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ORIGINAL ARTICLE

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Preservative treatment of wood-based composites with 3-iodo-2-propynyl butylcarbamate using supercritical carbon dioxide impregnation

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Abstract Five structural-use wood-based composites [medium density fiberboard (MDF), hardwood plywood, softwood plywood, particleboard, oriented strand board (OSB)] were treated with 3-iodo-2-propynyl butylcarbamate (IPBC) using supercritical carbon dioxide (SC-CO_2) as a carrier solvent. Treatment was conducted at 35°C/7.85 MPa (80 kgf/cm^2), 35°C/9.81 MPa (100 kgf/cm^2), 35°C/11.77 MPa (120 kgf/cm^2), 45°C/7.85 MPa, 45°C/9.81 MPa, 45°C/11.77 MPa, 55°C/7.85 MPa, 55°C/9.81 MPa, and 55°C/11.77 MPa. A decay test was carried out in the laboratory according to the modified Japanese standard method in which untreated and treated specimens were exposed for 12 weeks to a monoculture of the white-rot fungus *Trametes versicolor* (L.: Fr.) Pilat or the brown-rot fungus *Fomitopsis palustris* (Berk. et Curt.) Gilbn. and Ryv. IPBC/SC-CO_2 treatment protected the treated materials from decay by the two fungi, although the relative efficiency against decay varied with the treatment conditions and the type of wood-based composite. Better performance for MDF, hardwood plywood, and particleboard was generally obtained at high temperatures and pressures, whereas softwood plywood and OSB were most protected at low temperatures under any of the pressure levels tested.

Key words Supercritical carbon dioxide · 3-Iodo-2-propynyl butylcarbamate (IPBC) · Decay resistance · Wood-based composites

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Introduction

Because conventional preservative treatment methods can adversely affect wood-based composites, alternative treatment methods are gaining increasing importance with the increased use of wood-based composites as building or structural components under conditions suitable for biological attack. Pressure treatment with conventional waterborne preservatives resulted in permanent swelling and delamination of waferboard.[1] Moreover, redrying at high temperatures after pressure treatment may cause sheet twisting[2] and reduce mechanical properties.[3] Glue-line treatment with boric acid requires a selective glue formulation and manufacturing process to avoid leaching of boric acid from the products.[4] Of various preservative treatments, such as an aqueous mixture with wax emulsion, a mixture with a liquid resin, and dip treatment of finished panels, only the wax emulsion treatment with ammoniacal copper arsenate (ACA) made the treated materials suitable for underground use.[5] However, ACA-treated hardwood particleboard did not remain resistant to biological attack in a subtropical climate with prolonged exposure owing to the loss of preservative compounds.[6] Furthermore, vapor-boron treatment of wood-based composites is thought to be applicable only to nonleaching, protected conditions.[7]

Another alternative approach is the use of supercritical carbon dioxide (SC-CO_2) as a carrier solvent for biocides. We previously demonstrated the feasibility of SC-CO_2 as a carrier of biocide for preservative treatment of wood-based composites. SC-CO_2 impregnation without biocide did not have any unfavorable effect on the composites.[8] Similarly, impregnation of the fungicide 3-iodo-2-propynyl butylcarbamate (IPBC) into five commercially available wood-based composites did not seem to produce any conspicuous negative defects in terms of physical or mechanical properties when treated at 50°C and 9.81 MPa for 30 min.[9] Acda et al.[10] previously suggested that SC-CO_2 may be suitable for treating of wood-based composites with tebuconazole at pressures ranging from 1800 psig (12.41 MPa) to 4500 psig (31.03 MPa) and temperatures ranging from 40° to 75°C.

Although the solubility of biocides in SC-CO_2 generally increases with increasing pressure or temperature,[11] treatment at lower temperatures and pressures are practically and economically beneficial. It is therefore worthwhile to examine the treatment conditions that would not allow high solubility of biocides.

The purpose of the current investigation was to determine whether SC-CO_2 was applicable to the treatment of wood-based composites with IPBC according to a laboratory decay test. Selection of optimum treatment conditions for each wood-based composite was also attempted based on a statistical comparison of the results of the decay test and the treatment parameters.

