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Lactic acid/wood-based composite material. Part 2: Physical and mechanical performance

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ABSTRACT

The synthesis of an innovative bio-composite material based on wood and lactic acid oligomers has been reported in Part 1. As a continuation of this previous work, this paper examines the bio-composite material's physical and mechanical performance. Properties were assessed in terms of dimensional stability, decay resistance, leaching, bending, shearing, compression and hardness testing. It has been shown that physical performance of the bio-composite was highly improved, in spite of high leaching mass loss. The mechanical structural properties were not strongly affected, except in decrease of shearing resistance due to the middle lamella degradation. An increase in hardness properties was also noticed.

1. Introduction

"Green" or "eco"-composites are showing potential in terms of lifecycle environmental impact, including origin, processing, utilisation and degradation. In Part 1 of this study, it has been shown that lactic acid oligomers can polymerise into the wood cells walls. Without a chemical catalyst, oligomer in situ polymerisation leads to densified wood composite. Otherwise, the permeation of sulphuric acid or tin octoate into the melting oligomers damages wood cell cohesion, which translates into severe softening of the composite during the heating stage. However, this degradation seems to be compensated by a more advanced polymerisation achieved by an extended heating stage, inducing material re-hardening.

Wood chemical modification is mostly intended to improve dimensional stabilisation and mechanical properties of wood. For this purpose, many monomers and impregnation compounds have been used for wood treatment, such as phenol, urea, melamine–formaldehyde resins, polyethylene glycol and isocyanate resins. For example, Gao and Li (2007) reported the chemical modification of poplar wood with foaming polyurethane resins, leading to the improvement of the dimensional stability due to the wood hydroxyl groups blocked by the isocyano groups, and of the mechanical properties due to the reinforcement of foam in the cell walls and void spaces. To obtain improvement of both properties, triethanolamine, diethylenetriamine, triethylenediamine and *N*-methyl morpholine were used as catalysts and improved both stability and mechanical properties (Gao et al., 2009).

When monomers are able to penetrate into the cell wall, which is achievable if a chemical interaction is possible, properties can be highly improved. Numerous treatments have been widely reported, such as glycidyl methacrylate and diallyl phthalate, for dimensional stability of wood fibres (Rozman et al., 1996). Glycidyl methacrylate can also be used as a coupling agent between solid rubber wood and styrene, as suggested by Devi and Maji (2008). Şolpan and Güven (1998, 1999a,b) improved the mechanical stability of oak, spruce and beech by radiation-induced in situ copolymerisation of acrylonitrile and methyl methacrylate, and glycidyl ether monomers or monomer mixtures.

This study has examined the physical and mechanical performance of softened and hardened composites obtained by lactic acid oligomer in situ polymerisation. More specifically, anti-swelling efficiency, biological resistance, leaching resistance, flexural, shearing, compression and hardness characteristics have been determined.

2. Methods

2.1. Materials

Details of the materials tested in this study are summarised in Table 1. Precise details of the manufacture and processing of the composites are given in Part I.

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Table 1
Chemical and processing characteristics of samples used for testing.

Sample reference	Wood specie	Base agent	Catalyst	Heating characteristics
PrP B SH	Beech	LA oligomers	Ø	120 °C/1 h
PrP B EH	Beech	LA oligomers	Ø	120 °C/1 h + 103 °C/96 h
PPSA B SH	Beech	LA oligomers	H_2SO_4 (0.6 wt%)	120 °C/1 h
PPSA B EH	Beech	LA oligomers	H_2SO_4 (0.6 wt%)	120 °C/1 h + 103 °C/96 h
PPTO B SH	Beech	LA oligomers	Tin (II) octoate (5 wt%)	120 °C/1 h
PPTO B EH	Beech	LA oligomers	Tin (II) octoate (5 wt%)	120 °C/1 h + 103 °C/96 h
PrP P SH	Pine sapwood	LA oligomers	Ø	120 °C/1 h
PrP P EH	Pine sapwood	LA oligomers	Ø	120 °C/1 h + 103 °C/96 h
PPSA P SH	Pine sapwood	LA oligomers	H_2SO_4 (0.6 wt%)	120 °C/1 h
PPSA P EH	Pine sapwood	LA oligomers	H_2SO_4 (0.6 wt%)	120 °C/1 h + 103 °C/96 h
PPTO P SH	Pine sapwood	LA oligomers	Tin (II) octoate (5 wt%)	120 °C/1 h
PPTO P EH	Pine sapwood	LA oligomers	Tin (II) octoate (5 wt%)	120 °C/1 h + 103 °C/96 h

2.2. Physical characterisation

2.2.1. Anti-swelling efficiency

The anti-swelling efficiency (ASE) index was determined in order to evaluate dimensional stability of treated wood specimens. Treated and untreated wood blocks ($20 \times 20 \times 20\ mm^3$) were stored at a temperature of 20 °C and relative humidity of 100% until the samples reached equilibrium moisture content (EMC was considered to be reached when the mass change of the samples within 24 h had been less than 0.5%). The samples were then oven dried. Volumetric swelling coefficients were calculated according to the formula:

$$S(\%) = \frac{V_2 - V_1}{V_1} \times 100$$

where V_2 is the volume of the moisture-saturated blocks and V_1 is the volume of oven-dried blocks.

