Moisture sorption, biological durability, and mechanical performance of WPC containing modified wood and polylactates

Article *in* BioResources · August 2012

DOI: 10.15376/biores.7.4.4575-4585

3 authors:


Kristoffer Segerholm
Södra


Rebecca E. Ibach
USDA Forest Service Forest Products Laboratory


Mats Westin
RISE Research Institutes of Sweden

Proceedings IRG Annual Meeting (ISSN 2000-8953)

IRG/WP 13-40648

THE INTERNATIONAL RESEARCH GROUP ON WOOD PROTECTION

Section 4 **Processes and Properties**

Moisture and Fungal Durability of Wood-Plastic Composites Made With Chemically Modified and Treated Wood Flour

B. Kristoffer Segerholm, Rebecca E. Ibach*

KTH Royal Institute of Technology, div of Building Materials, and SP Technical Research Institute of Sweden, Stockholm, Sweden, segis@kth.se

* USDA Forest Service Forest Products Laboratory, Madison, Wisconsin, USA, ribach@fs.fed.us

Paper prepared for the 44th IRG Annual Meeting
Stockholm, Sweden
16-20 June 2013

IRG SECRETARIAT
Box 5609
SE-114 86 Stockholm
Sweden
www.irg-wp.org

Moisture and Fungal Durability of Wood-Plastic Composites Made With Chemically Modified and Treated Wood Flour

B. Kristoffer Segerholm [1], Rebecca E. Ibach [2]

[1] KTH Royal Institute of Technology, div of Building Materials, and SP Technical Research Institute of Sweden, Stockholm, Sweden, segis@kth.se

[2] USDA Forest Service Forest Products Laboratory, Madison, Wisconsin, USA, ribach@fs.fed.us

ABSTRACT

Evaluating the fungal durability of wood-plastic composites (WPCs) is complicated by the influence of slow moisture sorption. Recently, the American Wood Protection Association (AWPA) Standard Method E10, Testing Wood Preservatives by Laboratory Soil-Block Cultures, was modified to incorporate not only solid wood, but also wood-based composites and WPCs. To simulate long term WPC performance, conditioning of the specimens is now required prior to fungal exposure to increase the moisture content of the specimens. The moisture and fungal durability, as well as the mechanical properties, of two different WPCs were investigated in the laboratory following this new AWPA E10-12 Standard. Wood flour was modified with acetic anhydride and then extruded with high density polyethylene (HDPE). Wood flour was treated with an isothiazolone-based solution and then injected molded with polypropylene (PP). WPCs were conditioned by water soaking either 2 weeks at 22 ˚C or 5 days at 70 ˚C. Weight and moisture content of the WPCs were monitored. Results showed that the acetylation decreased the moisture sorption of the WPCs and showed no mass losses due to decay. The WPC with an isothiazolone-based solution did not show any mass losses due to fungal decay.

Keywords: wood-plastic composites, fungal durability, moisture resistance, acetylation, isothiazolone

1. INTRODUCTION

Wood plastic composites (WPC) are a combination of wood in the form of flour, fibers or particles, and a thermoplastic matrix. The first generations of WPC were considered to be very resistant to biological decay, and one reason for this was the slow moisture transport into the material achieved by the polymer matrix. However, the outermost layer of the composite has been shown capable of reaching moisture levels high enough to initiate biological decay (Wang and Morrell 2004; Gnatowski 2009; Ibach *et al.* 2011). To protect the composites against biological attack it is possible to increase the WPC decay resistance by manufacturing the composite with either chemically modified wood (Ibach and Clemons 2002 and 2007; Hill 2006; Segerholm *et al.* 2007; Westin *et al.* 2008) or a wood preservative treatment (Shirp *et al.* 2008).

Acetylation is a single-site reaction where acetic anhydride is reacted with wood hydroxyl groups. The resulting wood material is fixed in a swollen state and exhibits good dimensional stability and resistance to decay by fungi and microorganisms (Rowell 2006). Compression molded WPCs made with acetylated wood-fiber had better mechanical properties and water resistance than unmodified WPC controls (Glasser et al. 1999, Khalil et al. 2002). Compression molded composites of acetylated ground wood fiber and high-density polyethylene (HDPE) decreased the equilibrium moisture content (EMC) and fungal decay of the WPC compared with composites made with unmodified wood or butylene- or propylene-oxide modified wood (Ibach and Clemons 2002). Injection molded and extruded WPCs made with acetylated wood-fiber that was produced by grinding previously acetylated solid wood had decreased moisture content and were highly resistant to brown-rot decay compared with unmodified WPC controls (Westin et al. 2008). Extruded WPCs made with acetylated flour decreased the moisture content and fungal decay compared with the unmodified WPCs, even after a 2-week water soak preconditioning at 22 ˚C (Ibach and Clemons 2007).