Materials and methods

Specimens (210 × 30 mm × thickness) were prepared from five commercially available structural-use wood-based composites: medium density fiberboard (MDF) made from hardwood fibers [oven-dried density (o.d.d) 0.67 g/cm^3; thickness 11.9 mm; adhesive melamine-urea-formaldehyde (MUF) resin], hardwood plywood [five plies; o.d.d. 0.56 g/cm^3; thickness 12.1 mm; adhesive phenol-formaldehyde (PF) resin; type I –boiled-water resistant for exterior use], softwood plywood (five plies; o.d.d. 0.56 g/cm^3; thickness 11.7 mm; adhesive PF resin; type I –boiled-water resistant for exterior use), particleboard made from both hardwood and softwood particles (o.d.d. 0.75 g/cm^3; thickness 11.7 mm; adhesive MUF resin), and aspen oriented strand board (OSB) (o.d.d. 0.62 g/cm^3; thickness 12.2 mm; adhesive PF resin). For details of physical and mechanical properties of the test materials, refer to a previous paper.[8] The specimens were double-coated with epoxy resin on each cut end and conditioned at 60°C for 72 h prior to IPBC/SC-CO_2 treatment.

Treatment was conducted at 35°C/7.85 MPa (80 kgf/cm^2), 35°C/9.81 MPa (100 kgf/cm^2), 35°C/11.77 MPa (120 kgf/cm^2), 45°C/7.85 MPa, 45°C/9.81 MPa, 45°C/11.77 MPa, 55°C/7.85 MPa, 55°C/9.81 MPa, and 55°C/11.77 MPa. Two specimens of each wood-based composite were placed in the treatment vessel for each run. The treatment vessel was immersed in a temperature-controlled waterbath and preheated to the desired temperature. Liquefied CO_2 (99.5% purity) (Kyoto Teisan, Kyoto, Japan) was introduced into the treatment vessel until the pressure in the vessel was equal to that of the CO_2 source. The pressure was raised to the target level by introducing liquefied CO_2 into the treatment vessel with a double pressure pump at a rate of 9 ml/min. After CO_2 reached its critical pressure, 4 g of IPBC (99.1% a.i.) (Arch Chemicals, Cheshire, CT, USA) dissolved in 20 ml ethanol (99.5% purity) (Nacalai Tesque, Kyoto, Japan) was immediately introduced into the treatment vessel using another double pump at a rate of 2 ml/min. Direct introduction of either CO_2 or IPBC into the treatment vessel was used to avoid crystallization of IPBC in liquid CO_2. The treatment vessel was maintained under the test conditions for 30 min to allow IPBC/SC-CO_2 to circulate evenly inside the vessel. At the end of treatment, pressure was released into the ambient atmospheric pressure, and the treated materials were recovered for subsequent testing. Each treatment condition was conducted three times, giving six replicates for each wood-based composite.

A decay test was conducted with unweathered specimens of untreated control and treated materials according to JIS K 1571.[12] For each of the wood-based composites, six blocks (24 × 28 mm × thickness) were equally cut from both ends of the six treated specimens after cutting off the resin coat; thus, 36 blocks were obtained for each treatment condition. All blocks were oven-dried at 60°C for 48 h, weighed, and then sterilized with gaseous ethylene oxide before the decay test. Three blocks were exposed to a monoculture of either the white-rot fungus *Trametes versicolor* (L.: Fr.) Pilat (fungal accession number of the Forestry and Forest Products Research Institute, Tsukuba, Japan: FFPRI 1030) or the brown-rot fungus *Fomitopsis palustris* (Berk. et Curt.) Gilbn. and Ryv. (FFPRI 0507) in a glass jar at 26° ± 2°C for 12 weeks. Three decay jars were used to test nine replicates of each treated material against the two fungi. The remaining 18 blocks were exposed to the same test units but with inactivated fungi to correct for weight changes associated with unforeseen causes other than decay fungi. The percent mass loss was calculated from the corrected difference in the oven-dried weight of each block before and after the decay test. The mass losses of untreated wood-based composites were statistically analyzed by Tukey's test to compare the natural decay resistance of the test materials. To compare the efficacy of treatment, the relative efficiency against decay (RED) was also calculated according to the following equation.

