

EFFECTS OF ELLAGIC ACID AND DERIVATIVES THEREOF ON WATER REPELLENCY AND DIMENSIONAL STABILITY

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PRZEWLOKA, S. R. & SHEARER, B. J. 2008. Effects of ellagic acid and derivatives thereof on water repellency and dimensional stability. Derivatives of the insoluble polyphenolic ellagic acid and water soluble tetraguanidinium, tetracholine, tetrakis-benzyltrimethylammonium and tetrakis-tetramethylammonium ellagates and 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl, were prepared. The effects of these compounds upon water repellency and dimensional stability when impregnated into timber were examined. Water soluble ellagates improved water repellent effectiveness up to 32%. Ellagic acid and 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl increased initial swelling rates due to their solvent carriers. All trial compounds provided the timber wafers with additional dimensional stability predominantly by non-bonded, non-leachable bulking. Generally a reduction in the extent but not the rate of moisture uptake was noted for the compounds investigated.

Keywords: Water repellency, dimensional stability, ellagic acid, *Pinus radiata*

PRZEWLOKA, S. R. & SHEARER, B. J. 2008. Kesan asid elagik dan terbitannya terhadap rintangan air dan kestabilan dimensi. Terbitan asid elagik polifenol tak larut serta tetraguanidinium larut air, tetrakolin larut air, elegat tetrakis-benziltrimetilamonium larut air, elegat tetrakis-tetrametilamonium larut air dan 2,3,4,2',3',4'-heksametoksi-6,6'-diisocyanato-difenil larut air disediakan. Kami mengkaji kesan sebatian ini terhadap rintangan air dan kestabilan dimensi apabila ia diisi tepu ke dalam kayu. Elegat larut air meningkatkan keberkesanan rintangan air sebanyak 32%. Asid elagik dan 2,3,4,2',3',4'-heksametoksi-6,6'-diisocyanato-difenil meningkatkan kadar pengembangan awal kerana pembawa yang terdapat dalam pelarutnya. Semua sebatian yang diuji menambah kestabilan dimensi wafer kayu terutamanya kerana sebatian tersebut tidak terikat kepada wafer dan tidak pula terlarut resap. Umumnya sebatian yang dikaji menunjukkan pengurangan takat kelembapan walaupun kadar pengambilan kelembapannya tidak berubah.

INTRODUCTION

Wood is an excellent material for numerous building applications but its exterior use suffers from its hygroscopic nature, causing the wood to swell when wet and shrink when dry (Borgin 1961). Wood is an anisotropic material; it swells and shrinks to different degrees in the three anatomical directions—longitudinally, tangentially and radially (Rowell & Banks 1985). Repeated changes in the moisture content of timber lead to continual deterioration (warping, twisting, bending, checking and splitting). Wood-water interaction can be reduced by preventing or controlling the rate of liquid water uptake (using water repellents) or the extent of wood swelling at equilibrium conditions (dimensional stabilizers).

Water repellents and dimensional stabilizers are described as one of three types (Figure 1) (Rowell & Banks 1985). A treatment may improve water repellency but not dimensional stability (Type I) where the rate of moisture uptake is reduced but at equilibrium the extent of swelling is almost the same for the treated and untreated wood. Improvement in dimensional stability but not water repellency (Type II) shows a reduction in the extent of swelling but not the rate at which moisture is taken up. When a treatment reduces both the rate and the extent of swelling (Type III) both water repellency and dimensional stability are improved.