Isothiazalone biocides hold greater than one-third of the market of biocides for plastics (Markarian 2006). They have broad anti-fungal properties, are cost effective, have lower toxicity and have better resistance to UV degradation. As a result, growth for isothiazalones have been in applications such as PVC roofing membranes, pool liners, vinyl flooring, exterior signs and interior wall coverings

WPCs have a thermoplastic-rich surface layer that is created during their processing (through extrusion, compression molding, or injection molding) that produces high levels of water repellency (Clemons and Ibach 2004). To simulate long-term field conditions in laboratory decay tests, it is necessary to expose WPCs to moisture conditions for long periods of time or at elevated temperatures, which ensures a moisture content high enough (around 25%) to support fungal growth (Ibach et al. 2004; Lopez et al. 2005; Shirp and Wolcott 2005; Manning and Ascherl 2007; Kim et al. 2008, 2009; Lomelí-Ramírez et al. 2009; Defoirdt et al. 2010; Fabiyi et al. 2011; Segerholm et al. 2012).

The objective of this study was to determine the decay resistance of WPC's made with wood flour either chemically modified (acetylated; extruded) or treated (isothiazolone-based; injection molded). Two methods of preconditioning (2 week at 22 ˚C, or 5 day at 70 ˚ water soak) were performed to accelerate the moisture sorption and ultimately the decay of the WPCs. Biological durability was evaluated through the soil block test (AWPA 2012).

2. EXPERIMENTAL METHODS

2.1 Materials extruded WPC

The wood flour used for the extruded WPCs was western pine, nominal 40 mesh (420µm), from American Wood Fibers (Schofield, Wisconsin). The HDPE was from reprocessed milk bottles (Muehlstein and Co., Inc., Roswell, Georgia) with a melt flow index of approximately 0.7 g/10min. The wood flour was modified with acetic anhydride from Aldrich Chemical Company (Milwaukee, Wisconsin). The lubricant was TPW-113 from Struktol Company of America (Stow, Ohio). It is a complex blend of modified fatty acid esters. Table 1 shows the WPC blend compositions.

2.1.1 Acetylation of wood flour

The flour was oven dried and then boiled in acetic anhydride in a 1-L glass reactor for 4 h. The treated flour was washed and oven dried, and its weight percentage gain (WPG) was calculated. Percentage acetyl content was determined using anion exchange high-performance liquid chromatography (HPLC) with a suppressed conductivity detector. Previously described method was followed (Ibach *et al.* 2000). The average acetyl content of the acetylated pine flour was 22.65 percent and the unmodified pine flour was 1.95 percent.

2.1.2 Profile extrusion

Profile extrusion was performed on a reconfigured 32-mm twin-screw extruder. All components were fed into the main feed throat. HDPE was melted and then blended with the wood flour. The material was then forced through a die into 3- by 13-mm specimens for soil block testing. Addition of 6% lubricant helped prevent tearing of the material as it exited the die.

2.2 Materials injection moulded WPC

The wood flour used for the injection moulded WPCs was scots pine sapwood, it was prepared in a two-step milling process further described in Segerholm et al. 2007, the particle size fraction 0.5-1.0 mm was used. The wood flour was either left untreated or impregnated with an isothiazolone-based solution supplied by Viance LLC resulting in approximately 700 ppm active ingredients. The polypropylene used was BE345MO from Borealis.

2.2.1 Injection molding

The blends were compounded on a twin screw extruder and granulated. The granules were then injection molded into square plates measuring 100 x 100 x 4 mm^3. Table 1 shows the WPC blend compositions.

Table 1. Composition of 4 WPC blends. All percentages are weight percent.

Wood Treatment	Processing method	Wood content [%]	Polymer [%]	Lubricant [%]
None[a]	Extrusion	50	HDPE (44)	6
Acetylated[a]	Extrusion	50	HDPE (44)	6
None[b]	Injection molded	50	PP (50)	-
Impregnation[b]	Injection molded	50	PP (50)	-

[a]Western pine, [b]Scots pine sapwood

2.3 Specimen preparation for soil block test

The extruded WPC was cut in length to 100 mm long specimens. The injection molded WPC plates were cut into strips measuring 100 x 10 x 4 mm^3.