RED = [(% mean mass loss of untreated samples) − (% mean mass loss of treated samples)]/(% mean mass loss of untreated samples) × 100

The effects of treatment parameters were analyzed by two-way 3 × 3 factorial analysis of variance (ANOVA) using a computer program (VassartStats; Vassar College, Poughkeepsie, NY, USA) to select the optimum treatment condition for each wood-based composite.

Results and discussion

Table 1 shows the percent mass loss of untreated wood-based composites by the white-rot fungus *T. versicolor* and the brown-rot fungus *F. palustris*. Statistical analyses showed that each type of wood-based composite had a different natural durability against the decay fungi. The white-rot fungus *T. versicolor* preferably attacked untreated hardwood plywood and OSB, whereas the brown-rot fungus *F. palustris* readily attacked the untreated softwood plywood and OSB. MDF and hardwood plywood specimens seemed to be resistant to *F. palustris*. Particleboard was naturally resistant to both test fungi. These phenomena are probably due to the selective or partial degradation (or both) of board constituents by the test fungi. White-rot

Table 1. Decay resistance of untreated wood-based composites

Wood-based composite	Mass loss (%) against test fungi[a,b]	
	Trametes versicolor	*Fomitopsis palustris*
Medium-density fiberboard	8.30 ± 1.16 (B)	2.58 ± 0.35 (A)
Hardwood plywood	26.14 ± 2.09 (D)	3.93 ± 1.79 (A)
Softwood plywood	21.58 ± 1.56 (C)	25.62 ± 6.31 (B)
Particleboard	2.13 ± 0.06 (A)	1.83 ± 0.55 (A)
Oriented strand board	26.86 ± 2.73 (D)	29.22 ± 3.15 (B)

[a] Mean ± SD of nine test blocks
[b] Values in the column with different letters are significantly different by Tukey's test at $P < 0.01$

Table 2. Relative efficiency against decay of 3-iodo-2-propynyl butylcarbamate/supercritical CO_2 treatments of composites

Treatment conditions	RED of treatment on wood-based composites against *Trv* and *Fop*[a]					
	Medium-density fiberboard (*Trv*)	Hardwood plywood (*Trv*)	Softwood plywood		Oriented strand board	
			Trv	*Fop*	*Trv*	*Fop*
35°C/7.85 MPa	63	11	16	90	21	−13
35°C/9.81 MPa	43	12	45	86	14	15
35°C/11.77 MPa	42	10	27	85	3	9
45°C/7.85 MPa	39	−13	0	21	4	−28
45°C/9.81 MPa	63	4	−4	49	9	−24
45°C/11.77 MPa	25	32	9	80	−3	−3
55°C/7.85 MPa	43	0	6	50	3	−13
55°C/9.81 MPa	47	8	18	15	−12	5
55°C/11.77 MPa	92	88	20	93	18	12

RED, relative efficiency against decay; *Trv*, *Trametes versicolor*; *Fop*, *Fomitopsis palustris*
[a] RED is calculated as: [(percent mass loss of the untreated specimens) − (percent mass loss of the treated specimens) / (percent mass loss of the untreated specimens)] × 100

fungi, which are capable of degrading both cellulose and lignin, more readily degraded the syringyl elements of hardwood lignin than the guaiacyl elements of softwood lignin.[13,14] It is also well known that wood-based composites containing a high percentage of naturally durable species or heartwood significantly contribute to higher decay resistance.[15,16]

Table 2 shows the efficacy of IPBC/SC-CO_2 treatment in suppressing attacks by the test decay fungi. Although decay resistance clearly varied with the treatment conditions and the type of wood-based composite, the current treatment conditions improved the decay resistance of most of the wood-based composites, except for some OSBs. Such enhancement was expected, as IPBC has been shown to be as effective as pentachlorophenol (PCP)[17] and is possibly more soluble in SC-CO_2 than is PCP, even at the temperatures and pressures of the current investigation. The highest RED values also support this evidence: 92, 88, 45, and 21 against *T. versicolor* for MDF, hardwood plywood, softwood plywood, and OSB, respectively, and 93 and 15 against *F. palustris* for softwood plywood and OSB, respectively. When the untreated specimens sustained little mass loss by decay, it was not worth comparing the REDs among the treatment conditions. Thus, RED figures for MDF, hardwood plywood, and particleboard against *F. palustris* and for particleboard against *T. versicolor* are not included in Table 2.