Water repellents are applied to wood to prevent or reduce the rate of water flow into the

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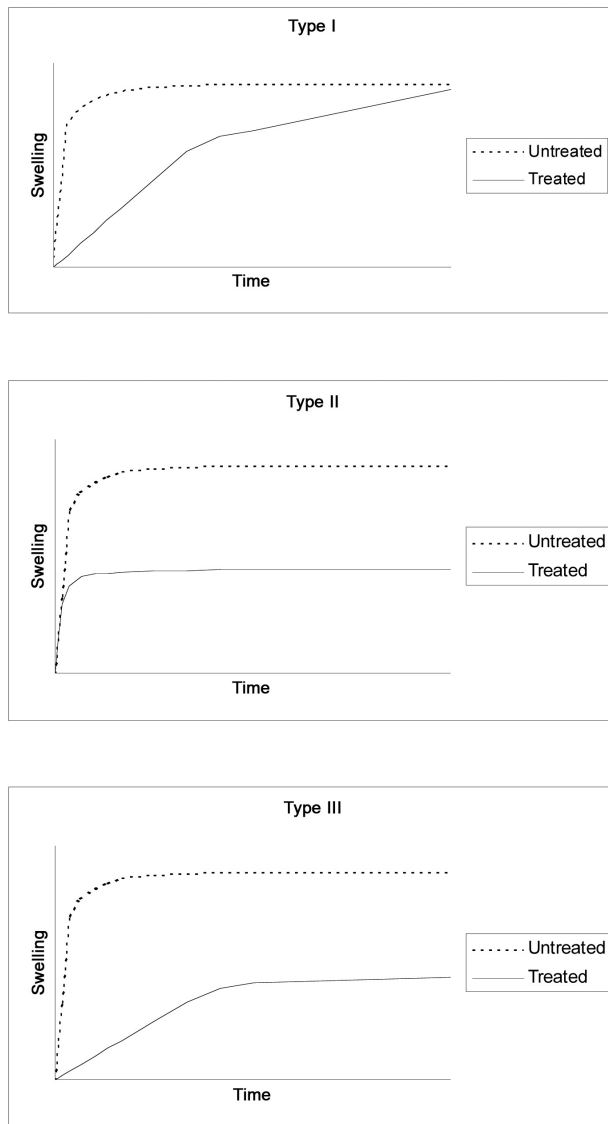


Figure 1 Treatment of wood for water repellency and dimensional stability (Rowell & Banks 1985)

cellular structure. An effective water repellent has two modes of action, it prevents water being taken up by the capillary system and it renders the cell walls inert to water. Water is kept out of the capillary system by coating the entire wood surface or by impregnating it with a hydrophobic material deposited in such a way that water uptake is hindered (Borgin 1961).

The effectiveness of a water repellent is dependent upon a number of parameters including method of treatment, carrier solvent, wood species and preservative distribution. Variables affecting the test methodology include water temperature and purity (Banks 1971), sample dimensions, original wood moisture content, sorption hysteresis and simulated weathering (Vinden 1996).

Water soak testing is generally employed to determine the dimensional stability of treated samples. As dimensional stability is based upon comparison of untreated and treated samples, the treated sample must be from the same source as the control. Specimen size and geometry are important in dimensional stability trials. The tangential dimension changes the most during swelling and shrinking; specimens are thus prepared with maximum tangential and minimal longitudinal dimensions.

Dimensional stabilization by chemical treatment can include moisture uptake reduction, chemical crosslinking and bulking (Rowell & Banks 1985). Treatment of wood with chemicals (anhydrides, isocyanates, acid chlorides, aldehydes and epoxides) that are not merely deposited in the wood but undergo reactions with wood hydroxyl groups is desirable. Alternatively, heat treatment reduces the hygroscopicity by thermal degradation of the hemicellulose component of the wood cell wall (Stamm *et al.* 1946, Seborg *et al.* 1953).

Bulking treatments improve stability by occupying sites within the wood cell wall to the exclusion of water (Rowell & Banks 1985). Waxes are commonly incorporated in composite panel manufacture as bulking agents that also alter the surface chemistry of the wood (Zhang *et al.* 2007). Wood volume increases are directly proportional to the theoretical volume of chemical added (Rowell & Ellis 1978). When wood modified by cell wall bulking comes into contact with water, little additional swelling (dimensional instability) occurs. The three classes of bulking treatments are (a) non-bonded and leachable, (b) non-bonded and non-leachable and (c) bonded and non-leachable (Rowell & Banks 1985).

