

## STUDY OF DIMENSIONAL STABILITY, DECAY RESISTANCE, AND LIGHT STABILITY OF PHENYLISOTHIOCYANATE MODIFIED RUBBERWOOD

Krishna K. Pandey\*, Jayashree, and H. C. Nagaveni

Rubber wood (*Hevea brasiliensis*) was esterified with phenylisothiocyanate, and dimensional stability, decay resistance, and photo stability of the modified wood was assessed. The chemically modified wood was characterized by FTIR and CP/MAS <sup>13</sup>C NMR spectroscopy. Unmodified and modified samples were exposed to a brown rot (*Polyporus meliae*) and a white rot (*Coriolus versicolor*) fungus for 12 weeks. Modified wood samples exhibited good dimensional stability and were very resistant to decay. However, phenylisothiocyanate modification of wood was not effective in decreasing photo-yellowing.

*Keywords:* Dimensional stability; Phenylisothiocyanate; Photodegradation; Weathering; Lignin; Chemical modification; Wood

*Contact information:* Institute of Wood Science and Technology, 18<sup>th</sup> Cross Malleswaram, Bangalore-560003, India; \*Corresponding author: kkpandey@icfre.org; krishna\_pandey@indiatimes.com

### INTRODUCTION

During exterior use, wood constituent polymers are readily degraded by weathering. The main problems associated with wood under outdoor use are its dimensional instability due to moisture absorption/desorption, breakdown of wood polymers by UV light, and its decay by microorganisms (Rowell 1983; Feist and Hon 1984; Williams 2005; Rowell 2005).

Wood is a 3-dimensional polymeric composite made of cellulose, hemicelluloses, and lignin. These three polymers constitute the cell wall and are responsible for the physical and chemical properties exhibited by the wood. All of these wood polymers have accessible hydroxyl groups. Wood attracts moisture through hydrogen bonding, making it dimensionally unstable, which in turn promotes biological degradation. Phenolic groups present in lignin are primarily involved in the UV light absorption leading to its photochemical degradation.

Chemical modification of cell wall polymers is one of the effective methods to induce dimensional stability, UV resistance, and biological resistance in wood (Rowell 1983; Feist and Hon 1984; Plakette et al. 1996; Matsuda 1996; Takahashi 1996; Evans et al. 2000; Chang and Chang 2001; 2006; Williams 2005; Rowell 2005; Rowell 2006; Hill 2006). All of these properties of wood are a result of chemical degradation reactions which can be prevented or, at least, slowed down if the cell wall chemistry is altered (Rowell 2005). The dimensional stability can be improved by bonding cell wall polymers with hydrophobic groups or bulking cell wall polymers with bonded chemicals. Resistance to ultraviolet radiation can be achieved by bonding UV absorbers or blockers

to lignin (Kiguchi and Evans 1998). The improved dimensional stability is obtained generally by replacing hydrophilic hydroxyl groups of wood constituents by more hydrophobic groups through either esterification with chemicals such as acid anhydrides, acid chlorides, isocyanates, or etherification with alkylene oxides. Some of these reaction systems have been found to be very effective. Wood modified with acetic anhydride, propylene and butylene oxide, and methyl isocyanate exhibit very good dimensional stability, acetylated wood being the most stable (Rowell 2005). The extent of dimensional stability increases with increasing weight percent gain (WPG) and attains an optimum value around 20-30 WPG and then decreases with further modification (Rowell 2005). The decay resistance of chemically modified wood has been tested for different types of organisms including termites (Takahashi 1996; Rowell 2005), decay fungi (Nilsson et al. 1988; Rowell et al. 1989; Imamura et al. 1989; Chen 1992), and marine borers ((Johnson and Rowell 1988). Chemically modified wood has also been shown to improve ultraviolet resistance of wood (Feist et al. 1991a, 1991b; Hon 1995; Plakette et al. 1996; Evans et al. 2000, 2002; Chang 2001a; 2001b; 2006; Pandey and Chandrashekar 2006; Prakash et al. 2006).

In this work we have studied weathering properties (dimensional stability, UV and fungal resistance) of rubberwood modified with phenylisothiocyanates. The aim of this research was to investigate the possibility of protection of wood by reacting wood with phenylisothiocyanates. Phenylisothiocyanates have been shown to provide good decay resistance (Chem 1992). Dimensional stability and UV resistance of phenylisothiocyanate-modified wood have not been evaluated. The scheme for reaction of phenylisothiocyanate with wood is shown in Fig 1.