Most of the treatments did not suppress both decay fungi to a satisfactory mean mass loss of less than 3%. In this study, only the treatment of hardwood plywood at 55°C/11.77 MPa produced a satisfactory performance. The mean mass losses of untreated particleboard by the two test fungi and MDF by *F. palustris* were not high enough to allow a comparison of decay resistance between the untreated and treated groups. The lower decay resistance of treated OSB and other samples was probably due to insufficient retention of IPBC, which seemed to reflect the importance of the board composition, its internal structure, and the resultant permeability of the products.

Table 3 shows the theoretical maximum retention of IPBC in the wood-based composites. These were calculated from the void volume in correlation with their density and moisture contents, with the assumption that 4 g of IPBC was evenly distributed inside the treatment vessel ($2.36 \times 10^{-3}\,m^3$), and all of the IPBC in the voids of each sample was thoroughly deposited in the sample. Toxic threshold values against *T. versicolor* and *F. palustris* determined for *Cryptomeria japonica* sapwood blocks vacuum/soak-impregnated with IPBC were 0.29–0.59 kg/m^3.[18] Although the theoretical retention in the wood-based composites was

Table 3. Theoretical maximum retention of IPBC in treated composites[a]

Wood-based composite	Density (g/cm^3)	Moisture content (%)	Maximum retention (kg/m^3)
Medium-density fiberboard	0.67	6.87	0.88
Hardwood plywood	0.56	7.65	1.01
Softwood plywood	0.56	8.10	1.00
Particleboard	0.75	6.81	0.78
Oriented strand board	0.62	6.54	0.95

IPBC, 3-iodo-2-propynyl butylcarbamate
[a] With the assumptions that 4 g of IPBC introduced into a treatment vessel with a volume of 2.36 $\times 10^{-3}$ m^3 was evenly distributed and that all of the IPBC inside the voids of each sample was thoroughly deposited

Table 4. ANOVA for the mass loss of IPBC/supercritical CO_2-treated composites against *Trametes versicolor*

Source of variation	Sum of squares	Degrees of freedom	Mean square	*F*	*P*
Medium-density fiberboard					
Temperature (*t*)	30.98	2	15.49	11.08	<0.0001**
Pressure (*p*)	1.96	2	0.98	0.70	0.4999
t-p Interaction	147.17	4	36.79	26.31	<0.0001**
Error	100.69	72	1.40		
Total	280.79	80			
Hardwood plywood					
Temperature	641.84	2	320.92	33.75	<0.0001**
Pressure	1982.62	2	991.31	104.24	<0.0001**
t-p Interaction	1565.37	4	391.34	41.15	<0.0001**
Error	684.72	72	9.51		
Total	4874.55	80			
Softwood plywood					
Temperature	475.24	2	237.62	16.76	<0.0001**
Pressure	120.58	2	60.29	4.25	0.0180*
t-p Interaction	142.96	4	35.74	2.52	0.0485*
Error	1021.09	72	14.18		
Total	1759.87	80			
Particleboard					
Temperature	26.80	2	13.40	83.90	<0.0001**
Pressure	12.34	2	6.17	38.63	<0.0001**
t-p Interaction	20.36	4	5.09	31.86	<0.0001**
Error	11.50	72	0.16		
Total	71.00	80			
Oriented strand board					
Temperature	113.23	2	56.62	2.73	0.0720
Pressure	27.80	2	13.90	0.67	0.5149
t-p Interaction	405.14	4	101.29	4.88	0.0015*
Error	1494.93	72	20.76		
Total	2041.09	80			

ANOVA, analysis of variance
* $P < 0.05$; ** $P < 0.01$

much higher than the threshold values, the levels of protection were far below our expectations. This means that only a small portion of the IPBC introduced into the treatment vessel was taken up by the samples. Another study indicated that the IPBC treatment of pine sapwood blocks using SC-CO_2 was ineffective at a retention of 1.56 kg/m^3 when leached specimens were tested against *T. versicolor*.[11] These results suggest that at least twice as much IPBC must be incorporated under the present treatment conditions to obtain satisfactory performance of the treated materials against decay.

