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ORIGINAL ARTICLE

Effect of moisture content on manufacturing cement-bonded particleboard using supercritical CO_2

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Abstract This study examined the effects of moisture content (MC) on the manufacture of cement-bonded particleboard (CBP) using supercritical CO_2 in the curing process. Significant correlations were found between MC and the performance of CBP: the internal bond strength, modulus of rupture, and modulus of elasticity values of CBP achieved their maximums, when the MC of boards was approximately 30%. This finding indicated that during the curing phase of manufacturing CBP, a MC of about 30%, which is nearly equal to the water–cement (w/c) ratio of about 0.34, contributes to improved mechanical properties. However, the mechanical properties decreased when the MC was below 30%, which had a negative effect on board performance, indicating that carbon dioxide could not fully react and no carbonation occurred during the curing process. Maintaining a MC of approximately 30% as an ordinary condition of the cement required in the curing of CBP could promote the reaction of carbon dioxide to form calcium carbonate ($CaCO_3$), which leads to increased final strength of CBP. Both X-ray diffractometry and thermal gravimetry observation agreed well with these results and clarified that the increase of $CaCO_3$ content caused by carbonation with increased MC of boards contributed to improving the mechanical properties of CBP.

Keywords Cement-bonded particleboard · Moisture content · Water–cement (w/c) ratio · Supercritical CO_2 · Carbonation

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Introduction

Cement-bonded particleboard (CBP) consists of cement, wood particles, water, and chemical admixture. When all of the materials are mixed, cement powder upon contact with water undergo a hardening reaction (curing) that bonds the wood particles together. In this process, water is essential for cement to harden by hydration [1] and using the optimum amount of water in the presence of moisture content (MC) during the manufacture of CBP is important. Geimer et al. [2] and Simatupang et al. [3] showed that the amount of water is a key factor in the production of wood–cement composites with carbon dioxide injection on the cement setting process, but excessive water has a detrimental effect on the properties of cement-bonded material [4]. In the field of cement-based materials, Boutouil and Levacher [5] investigated the effect of high initial water content and hydrated cement paste on the mechanical performance and porosity. Furthermore, Qi and Cooper [6] examined the effects of wood fiber/cement ratio, water/cement ratio, and carbon dioxide injection time on the properties of wood–cement composites. This research showed that the wood fiber/cement ratio, the water/cement

ratio and their interaction significantly influenced the carbonation reaction and strength of wood–cement composites subjected to 4 min of carbon dioxide injection during the setting process. In general, the carbonation degree was affected more by the water/cement ratio, while the strength of composites was affected more by the wood/cement ratio. Water takes part in the curing of cement by carbon dioxide, a process in which carbon dioxide dissolves in water to form carbonic acid (H_2CO_3). In the latter case of cement hydration, under the influence of carbon dioxide, the formed calcium hydroxide is carbonized to calcium carbonate ($CaCO_3$). These phases are mainly responsible for strength development of cement [7].

Qi and Cooper [6] clarified that the injection of CO_2 gas during pressing is one of the methods to reduce the pressing (setting) time of cement, but the resulting board properties are almost the same as those produced by the conventional method [8], and there is the disadvantage that at least 14 days are required to achieve complete curing [9]. In our previous study [10], we found that the performance of CBP can be changed by the curing treatment with supercritical CO_2. The major advantage of using the supercritical CO_2 treatment was the rapid curing of cement. During a shorter treatment time (30 min), the mechanical properties [internal bond (IB) strength, modulus of rupture (MOR) and modulus of elasticity (MOE)] of CBP achieved the maximum values, which indicated that supercritical CO_2 greatly accelerated the curing process and enhanced the mechanical properties of the boards. The dimensional stability was also improved significantly when boards were treated with supercritical CO_2 for a shorter time. With a longer time span of treatment, IB, MOR and MOE values decreased, with the resulting negative effect on board performance. We concluded that supercritical CO_2 treatment over a longer time span leads to degradation of CBP because of the carbonation of cement.