2.4 Soil Block Test

Initial oven-dried weight was determined by drying for 24 h at 105 °C in a forced-draft oven, cooling in a desiccator for 1 h, and then weighing each specimen. Specimens were preconditioned by either water soaking for either 2 weeks at 22 ˚C, or 5 days at 70 ˚C, weighed, and moisture content was calculated. At the end of preconditioning, five specimens of each blend were air dried for 24 h, oven dried for 24 h in a forced-draft oven, cooled for 1 h in a desiccator, and then weighed. Percentage mass loss was calculated due to water/temperature conditioning.

A modified soil block test procedure based on ASTM D 1413 (ASTM 2012) and outlined in Clemons and Ibach (2004) was used to evaluate the specimens. Five replicates of each blend were autoclaved wet and then placed in horizontal soil bottles under one of two fungal exposure conditions:

1. No fungus (nf)
2. *G.trabeum,* a brown-rot fungus (br)

After 12 weeks exposure, specimens were taken out, wiped to remove fungal mycelium if present, weighed, oven dried for 24 h at 105 °C in a forced-draft oven, cooled in a desiccator for 1 h, and weighed again. Mass loss and moisture content were calculated.

3. RESULTS AND DISCUSSION

3.1 *Soil Block Test*

The percentage overall WPC mass loss and moisture content of the extruded WPC made with unmodified and acetylated wood flour specimens are in Table 2. The unmodified WPCs had high mass loss after exposure to the brown-rot fungus *G.trabeum* with both conditionings. The unmodified WPC at elevated temperature (70 °C/5 days) had 11.9% mass loss, and the room temperature (22 °C/2 weeks) had 9.5%. This is based on the entire composite weight. Assuming there is no loss of the HDPE polymer, then these figures are doubled to 23.8% and 19%, respectively, if only based on the percentage wood content. There is some mass loss just from water soaking at both temperatures, but no further loss in the sterile soil bottles with no-fungus.

For solid wood, it is necessary to keep the MC below the fiber saturation point (about 25% to 30%) to prevent fungal decay (Carll and Highley, 1999). With each water soak conditioning of the unmodified WPC specimens, the MC reached the threshold for decay to occur (25.2% and 18.9%). Based on only wood content, this equals MCs of 50.4% and 37.8%, which is well above the FSP. The acetylated WPC blend had no mass loss from decay, after subtracting out the losses from water soaking. The elevated water soak at 70 °C increased the MC of the WPCs compared to the 22 °C.

Table 2. The percentage overall WPC mass loss and moisture content of extruded WPCs made with unmodified and acetylated wood flour and HDPE.

Wood Treatment	**Water soak (°C/time)**	**Exposure**	**Mass loss [%] (stdev)**	**MC [%] (stdev)**
None	22°C /2 weeks	Brown-rot	9.5 (1.4)	19.3 (2.1)
None	22°C /2 weeks	No fungus	1.7 (0.1)	19.5 (0.7)
None	22°C /2 weeks	Just water soak	1.9 (0.1)	18.9 (0.3)
None	70°C/5 days	Brown-rot	11.9 (1.0)	19.9 (1.3)
None	70°C/5 days	No fungus	3.1 (0.1)	18.1 (0.9)
None	70°C/5 days	Just water soak	3.8 (0.1)	25.2 (0.3)
Acetylated	22°C /2 weeks	Brown-rot	1.2 (0.0)	9.1 (0.8)
Acetylated	22°C /2 weeks	No fungus	1.0 (0.0)	5.8 (0.4)
Acetylated	22°C /2 weeks	Just water soak	1.1 (0.0	8.0 (1.4)
Acetylated	70°C/5 days	Brown-rot	2.0 (0.2)	12.5 (1.5)
Acetylated	70°C/5 days	No fungus	1.7 (0.1)	8.5 (0.7)
Acetylated	70°C/5 days	Just water soak	2.0 (0.1)	14.5 (1.3)

The percentage overall WPC mass loss and moisture content of injection molded WPCs made with unmodified and isothiazolone-based solution treated wood flour and PP are in Table 3.
The isothiazolone-based solution treated WPCs had no mass losses (after subtracting out the water conditioning) compared to the injection molded unmodified WPCs. The isothiazolone-based solution treated WPC provided protection against *G.trabeum* in this soil block test compared to the unmodified WPC. The 70 °C elevated water soak increased the MC and ultimately the mass loss for the brown-rot exposure.