Analyses of variance were conducted to examine the effects of temperature and pressure and their interaction on the mass loss of each wood-based composite by *T. versicolor* and *F. palustris*, as shown in Tables 4 and 5, respectively. Particleboard was included in the analyses because the material inevitably decays after long practical service, and the decay test data can be used for selecting the optimal treatment condition. The results indicated that temperature had a significant independent effect on the decay resistance of all of the wood-based composites against the two test fungi except for OSB against *T. versicolor* whereas pressure had less of an effect, and no significant effects were seen with MDF and OSB against *T. versicolor* and hardwood plywood against *F. palustris*. The interaction between the treatment temperature and pressure also significantly

Table 5. ANOVA for the mass loss of IPBC/supercritical CO_2-treated composites against *Fomitopsis palustris*

Source of variation	Sum of squares	Degrees of freedom	Mean square	F	P
Medium-density fiberboard					
Temperature (t)	20.73	2	10.37	39.40	<0.0001**
Pressure (p)	2.32	2	1.16	4.41	0.0156*
t-p Interaction	4.40	4	1.10	4.18	0.0042**
Error	18.94	72	0.26		
Total	46.39	80			
Hardwood plywood					
Temperature	11.59	2	5.80	13.73	<0.0001**
Pressure	1.50	2	0.75	1.78	0.1760
t-p Interaction	4.16	4	1.04	2.47	0.0522
Error	30.38	72	0.42		
Total	47.64	80			
Softwood plywood					
Temperature	1517.06	2	758.53	46.11	<0.0001**
Pressure	1382.56	2	691.28	42.02	<0.0001**
t-p Interaction	1468.78	4	367.19	22.32	<0.0001**
Error	1184.39	72	16.45		
Total	5552.79	80			
Particleboard					
Temperature	14.94	2	7.47	34.32	<0.0001**
Pressure	1.42	2	0.71	3.26	0.0441*
t-p Interaction	3.95	4	0.99	4.53	0.0025**
Error	15.67	72	0.22		
Total	35.97	80			
Oriented strand board					
Temperature	696.55	2	348.28	16.36	<0.0001**
Pressure	670.07	2	335.04	15.74	<0.0001**
t-p Interaction	178.51	4	44.63	2.10	0.0896
Error	1532.29	72	21.28		
Total	3077.43	80			

* $P < 0.05$; ** $P < 0.01$

affected the decay resistance of all of the wood-based composites against *T. versicolor*, but not those of hardwood plywood and OSB against *F. palustris*.

The effects of the interaction between the treatment temperature and pressure on the decay resistance of wood-based composites against *T. versicolor* and *F. palustris* are shown in Figs. 1 and 2, respectively. Overall, treatment at 55°C gave better results than treatment at either 35°C or 45°C for MDF, hardwood plywood, and particleboard. This was much more pronounced at 11.77 MPa. On the other hand, the treatment of softwood plywood and OSB at 35°C gave a better performance than treatment at other temperatures, especially at 9.81 MPa. The effect of treatment was probably associated with the CO_2 density in the treatment vessel.

Based on the quantity of CO_2 introduced into the treatment vessel, the CO_2 density was approximately 499.92 and 628.18 kg/m^3 at 55°C/11.77 MPa or 35°C/9.81 MPa, respectively. Decreasing the temperature from 55°C or 45°C or 35°C at 11.77 MPa increased the CO_2 density to 577.25 and 696.10 kg/m^3, respectively. However, an increased density adversely affected the decay resistance of the treated materials. In contrast, when the pressure was reduced to 7.85 MPa at 35°C, the CO_2 density fell to 420.20 kg/m^3. These results suggest that SC-CO_2 at an elevated temperature initially resulted in increased retention at a constant pressure because of a reduction in the density of CO_2, whereas a further temperature increase adversely decreased retention as volatility increased.[19] The present results therefore suggest that at 35°C and a pressure higher than 9.81 MPa retention of IPBC in softwood plywood and OSB decreased (based on the results of decay tests). However, the IPBC/SC-CO_2 treatment of MDF, hardwood plywood, and particleboard at 55°C/11.77 MPa seemed to result in better performance.

