

Fig. 6. Scanning electron microscopy of the fractured surface of CO_2 gas-cured board without $MgCl_2$

tional cold-pressing method followed by curing treatment using either gaseous or supercritical CO_2 were improved significantly, and the curing was accelerated dramatically between 10 and 60 minutes. Rapid carbonization might enhance cement hydration and inhibitors; for example, free carbohydrates that retard the hydration of cement lose their effect.

The improvement effect of the CO_2 curing method might be due to the accelerated formation of the hydration products (e.g., calcium carbonate and calcium silicate), which are responsible for the strength development of cement. The addition up to 5.0% $MgCl_2$ to accelerate the setting of cement did not affect the properties of oil palm fronds cement-bonded board when the board was cured with either gaseous or supercritical CO_2. Therefore, this CO_2 curing technology does not need any setting accelerators, which produce a decrease in the dimensional stability of cement-bonded board.

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