Overall there were lower mass losses for the injection molded WPCs compared to the extruded, which is expected (Clemons and Ibach, 2004). There is more mass loss from the elevated temperature water soak in both the extruded and injection molded composites. This may be due to water soluble extractives and/or hemicelluloses, but further analysis would be required.

Table 3. The percentage overall WPC mass loss and moisture content of injection molded WPCs made with unmodified and isothiazolone-based solution treated wood flour and PP.

Wood Treatment	**Water soak (°C/time)**	**Exposure**	**Mass loss [%] (stdev)**	**MC [%] (stdev)**
None	22°C /2 weeks	Brown-rot	1.9 (0.2)	10.5 (0.1)
None	22°C /2 weeks	No fungus	0.6 (0.1)	10.9 (0.2)
None	22°C /2 weeks	Just water soak	0.9 (0.0)	3.1 (0.0)
None	70°C/5 days	Brown-rot	4.1 (0.5)	13.8 (0.2)
None	70°C/5 days	No fungus	2.0 (0.0)	14.8 (0.2)
None	70°C/5 days	Just water soak	1.8 (0.0)	13.8 (0.1)
Impregnation	22°C /2 weeks	Brown-rot	0.8 (0.0)	10.6 (0.1)
Impregnation	22°C /2 weeks	No fungus	0.7 (0.0)	10.5 (0.1)
Impregnation	22°C /2 weeks	Just water soak	0.9 (0.0	3.0 (0.1)
Impregnation	70°C/5 days	Brown-rot	2.0 (0.1)	13.3 (0.1)
Impregnation	70°C/5 days	No fungus	1.9 (0.0)	13.3 (0.2)
Impregnation	70°C/5 days	Just water soak	1.8 (0.0)	11.9 (0.1)

4. CONCLUSIONS

- Acetylated and isothiazolone-based solution treated WPCs improved the decay resistance compared to unmodified WPCs.
- Conditioning at elevated temperature (70 °C) for 5 days increased the moisture content and accelerated the fungal decay of the WPCs compared to room temperature (22 °C) for 2 weeks.
- Decay rate was dependent upon processing method. Extruded composites had higher overall mass losses than injection molded.

5. ACKNOWLEDGEMENTS

EcoBuild Institute Excellence Centre established by VINNOVA, The Knowledge Foundation and the Swedish Foundation for Strategic Research is greatly acknowledged.

6. REFERENCES

American Society of Testing and Materials D 1413. 2012. Annual Book of ASTM Standards, Section 4 Construction, Volume 04.10 Wood. ASTM International, West Conshohocken, Pennsylvania.

American Wood Protection Association Standard Method E10. 2012. AWPA Book of Standards. Birmingham, Alabama.

Carll, C.G. and Highley, T.L. 1999. Decay of wood and wood-based products above ground in buildings. J. Test. Eval. 27(2): 150-158.

Clemons, C.M. and Ibach, R.E. 2004. Effects of processing method and moisture history on laboratory fungal resistance of wood-HDPE composites. Forest Products Journal 54(4): 50-57.

Defoirdt, N., S. Gardin, J. Van den Bulcke, and J. Van Acker. 2010. Moisture dynamics of WPC and the impact on fungal testing. Intl. Biodeterioration Biodegradation 64:65–72.

Fabiyi, J.S., A.G. McDonald, J.J. Morrell, and C. Freitag. 2011. Effects of wood species on durability and chemical changes of fungal decayed wood plastic composites. Composites Part A: Appl.Sci.Manufacturing 42:501–510.

Glasser, W.G., et al. 1999. Fiber-reinforced cellulosic thermoplastic composites. J. Appl. Polymer Sci. 73(7):1329-1340.

Gnatowski, M. 2009. Water Absorption and Durability of Wood Plastic Composites. In: Stark, N. (Ed.), Proceedings of the 10th International Conference on Wood & Biofiber Plastic Composites. Madison, WI. USA, pp. 90-109.

Hill, C. A. S. 2006. Wood modification: chemical, thermal and other processes. John Wiley & Sons Ltd. West Sussex, England.

Ibach, R. E., Gnatowski, M., and Hui. G. 2011. Laboratory and field evaluations of the decay resistance of WPC. Proceedings of the 11th International Conference on Wood & Biofiber Plastic Composites, Madison, WI, USA.