Although IPBC was highly soluble in SC-CO_2 at the high pressure of 25 MPa, as previously reported,[11] the current treatment conditions seemed to solubilize only a small amount of IPBC. However, as demonstrated here, solubility is not likely the sole factor that causes the treated materials to exhibit a satisfactory performance, and the interaction of treatment parameters such as temperature and pressure plays an important role in selecting the optimal treatment condition for each wood-based composite.

Conclusions

The use of SC-CO_2 as a carrier solvent for biocides during preservative treatment of wood-based composites is feasible under the present test conditions. Our results indicate that the success of SC-CO_2 treatment strongly depends on the temperature, the pressure, their interaction, the amount

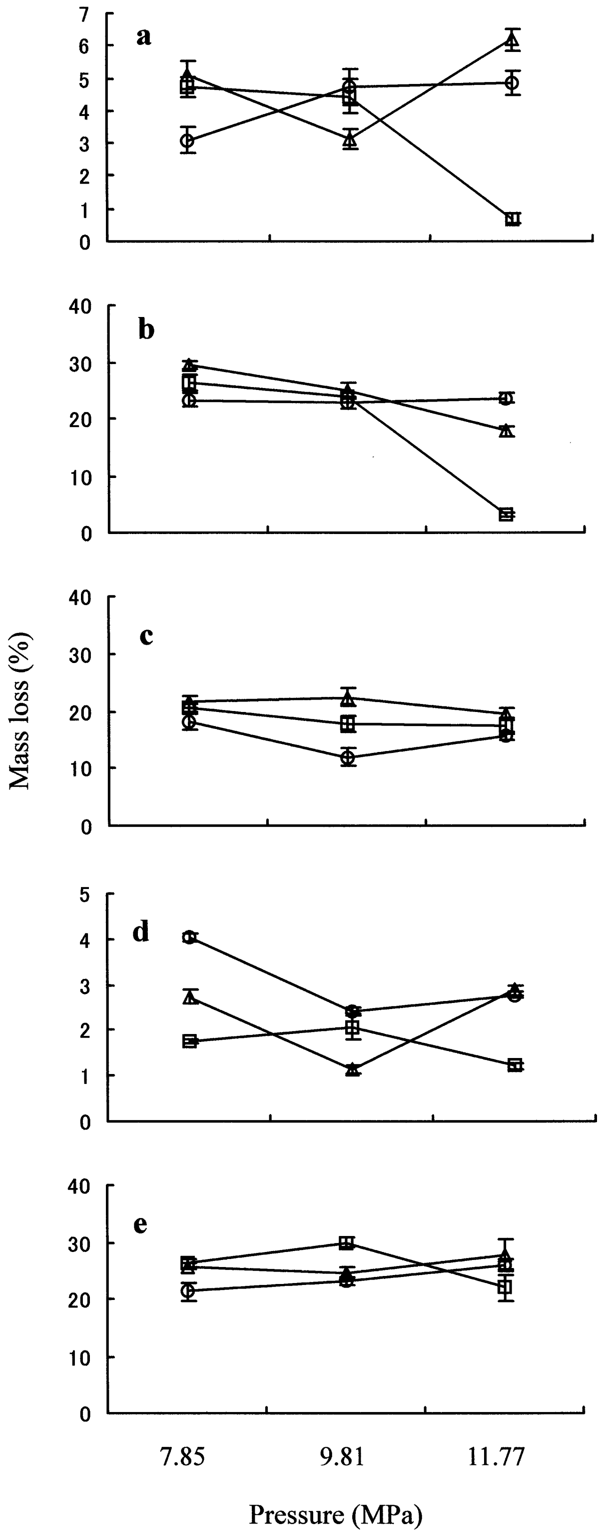

Fig. 1. Effect of the interaction between treatment temperature and pressure on the mass loss of medium-density fiberboard (**a**), hardwood plywood (**b**), softwood plywood (**c**), particleboard (**d**), and oriented strand board (**e**) with *Trametes versicolor*. *Open circles*, *triangles*, and *squares* represent treatment temperatures of 35°C, 45°C, and 55°C, respectively. *Vertical lines* represent standard errors