As a related issue, the effects of MC on the manufacture of CBP by supercritical CO_2 treatment during the curing process have not yet been revealed. During the cement hydration process, di-calcium silicate, Ca_2SiO_4 (C_2S) and tri-calcium silicate, Ca_3SiO_5 (C_3S) are hydrated to form calcium silicate hydrate, $Ca_3Si_2O_7{\cdot}3H_2O$ (CSH) and calcium hydroxide, $Ca(OH)_2$ (CH), respectively [11]. When carbon dioxide is added to cement, calcium carbonate, $CaCO_3$ is formed [8, 9, 12]. In this process, water is necessary to promote the reaction of CO_2. Therefore, in relation to the manufacture of CBP, the presence of these boards which equals to a sufficient water–cement (w/c) ratio during the curing process is important, because it will affect the final performances.

The objectives of this study were to investigate the effect of MC on the manufacture of CBP using supercritical CO_2 in the curing process and to clarify the optimum conditions of MC.

Materials and methods

Materials

Mixtures with proportionally equal amounts of particles of Japanese cypress (*Chamaecyparis obtusa* Endl) and Japanese cedar (*Cryptomeria japonica* D. Don) were used to manufacture CBP. The average size of the wood particles was 3.3 mm (*L*), 0.7 mm (*W*), and 0.1 mm (*T*). Ordinary Portland cement of Osaka Sumitomo was used as a binder; supercritical CO_2 was used as a curing accelerator. To produce supercritical CO_2, CO_2 gas was maintained in the liquid phase by passing the gas through a condenser. The liquid CO_2 was then pumped into the reaction cell surrounded by a water jacket set at 40°C at a pressure of 10 MPa.

Manufacture of CBP

Cement-bonded particleboard with a target density of 1.2 g/cm^3 was manufactured at a cement/wood particle/water weight ratio of 2.5:1.0:1.25. Hand-formed mats of 230 × 230 mm were cold pressed to a targeted thickness of 12 mm and kept in an oven set at 60°C for 24 h (setting process). Then, the mat of CBP was cut into four specimens of 50 × 210 mm each. Before the curing treatment using supercritical CO_2, the MC of specimens was adjusted by drying at 40, 60, 80, or 100°C for 10 h. Afterwards, one of the specimens was taken to measure the constant weight by oven drying at 105°C, so that the MCs of CBP before curing by supercritical CO_2 were controlled from 10 to 30%. To determine the percentage of MC of CBP before curing by supercritical CO_2, the weight of CBP after adjustment by oven drying at 40, 60, 80, or 100°C for 10 h (W_1) and the constant weight by oven drying at 105°C (W_2) were used with the following formula:

$$\mathrm{MC} = \frac{W_1 - W_2}{W_2} \times 100$$

The w/c ratios ranged from 0.07 to 0.34 under the assumption that the fiber saturation point of wood was 28%. According to our previous research finding [10] that during a shorter treatment time supercritical CO_2 greatly accelerated the curing process and enhanced the mechanical properties of CBP, we used a curing treatment with supercritical CO_2 for 30 min. For the treatment with supercritical CO_2, the specimens were placed in a reaction cell surrounded by a water jacket set at 40°C with a pressure of 10 MPa, followed by conditioning at ambient temperature before further testing. For comparison, the additional treatments were (1) curing treatment for 28 days at room temperature (conventional), in which the CBP was wrapped with a polyvinylchloride (PVC) film immediately after clamping, followed by drying in an oven at 80°C for 10 h and

conditioning at room temperature for 1 week; (2) neither conventional curing nor supercritical CO_2 curing treatment (control), in which the CBP was immediately dried at 80°C for 10 h after clamping, followed by 1 week of conditioning as already described; and (3) conventional board treatment, in which the CBP was cured for 28 days at room temperature, but followed by 30 min of supercritical CO_2 treatment.