Ibach, R. E. and Clemons, C. M. 2002. Biological Resistance of Polyethylene Composites Made With Chemically Modified Fiber or Flour, In: Humphrey, P. (Ed.), Proceedings of the 6th Pacific Rim Bio-Based Composites Symposium, Portland, Oregon. Vol. 2, pp. 574-583

Ibach, R. E. and Clemons, C.M. 2007. Effect of acetylated wood flour or coupling agent on moisture, UV, and biological resistance of extruded woodfiber-plastic composites, In: Barnes, H.M. (Ed.), Wood Protection, Forest Products Society, Madison, WI, pp. 139-147.

Ibach, R.E., Clemons, C.M., and Stark, N.M. 2004. Combined Ultraviolet and Water Exposure as a Preconditioning Method in Laboratory Fungal Durability Testing. In: Seventh International Conference on Woodfiber-Plastic Composites. Madison, Wisconsin: Forest Products Society.

Ibach, R.E., Rowell, R.M. and Lee, B.-G. 2000. Decay protection based on moisture exclusion resulting from chemical modification of wood. In: 5th Pacific Rim Bio-Based Composites Symposium. Canberra, Australia.

Khalil, H.P.S.A., Rozman, H.D., Ismail, H., Rosfaizal. and Ahmad, M.N. 2002. Polypropylene (PP)-Acacia Mangium composites: The effect of acetylation on mechanical and water absorption properties. Polymer-Plastics Technol. Engineering 41(3):453-468.

Kim, J.-W., Harper, D.P. and Taylor, A.M. 2008. Effect of wood species on water sorption and durability of wood–plastic composites. Wood Fiber Sci. 40:519–531.

Kim, J.-W., Harper, D.P., and Taylor, A.M. 2009. Effect of extractive on water sorption and durability of wood–plastic composites. Wood Fiber Sci. 41:279–290.

Lopez, J.L., Cooper, P.A., and Sain, M. 2005. Evaluation of proposed test methods to determine decay resistance of natural fiber plastic composites. Forest Products Journal. 55:95–99.\

Lomelí–Ramírez, M.G., H.G. Ochoa–Ruiz, F.J. Fuentes–Talavera, S. García–Enriquez, M.A. Cerpa–Gallegos, and J.A. Silva–Guzmán. 2009. Evaluation of accelerated decay of wood plastic composites by Xylophagus fungi. Intl. Biodeterioration Biodegradation 63:1030–1035.

Manning, M.J., and Ascherl F. 2007. Wood–plastic composite durability and the compelling case for field testing. In: 9th International Conference on Wood & Biofiber–Plastic Composites. Forest Products Society, Madison, Wisconsin. pp. 217–224.

Markarian, J. 2006. Steady growth predicted for biocides. Plastics Additives &Compounding. January/February pp. 30-33.

Rowell, R. M. 2006. Acetylation of wood: A journey from analytical technique to commercial reality. Forest Products Journal 56, 4-12.

Segerholm, B.K., Ibach, R.E. and Wålinder, M.E.P. 2012. Moisture sorption in artificially aged wood–plastic composites. Bioresources.com 7:1283–1293.

Segerholm, B. K., P. Walkenström, B. Nyström, M. E. P. Wålinder and P. Larsson Brelid. 2007. Micromorphology, moisture sorption and mechanical properties of a biocomposite based on acetylated wood particles and cellulose ester. Wood Material Science and Engineering, 2(3-4), 106–117.

Shirp, A., Ibach, R. E., Pendleton, D. E., Wolcott, M. P. 2008. Biological degradation of wood-plastic composites (WPC) and strategies for improving the resistance of WPC against biological decay. In: Schultz, T.P. et al. (Eds.), Development of commercial wood preservatives. ACS symposium series, 982, pp. 480-507.

Shirp, A., and Wolcott, M.P. 2005. Influence of fungal decay and moisture absorption on mechanical properties of extruded wood-plastic composites. Wood Fiber Sci. 37(4):643-652.

Wang, W., Morrell, J. J. (2004). Water sorption characteristics of two wood-plastic composites. Forest Products Journal 54, 209-212.

Westin, M., Larsson Brelid, P., Segerholm, B. K., Van den Oever, M. 2008. Wood Plastic Composites from modified wood. Part 3. Durability of WPCs with bioderived matrix. IRG/WP 08-40423. The International Research Group on Wood Protection, Stockholm.