The effects of a number of chemical treatments upon water repellency and dimensional stability were investigated. Based upon its ready availability as a pulping waste by-product and its insoluble nature, ellagic acid as well as its derivatives thereof (numerous water soluble ellagates (Przewlaka & Shearer 2002) and a diisocyanate (Reitze *et al.* 2001)) were examined. The effectiveness of ellagic acid as an antioxidant has been widely reported (Gali *et al.* 1992, Ramanathan & Das 1992, 1993), as is its inhibition of wood destroying decay fungi (Olteanu 1997) and insect pests (Klocke *et al.* 1986, Jones & Klocke 1987). It was envisaged that these compounds may also provide biological protection to the timber. The

benefit of incorporating a combined preservative and water-repellent treatment throughout the wood is that the material can be cut or resurfaced without performance loss, and water repellency would not be limited to the outer wood surface which is subject to maximum exposure and weathering (Zahora 1991).

Treatment of wood with water soluble ellagates will allow ellagate anions to enter the wood cell wall and precipitate by hydrolysis as ellagic acid (Przewloka & Shearer 2002). Ellagic acid is not soluble in water and is, therefore, non-leachable. Interaction with cellulose to the exclusion of water may also occur (by hydrogen bonding with free cellulose hydroxyl groups). Use of water as the solvent is advantageous due to its lack of toxicity and ability to swell the cell wall. The latter contributes to a larger uptake of preservative resulting in a more evenly distributed treatment (Svensson *et al.* 1987).

Ellagic acid in *N*-methylpyrrolidone may provide non-bonded and non-leachable bulking by precipitation of the insoluble free acid within the cell wall. The ellagic acid molecule is sufficiently small to penetrate the cell wall. The polar nature of the *N*-methylpyrrolidone should allow some swelling of the cell wall. Swelling with *N*-methylpyrrolidone is not anticipated to be of the same magnitude compared with swelling with water, as the larger size of *N*-methylpyrrolidone may reduce the solvents accessibility to the cellulose. No chemical crosslinking is expected.

It is hypothesized that 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl will react with the hydroxyl groups of the cellulose and decrease hygroscopicity via bonded and non-leachable bulking of the wood cell. The number of free hydroxyl groups available to hydrogen bond with water will thus be reduced. The two reactive isocyanate groups of 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl may

also give rise to crosslinking. Dioxane, the diisocyanate solvent carrier, has been shown to cause 62% relative swelling of wood cell walls compared with water (Stamm 1964). Therefore, it is expected that swelling of the cell wall will occur allowing greater interaction between cellulose and the diisocyanate.

MATERIALS AND METHODS

Anal grade chemicals were used. The purity of ellagic acid was > 98%. Tetraguanidinium, tetracholine, tetrakis-benzyltrimethylammonium and tetrakis-tetramethylammonium ellagates were prepared according to the methods described by Przewloka and Shearer (2002). 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl was synthesized by the procedure of Reitze *et al.* (2001).

Wafers 6.5 (longitudinal) × 25 (radial) × 50 mm (tangential) were cut from kiln-dried flatsawn boards of fast grown *Pinus radiata* supplied by Central Highlands Timber, Ballarat, Victoria, Australia. Prior to treatment, all samples were conditioned for three weeks (25 °C and 65% relative humidity) to constant weight with a moisture content of approximately 12%.

The test specimens were treated by a modified Bethel process consisting of a 30 min initial vacuum (-85 kPa) followed by a soak period of 1 hour, after which the treatment solution was removed. A final vacuum (-85 kPa) was applied for 15 min to remove excess treatment solution. Three concentrations (5, 1 and 0.1%) of each trial compound were examined in triplicate. Loadings are summarized in Table 1. Following treatment, the wafers were oven dried at 105 °C for 24 hours and reweighed. The treated wafers were then reconditioned for three weeks (25 °C and 65% relative humidity) to constant weight to allow chemical fixation.