Evaluation of CBP properties

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 × 210 mm samples for the static bending test and 50 × 50 mm samples for the IB strength, thickness swelling (TS), and water absorption (WA) tests. Four test samples were prepared from each treatment group for the foregoing tests. The static bending test was conducted using a three-point bending test over an effective span of 180 mm (15 times the board thickness) at a loading speed of 10 mm/min. The chemical changes and the mineral decomposition of the CBP as the effects of MC on manufacturing using supercritical CO_2 were examined using X-ray diffractometry (XRD) and thermal gravimetry analysis (TGA).

XRD analysis

Powdered samples of 100-mesh pass taken from an IB test specimens were examined by XRD analysis using a method widely applied to the analysis of cement hydration [13, 14]. Step scan was measured using XRD (RINT 2100; Rigaku) at 40 kV and 40 mA; 2θ ranged from 5.0° to 40.0° when scanning at 0.02° and 2.0°/min. The amounts of unreacted clinkers taken at $2\theta = 32.3°$ and 32.6°, calcium hydroxide at $2\theta = 18.0°$, and calcium carbonate at $2\theta = 29.4°$ and 36.0° for the samples were determined and compared.

TGA analysis

Powdered samples of 100-mesh pass were examined by a thermogravimetric analyzer (TGA 2050; TA Instrument, Japan). Thermal properties of the specimens were observed from room temperature to 800°C at a heating rate of 10°C/min under nitrogen flow.

Results and discussion

Effect of MC on manufacturing CBP using supercritical CO_2

The average values for the IB strength of CBP cured under various conditions are presented in relation to the MC at the curing condition in Fig. 1. The figure shows the linear relationships between the MC and the IB strength of CBP treated with supercritical CO_2. With the increase of MC to about 30%, the IB values of CBP treated with supercritical CO_2 were significantly increased compared to control values. The maximum average IB values of CBP treated by supercritical CO_2 were 1.5 MPa at CBP with MC of about 30%, equaling the w/c ratio of about 0.34. This condition with the MC of about 30% and treatment with supercritical CO_2 allowed the most rapid curing of CBP in the range of this experiment and contributed to increasing the IB strength of CBP. Furthermore, the IB value of CBP treated by supercritical CO_2 for 30 min after conventional curing was improved almost twofold compared to that of a conventional board after 28 days. The IB value of CBP treated by supercritical CO_2 for 30 min after conventional curing was 1.9 MPa. In contrast, the IB value of conventionally cured board after 28 days was 0.7 MPa. On the other hand, when the MC of CBP was less than 30%, e.g., from approximately 24 to 11%, it had a negative effect on the IB strength of CBP. The IB value in these cases ranged from 1 to 0.7 MPa. This result relevance with insufficient water for the chemical reaction because w/c ratio from around 0.24–0.07. It seemed reasonable to conclude that during the curing of CBP using supercritical CO_2, the MC should be about 30% with a w/c ratio of 0.34.

Figure 2 shows the MOR and MOE values of CBP treated with supercritical CO_2 and conventional curing board in relation to the MC conditions during the curing process. The maximum average MOR and MOE values were 16.4 MPa and 8.4 GPa, respectively, for the boards with MC of

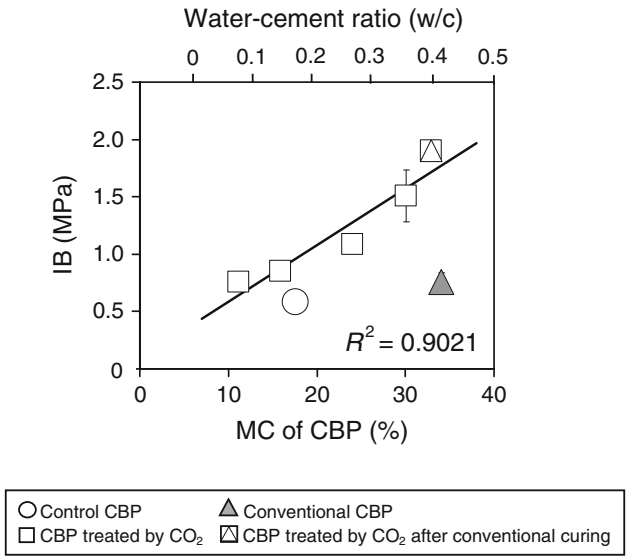


Fig. 1 Effect of MC before curing by supercritical CO_2 on internal bond strength (*IB*) of cement-bonded particleboard (*CBP*). The *solid line* represents the regression line of CBP treated by supercritical CO_2, and CBP treated by supercritical CO_2 after conventional curing