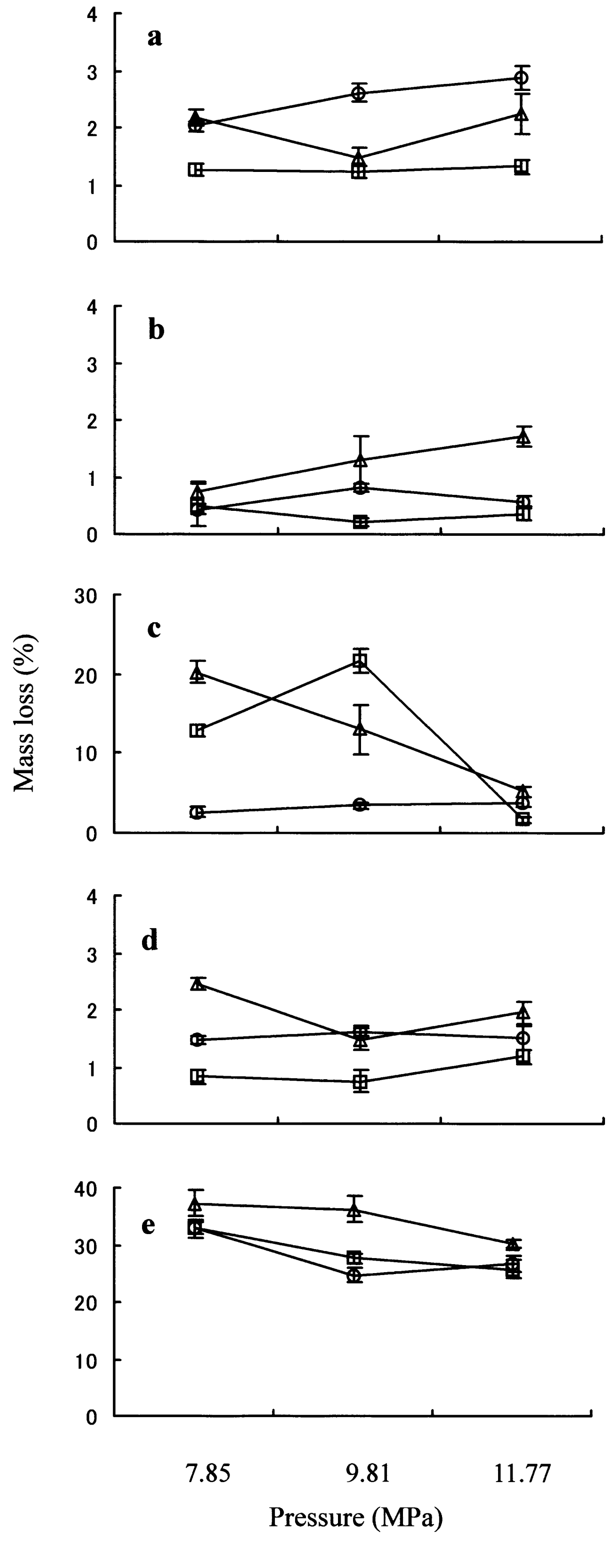

Fig. 2. Effect of the interaction between treatment temperature and pressure on the mass loss of medium-density fiberboard (**a**), hardwood plywood (**b**), softwood plywood (**c**), particleboard (**d**), and oriented strand board (**e**) with *Fomitopsis palustris*. *Open circles*, *triangles*, and *squares* represent treatment temperatures of 35°C, 45°C, and 55°C, respectively. *Vertical lines* represent standard errors

of biocide applied, and the type of wood-based composite treated. The treatment of wood-based composites with IPBC using SC-CO_2 produced a better performance at 55°C/11.77 MPa for MDF, hardwood plywood, and particleboard and at 35°C/9.81 MPa for softwood plywood and OSB based on the results of the decay test and statistical analyses of the treatment parameters.

The differences in optimal treatment conditions among the wood-based composites might be partly explained by the relation between the permeability of the composites and the density of CO_2 under the treatment conditions. However, most of the treatments did not suppress either decay fungus to a satisfactory level of decay resistance (mean mass loss <3%). Accordingly, the results obtained indicated that the amount of IPBC introduced into the wood-based composites was not high enough to prevent decay satisfactorily, although no definite conclusions can be drawn until chemical analyses of the retention results are undertaken. An additional study is therefore needed to determine the required amount of IPBC to be incorporated.

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