The ASE percentage was calculated from the wet and oven-dried volumes of treated and untreated wood specimens according to the formula:

$$\mathrm{ASE}(\%) = \frac{S_U - S_T}{S_U} \times 100$$

where S_U is the volumetric swelling coefficient of untreated blocks and S_T is the volumetric swelling coefficient of treated blocks.

2.2.2. Decay resistance

Fungal durability after treatment was evaluated according to rapid testing adapted from EN 113 European standard. Fungal resistance was performed on PrP B EH, PrP P EH, PPSA B EH, PPSA P EH, PPTO B EH and PPTO P EH ($50 \times 15 \times 5\ mm^3$) samples. Beech (*Fagus sylvatica*) samples were evaluated against *Gloeophyllum trabeum* and *Coriolus versicolor*, while pine *(Pinus sylvestris)* samples were evaluated against *Gloeophyllum trabeum* and *Poria placenta*. Every sample was leached according to the leaching process described below and then oven-dried at 103 °C until constant weight before fungal exposure. Petri dishes (9 cm diameter) were filled with sterile culture medium prepared by mixing 30 g of malt (Malzextrakt pulverisiert from Roth) and 20 g of agar (Agar agar, dänish, pulverisiert from Roth) in distilled water (1 L), inoculated with the fungi and incubated at 25 °C and 75% relative humidity to allow full colonisation by the mycelium. Two blocks (treated and untreated as control) were aseptically inoculated in each petri dish. A total of 10 treated and untreated replicates were tested per treatment over an exposure time of 8 weeks. Only visual observations were performed.

2.2.3. Leaching procedure

Leaching was performed by the following method, adapted from NF X 41-565 French standard. Ten treated or untreated wood specimens ($5 \times 15 \times 50\ mm^3$) were placed in a flask filled with 450 mL distilled water agitated at 20 °C. The leaching water was collected after intervals of 1, 2 and 4 h. Water was changed each time and collected together (period 1). Specimens were then air-dried for 16 h before the second leaching stage, where water was collected after intervals of 8, 16 and 48 h. Water was changed each time and collected separately (period 2, period 3 and period 4, respectively). After the entire leaching process, samples were oven-dried at 103 °C until constant weight. The resultant leaching rate of the wood treatments was expressed by the following equation:

$$\mathrm{LR}(\%) = \frac{w - w_{LT}}{w - w_0} \times 100$$

where w is the treated specimen weight before leaching procedure, w_{LT} is the treated leached dried specimen weight and w_0 is the dried specimen weight before treatment. This leaching rate consists of the leached oligomer weight divided by the total oligomers weight in the sample before leaching.

2.2.4. Colour measurement

The surface colour of radial wood section was determined according to NF ISO 7724-3 European standard using a Minolta CR-200 reflectometer. The colour in the CIELAB system is characterised by three parameters, L^*, a^* and b^*. L^*, a^* and b^* colour coordinates of each group of samples were measured during the heating period. These values were then used to calculate the colour change ΔE^* as a function of heating time according to the following formula:

$$\Delta E^* = \sqrt{\left(L_f^* - L_i^*\right)^2 + \left(a_f^* - a_i^*\right)^2 + \left(b_f^* - b_i^*\right)^2}$$

where (i) and (f) are initial and final values. A low ΔE^* corresponds to a low colour change or a stable colour. Colour measurements were performed on treated solid wood cubical specimens ($20 \times 20 \times 20\ mm^3$).

2.2.5. SEM observations

Scanning electron microscopy was performed on a MEB Hitachi S-520 apparatus. Sample surfaces were coated by a gold/palladium blend in a Fisons Instruments Polaron SC7610 sputter coater.

2.3. Mechanical characterisation

Before testing, samples were conditioned at 20 °C and 65% relative humidity until their weight became stable.

2.3.1. Bending test

Bending was performed by the three-point bending test method adapted from NF B 53-643 (EN 1533) European standard using a 30 kN capacity universal test machine and applying a constant