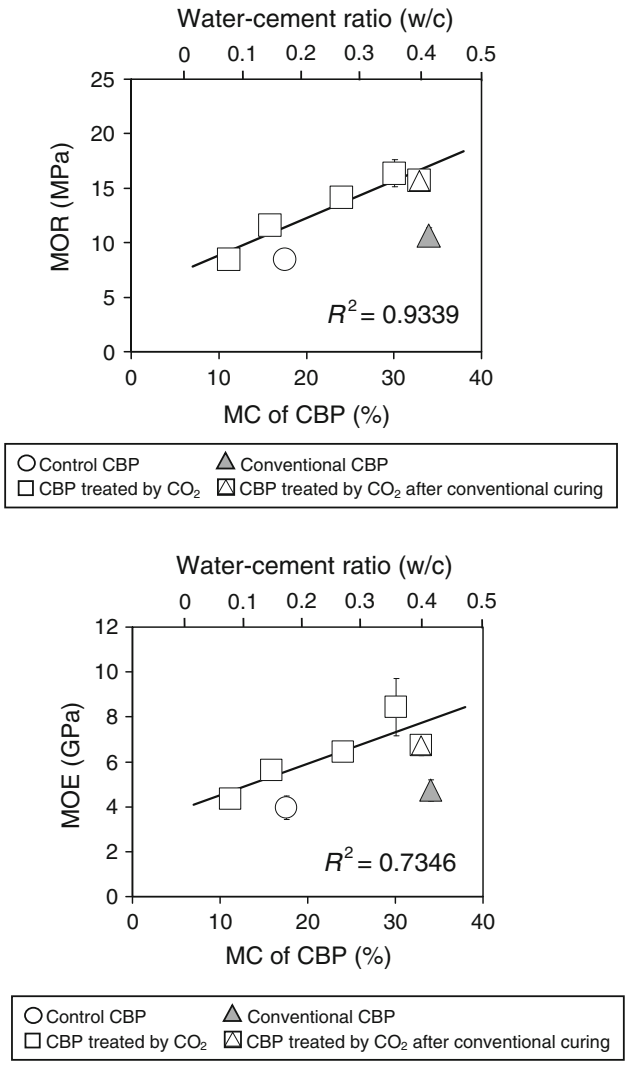


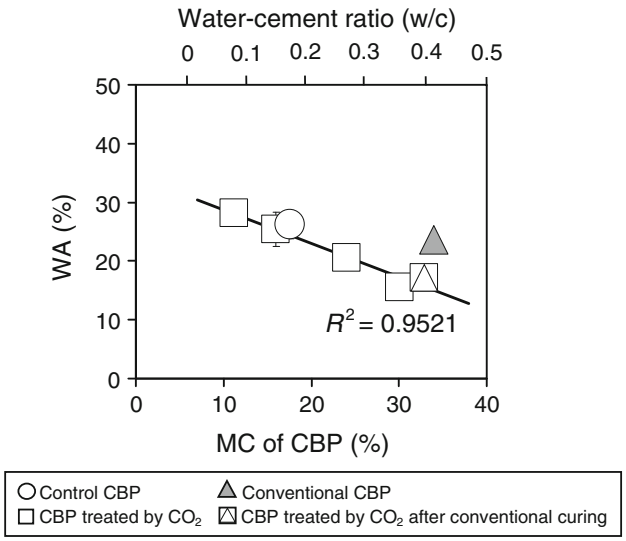
Fig. 2 Effect of MC before curing by supercritical CO_2 on bending strength of cement-bonded particleboard (*CBP*). *MOR* modulus of rupture, *MOE* modulus of elasticity. The *solid line* represents the regression line of CBP treated by supercritical CO_2, and CBP treated by supercritical CO_2 after conventional curing