Table 1 Mean preservative retention (kg/m³) of wafers

| Treatment | Retention (kg/m ³)* | | |
|--|---------------------------------|-------------|---------------|
| | 5% solution | 1% solution | 0.1% solution |
| Tetraguanidinium ellagate in water | 28.1 (0.8) | 5.7 (1.1) | 0.6 (4.4) |
| Tetracholine ellagate in water | 29.7 (0.6) | 5.9 (0.7) | 0.6 (0.8) |
| Tetrakis-benzyltrimethylammonium ellagate in water | 30.5 (0.5) | 5.9 (0.2) | 0.6 (1.4) |
| Tetrakis-tetramethylammonium ellagate in water | 29.5 (0.9) | 6.1 (1.0) | 0.6 (0.8) |
| Ellagic acid in <i>N</i> -methylpyrrolidone | 29.5 (2.3) | 6.1 (1.3) | 0.6 (0.7) |
| 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl in dioxane | 31.2 (2.1) | 6.3 (1.2) | 0.6 (0.8) |

*Percent variance in parentheses

Solvent uptakes for water (593 l/m³), *N*-methylpyrrolidone (601 l/m³) and dioxane (606 l/m³) were noted.

The standard method of the American Wood-Preservers' Association (1990) (M18-78) was used. Water repellency was determined by examining the tangential swelling and mass increase (measured to the nearest 0.0005 g) of the treated wafers after each sample had been immersed in distilled water at 24 °C for 0, 0.5, 1, 2, 4, 6, 8, 10, 15, 20, 25, 30 and 60 min. Dimensional measurements were taken (IGP Industrial digital calliper rule accurate to 0.005 mm) immediately after the wafers were removed from the water. Fresh distilled water was used for each wafer.

RESULTS AND DISCUSSION

Water repellency

Water repellent effectiveness (WRE) is usually expressed as a percentage reduction compared with an untreated control (Rowell & Banks 1985). All WRE values (Table 2) reported were based upon the %WRE calculation (Rowell & Banks 1985) outlined in Equation 1:

$$\%WRE = \frac{D_c - D_t}{D_c} \times 100 \quad (1)$$

where

D_c = swelling (or water uptake) of control during exposure in water for 't' min

D_t = swelling (or water uptake) of treated specimen, also for 't' min.

A significant difference in %WRE for treated and untreated wafers was found using Student's *t*-test ($\alpha = 0.05$, two-tailed). Treatment of wafers by water soluble ellagates improved WRE (Table 2) with values between 18 (for

1% tetraguanidinium ellagate treatment) and 32% (5% tetrakis-benzyltrimethylammonium ellagate). However, ANOVA test ($\alpha = 0.05$, single factor) demonstrated that the three concentrations of water soluble ellagates had no significant effect upon WRE. There was also no significant difference in WRE between each water soluble ellagate treatment.

Tetrakis-benzyltrimethylammonium (32, 25 and 24% for 5, 1 and 0.1% solutions respectively) and tetrakis-tetramethylammonium (31, 25 and 25%) ellagate treatments provided the greatest WRE. WRE values of 24, 25 and 22% respectively were noted for 5, 1 and 0.1% solutions of tetracholine ellagate. The smallest improvements in WRE were observed for tetraguanidinium ellagate treated wafers (24, 18 and 22%). Water soluble ellagates provided water repellency by non-bonded and non-leachable bulking of the cell walls.

Wafers treated with ellagic acid in *N*-methylpyrrolidone exhibited no improvement in water repellency. WRE values of -13 and -18% respectively in wafers treated by 5 and 1% ellagic acid solutions were observed. However, a WRE of 6% was observed for 0.1% ellagic acid treatment.

Consideration of the solvent system is necessary in comparing these results. Wafers treated with only *N*-methylpyrrolidone displayed a -5% WRE. Given %WRE was reasonably uniform regardless of the concentrations of water soluble ellagates in the wafers, similar trends may be expected for the ellagic acid treatment. If the result for the 0.1% solution is regarded as questionable (6% compared with -13 and -18%), then it may be postulated that wafer treatment by *N*-methylpyrrolidone increases initial swelling rate.