approximately 30%. This finding shows that MC of approximately 30% and treatment with supercritical CO_2 greatly accelerated the curing process and enhanced the MOR and MOE of CBP. The MOR and MOE values of CBP treated by supercritical CO_2 for 30 min after conventional curing increased and were remarkably higher than the values of conventional board after 28 days as well. The average MOR and MOE values of CBP treated by supercritical CO_2 for 30 min after conventional curing were 15.7 and 6.7 MPa, respectively, for the board with MC of approximately 32%. Compared to CBP treated with supercritical CO_2 with MC below 30%, i.e., from about 24–11%, the MOR and MOE values were decreased and had a negative effect on the board performances. This phenomenon was the result of an insufficient amount of water to react with the cement (a w/c ratio below 0.34), which decreased the bending strength of the CBP. The MOR and MOE values were 8.4 MPa and 4.3 GPa, respectively, for CBP treated with supercritical CO_2 with MC of about 11%. Qi and Cooper [6] also found that an excessively low or excessively high w/c ratio resulted in lower strength of the wood–cement composite, because of the reduced carbonation reaction by either lack of water reactant or the blockage of carbon dioxide pathways by an excessive amount of water. This research showed that when the w/c ratio was either 0.3 or 0.5, carbonation increased with a decreasing wood fiber/cement ratio to 0.42.

The changes to the physical properties as represented by the WA and TS values of the CBP after 24 h of water soaking are shown in Fig. 3. Significant correlations were observed between the WA and TS and the MC conditions of CBP under supercritical CO_2 treatment. The lowest average WA and TS values were 14.4 and 0.39%, respectively, for CBP treated with supercritical CO_2 with MC of approximately 30%. However, the WA and TS values increased with the decrease of MC of CBP, even when it was treated by supercritical CO_2. The WA and TS values of CBP treated by supercritical CO_2 with MC of approximately 11% were 28.1 and 0.8%, respectively, which were higher than the WA and TS values of CBP treated with supercritical CO_2 with MC of approximately 30%. This phenomenon indicated that curing of CBP by supercritical CO_2 with MC below 30% may result in decreasing the dimensional stability, apparently through increased WA ability.

Chemical changes and thermal properties

To obtain information about chemical changes as effects of MC on the manufacture of CBP using supercritical CO_2, we conducted XRD analysis, and the results are shown in Fig. 4. The peak intensity of calcium carbonate ($CaCO_3$) rose with increased MC during curing of CBP, indicating that carbonation of CBP was accelerated in the conditions of sufficient water. Compared to the control, during hydration in conventional CBP, dicalcium silicate (C_2S) and tricalcium silicate (C_3S) are hydrates that form a high content of calcium hydroxide [$Ca(OH)_2$]. The peak intensity of $Ca(OH)_2$ was increased and achieved 1362 cps. Moreover, the peak intensity of $Ca(OH)_2$ of CBP treated by supercritical CO_2 for 30 min after conventional curing with MC of approximately 32% was decreased; in contrast, the peak intensity of $CaCO_3$ was increased, indicating that carbonation still occurred and formed more $CaCO_3$ which improved the mechanical properties. The graph shows the peak intensity of $CaCO_3$ of CBP treated by supercritical CO_2 for 30 min after conventional curing achieved the value at around 621 cps. Furthermore, the peak intensity of $CaCO_3$ of CBP treated by supercritical CO_2 increased significantly with increased MC of CBP. The peak intensity



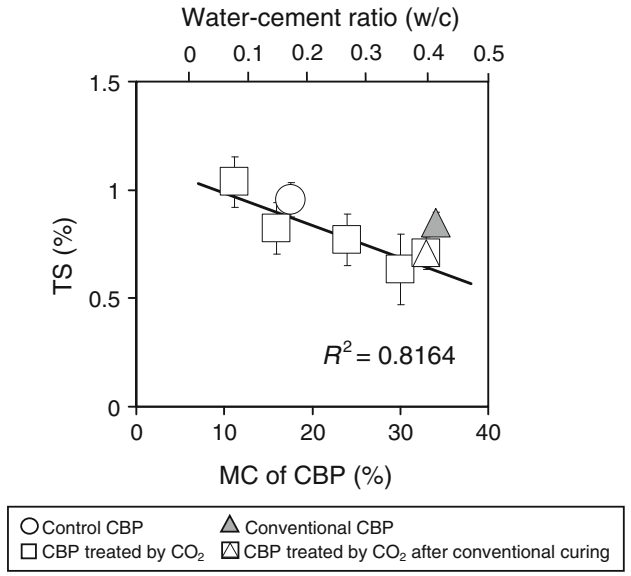


Fig. 3 Effect of MC before curing by supercritical CO_2 on the dimensional stability of cement-bonded particleboard (*CBP*). *WA* water absorption, *TS* thickness swelling. The *solid line* represents the regression line of CBP treated by supercritical CO_2, and CBP treated by supercritical CO_2 after conventional curing

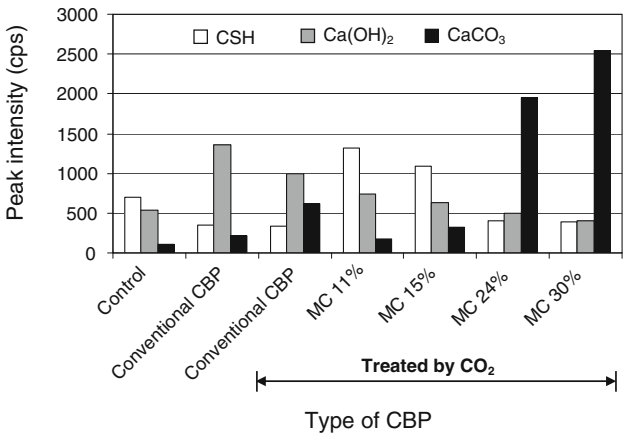


Fig. 4 Chemical change of various CBP by X-ray diffractometry (XRD) analysis

of $CaCO_3$ achieved the highest value of about 2537 cps in CBP treated by supercritical CO_2 with MC of approximately 30%. In contrast, the peak intensities of $Ca(OH)_2$ decreased, indicating that carbonation was accelerated when the crystalline phase of $CaCO_3$ increased significantly with increased MC of CBP. In relation to the performances of CBP, this finding suggests that during curing of CBP using supercritical CO_2, the MC of CBP should be about 30%, which is equal to a w/c ratio of 0.34, because this condition shows sufficient water for chemical reaction by carbon dioxide to form calcium carbonate ($CaCO_3$). Under those conditions, the curing can be complete, leading to a greater final strength of CBP. Qi and Cooper [6] also proved when the original water amount is not sufficient, there is less water in the mixture available for the carbonation reaction, resulting in lower carbonation degree and decreasing the strength of wood–cement composite.

The relationship between temperature and weight loss, and the derivative weight for various CBP in TG and DTG curves are shown in Fig. 5. Supported by the information about thermal properties from previous research [15, 16], the results clarify three significant weight loss steps. The first step occurs in the temperature range from 100 to 300°C, as reflected by the decomposition of CSH. The second step occurs at about 420°C and is due to the decomposition of $Ca(OH)_2$, and the third weight loss step occurs at about 700°C and can be attributed to the decomposition of $CaCO_3$. The TGA curves show that CBP treated with supercritical CO_2 with MC of about 30% loses approximately 24% of its mass over the heating temperature range from 25 to 800°C. This mass loss is mostly attributed to the decomposition of $CaCO_3$. However, the CBP treated with supercritical CO_2 with MC of about 11% loses approximately 16% of its mass, mostly attributed to the decomposition of $Ca(OH)_2$. As a comparison, the weight loss of CBP treated by supercritical CO_2 for 30 min after conventional curing with MC of about 32% was higher than that of conventional CBP after 28 days. The CBP treated by supercritical CO_2 for 30 min after conventional curing with MC of about 32% loses approximately 26% of its mass, also attributed to the decomposition of $CaCO_3$. According to the DTG curves, the highest decomposition peak of $CaCO_3$ was found in CBP treated with supercritical CO_2 with MC of about 30%. The decomposition peaks of $Ca(OH)_2$ were decreased with the increase in MC of CBP during the curing process, equaling the increased decomposition peaks of $CaCO_3$. This result indicated that carbonation occurred when the crystalline phase of $CaCO_3$