Table 2 Water repellent effectiveness (%WRE) of treated wafers

| Treatment | Water repellent effectiveness (%WRE)* | | |
|--|---------------------------------------|-------------|---------------|
| | 5% solution | 1% solution | 0.1% solution |
| Tetraguanidinium ellagate in water | 24 (13.0) | 18 (12.2) | 22 (11.9) |
| Tetracholine ellagate in water | 24 (8.4) | 25 (12.4) | 22 (13.1) |
| Tetrakis-benzyltrimethylammonium ellagate in water | 32 (3.7) | 25 (10.9) | 24 (11.8) |
| Tetrakis-tetramethylammonium ellagate in water | 31 (5.0) | 25 (10.4) | 25 (9.5) |
| Ellagic acid in <i>N</i> -methylpyrrolidone | -13 (12.9) | -18 (10.8) | 6 (10.4) |
| 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl in dioxane | -5 (4.1) | -17 (9.6) | -19 (7.7) |

*Percent variance in parentheses

The water repellent effectiveness of the *N*-methylpyrrolidone and dioxane blanks were -5 (7.1%) and -3 (12.0%) respectively.

Although the addition of ellagic acid to the system further amplified swelling by between 8 and 13%, a Student's *t*-test ($\alpha = 0.05$, two-tailed) illustrated that there was no significant change in %WRE when ellagic acid was added to *N*-methylpyrrolidone. An analysis of variance ($\alpha = 0.05$, single factor) established that no significant differences in WRE can be attributed to ellagic acid concentration. Ellagic acid in *N*-methylpyrrolidone is thus unsuitable as a water repellent.

Wafer treatment with 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl produced results comparable with ellagic acid treatment with WRE values of -5, -17 and -19% for 5, 1 and 0.1% treatment solutions respectively.

No obvious concentration gradient effect could be discerned with the diisocyanate treatment. Dioxane caused an increase in the initial swelling rate and Student's *t*-test ($\alpha = 0.05$, two-tailed) verified that the addition of diisocyanate to the system did not significantly affect this swelling. An analysis of variance ($\alpha = 0.05$, single factor) showed that concentration of the diisocyanate also had no significant effect upon %WRE. The 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl in dioxane imparted no water repellency to the treated wafers.

The solvents *N*-methylpyrrolidone and dioxane may both be responsible for the redistribution and/or removal of extractives during impregnation. Analysis of the solvent extracts would elucidate the extent of redistribution and removal.

Dimensional stability

Dimensional stability, or the extent of change in wood dimensions at equilibrium as a result

of moisture uptake, can be measured as a single dimensional component (tangential, radial or longitudinal) but is typically measured by taking into account all three dimensional changes (volumetrically). Dimensional stability values (Table 3) were determined according to Equations 2, 3 and 4 (Rowell & Banks 1985):

$$S = \frac{V_2 - V_1}{V_1} \times 100 \quad (2)$$

$$S = \frac{V_2 - V_1}{V_2} \times 100 \quad (3)$$

$$\%ASE = \frac{S_1 - S_2}{S_1} \times 100 \quad (4)$$

where

S = volumetric swelling or shrinking coefficient

V_1 = wood volume of an oven dried sample before conditioning or wetting

V_2 = wood volume after humidity conditioning or wetting with water

ASE = reduction in swelling or shrinking efficiency resulting from a treatment

S_1 = untreated volumetric swelling or shrinking coefficient

S_2 = treated volumetric swelling or shrinking coefficient.