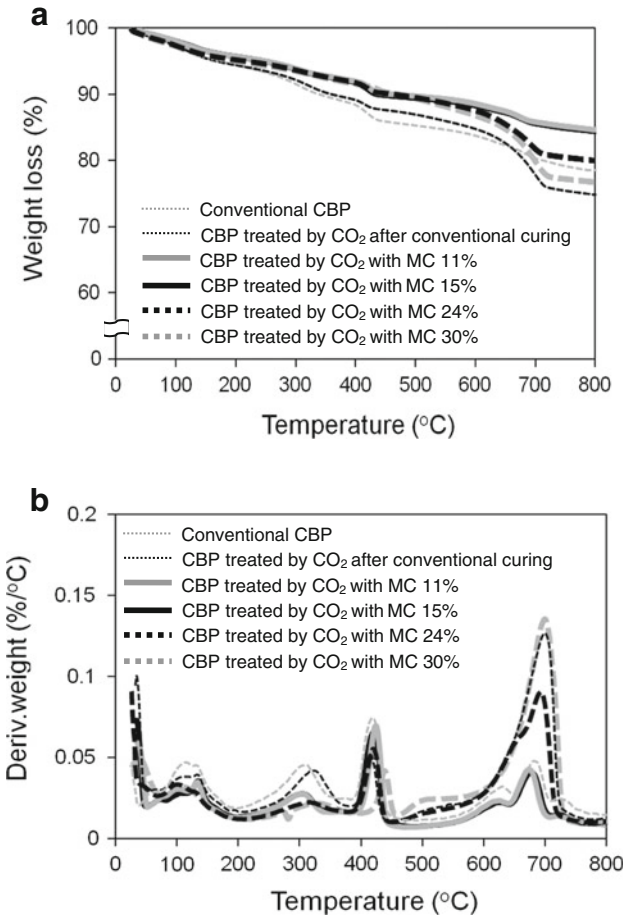


Fig. 5 Thermal properties of various CBP; thermal gravimetry (TG) (**a**); DTG (**b**)

increased significantly with increased MC of about 30% (equal to a w/c ratio of about 0.34) during the curing process with supercritical CO_2, and this condition contributed to improving the mechanical properties of the boards. Nonetheless, when the decomposition peaks of chemical components of $Ca(OH)_2$ and $CaCO_3$ were decreased, the boards did not carbonate successfully, which had a negative effect on the final performance of CBP.

Conclusions

The MC affects the performance of CBP during manufacturing that includes the use of supercritical CO_2 in the curing process. The IB, MOR and MOE values of the CBP treated with supercritical CO_2 with MC of about 30% achieved maximum values, indicating that this level of MC, equaling a w/c ratio of about 0.34, which is an ordinary condition of the cement required in the curing of CBP, and could promote the reaction of carbon dioxide to form calcium carbonate ($CaCO_3$), leading to an increase in the final strength of CBP. When CBP was manufactured using supercritical CO_2 with MC below 30%, i.e., with insufficient water and a w/c ratio below 0.34, the carbon dioxide could not fully react and CBP did not carbonate successfully. This condition had a negative effect on the CBP performance. Both XRD and TGA observation clarified the increase of $CaCO_3$ content caused by carbonation with increased MC of boards, and this phenomenon contributed to improving the mechanical properties of CBP.

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