A Student's *t*-test ($\alpha = 0.05$, two-tailed) demonstrated that wafer treatment by ellagat derivatives significantly affected swelling, improving dimensional stability at all three concentration levels tested. However, for all compounds examined there was no significant

Table 3 Dimensional stability (%ASE) of treated wafers

| Treatment | Dimensional stability (%ASE)* | | |
|--|-------------------------------|-------------|---------------|
| | 5% solution | 1% solution | 0.1% solution |
| Tetraguanidinium ellagate in water | 31 (9.4) | 25 (8.4) | 25 (9.1) |
| Tetracholine ellagate in water | 40 (9.5) | 29 (5.7) | 27 (12.8) |
| Tetrakis-benzyltrimethylammonium ellagate in water | 30 (12.2) | 33 (7.6) | 29 (8.8) |
| Tetrakis-tetramethylammonium ellagate in water | 42 (10.8) | 32 (8.6) | 31 (10.6) |
| Ellagic acid in <i>N</i> -methylpyrrolidone | 25 (12.8) | 21 (15.7) | 18 (13.6) |
| 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl in dioxane | 25 (9.7) | 24 (10.2) | 20 (13.4) |

*Percent variance in parentheses

The dimensional stability of the *N*-methylpyrrolidone and dioxane blanks were 19 (13.3%) and 30 (2.0%) respectively.

difference in %ASE attributable to solution type or solution concentration (ANOVA $\alpha = 0.05$, single factor).

Improvements in antismelling efficiency of between 25 (1 and 0.1% solutions of tetraguanidinium ellagate) and 42% (5% solution of tetrakis-tetramethylammonium ellagate) were noted for the water soluble ellagates trialed. Tetracholine and tetrakis-tetramethylammonium ellagates were the most effective dimensional stabilizers, improving antismelling efficiency by 40, 29, 27% and 42, 32, 31% respectively for treatment with 5, 1 and 0.1% solutions. Tetraguanidinium and tetrakis-benzyltrimethylammonium ellagate treatments resulted in antismelling efficiencies of 31, 25, 25% and 30, 33, 29% respectively for the 5, 1 and 0.1% solutions.

Improvement of dimensional stability was also achieved by treating wafers with ellagic acid in *N*-methylpyrrolidone. Swelling reductions of 25, 21 and 18% were noted for 5, 1 and 0.1% test solutions respectively. Wafers treated with the solvent carrier only, *N*-methylpyrrolidone, also exhibited reduction in swelling (19%).

It is proposed that *N*-methylpyrrolidone is responsible for imparting dimensional stability upon the wood by hydrogen bonding to the reactive hydroxyl sites of the cellulose, hemicellulose and lignin, thus reducing the hydroscopicity of the wood (therefore reducing water adsorption and swelling (Stamm 1964)). Ellagic acid may slightly increase the magnitude of this effect when present in the system but this effect is not significant (Student's *t*-test, $\alpha = 0.05$, two-tailed).

Similar trends were noted for ellagic acid treatment when wafers were treated with 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl in dioxane. Antismelling efficiencies of 25, 24 and 20% were achieved for 5, 1 and 0.1% solutions of the diisocyanate respectively. However, wafers treated with only dioxane exhibited an antismelling efficiency of 30%.

This result would suggest that dioxane treatment improves dimensional stability and the presence of the diisocyanate in the system slightly reduces the extent of the stabilization. It is most likely that dioxane is indeed responsible for the dimensional stability observed, but the presence of 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl has no significant effect

upon dimensional stability (Student's *t*-test, $\alpha = 0.05$, two-tailed).

The additional dimensional stability provided by the test compounds is most likely due to non-bonded and non-leachable bulking of the cell walls.

CONCLUSIONS

Water soluble tetraguanidinium, tetracholine, tetrakis-benzyltrimethylammonium and tetrakis-tetramethylammonium ellagates impart some %WRE. Ellagic acid in *N*-methylpyrrolidone and 2,3,4,2',3',4'-hexamethoxy-6,6'-diisocyanato-diphenyl in dioxane increased initial swelling rates, most probably due to the solvent carriers.

All trial compounds provided the timber wafers with moderate levels of additional dimensional stability (%ASE), most probably by non-bonded, non-leachable bulking of the cell walls.

The trial compounds displayed similar properties to Type II (Rowell & Banks 1985) water repellents and dimensional stabilizers. Generally a reduction in the extent of swelling, but not the rate at which moisture was taken up, was noted.

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