Fig. 1. Scheme for reaction between phenylisothiocyanate and wood

## EXPERIMENTAL

### Synthesis of Phenylisothiocyanate

Phenylisothiocyanate was synthesized in the laboratory with aniline and carbon disulphide using method described by Furniss et al. (1991). The reaction scheme for synthesis of phenylisothiocyanates is shown in Fig. 2.

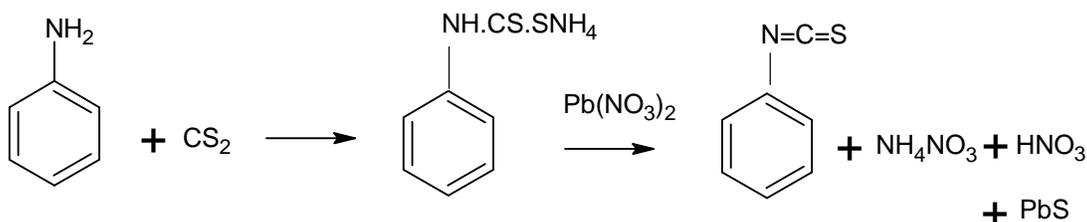


Fig. 2. The reaction scheme for synthesis of phenylisothiocyanate

The synthesized compound was dried on anhydrous magnesium sulphate and stored in an air-tight bottle containing molecular sieves.

### Wood Specimens

Specimens of rubberwood of size 20 × 20 × 5 mm (radial × tangential × longitudinal) were prepared from defect-free wood. Samples were extracted for six hours in toluene:ethanol:acetone mixture (4:1:1) in a Soxhlet extractor. Extracted samples were dried at 105 °C for 24 hours, and the oven dried-weight was determined.

### Chemical Modification of wood Specimens

Oven-dried samples were immersed in moisture-free pyridine in a reaction vessel for 20h in an air-tight container. The glass container was fixed with a water-cooled condenser. At the top of the condenser a glass bulb was fixed, filled with fused calcium chloride to provide moisture free atmosphere, and transferred in to an oil bath to maintain constant temperature. The reaction was carried out at three different temperatures i.e., 85 °C, 100 °C, and 120 °C up to 24 hours. The samples were then removed and soaked in cold acetone to arrest the reaction. Modified wood blocks were subsequently extracted with acetone:toluene (1:1) to remove un-reacted reagents and oven dried at 105 °C for 24 hours. The Weight Percent Gain (*WPG*) of specimens was calculated using eqn. 1,

$$WPG = [(W_t - W_o) / W_o] \times 100 \quad (1)$$

where  $W_o$  and  $W_t$  are oven dried weight of unmodified and chemically modified wood samples, respectively.

### Measurement of FTIR and CP/MAS <sup>13</sup>C NMR Spectra

FTIR spectra were obtained using a Nicolet Impact 400 FTIR spectrometer. The top surface of the treated samples was removed using a razor blade and analyzed by direct transmission (KBr pellet technique). NMR spectra were obtained using a Bruker DSX 300MHz, CP/MAS <sup>13</sup>C NMR spectrometer.

### Estimation of Dimensional Stability

The degree of Dimensional stability was determined by estimating the volumetric swelling coefficient (*S*) and anti-shrink /anti-swell efficiency (*ASE*), using the repeated water-soaking method described by Rowell and Ellis (1978). Samples were submersed in distilled water evacuated in vacuum desiccators. The vacuum was applied for 30 min, then released for 1 hour, reapplied for 30 min, and then released for 24 hours. Excess water was drained, and the weights of the specimens were determined. Volumes were measured by the mercury displacement method. The soaking process was continued until samples attained a constant volume. Specimens were again oven-dried, and their weight and volumes were determined. The volumetric swelling coefficient (*S*) was determined using eqn. 2,

$$S (\%) = 100 (V_2 - V_1) / V_1 \quad (2)$$

where  $V_2$  is the volume of the saturated sample and  $V_1$  that of the oven dried sample. ASE was determined using eqn. 3,

$$ASE (\%) = 100 (S_u - S_m) / S_u \quad (3)$$

where  $S_u$  and  $S_m$  are the swelling coefficients of unmodified and modified wood, respectively.

### Fungal Decay Test

Modified and unmodified *Hevea brasiliensis* wood samples were exposed to brown rot (*Polyporus meliae*) and white rot fungus (*Coriolus versicolor*) under the laboratory conditions. Six replicate blocks were used for each test. The samples were autoclaved and exposed to different fungi on 3% malt extract agar in culture bottles. Culture bottles were inoculated one week prior to the test. The samples were supported on glass rod to avoid contact with agar and kept at room temperature ( $25 \pm 5^\circ\text{C}$ ) and 60-70% relative humidity for 12 weeks. Samples were removed after 12 weeks, autoclaved, mycelium was removed and oven dried to constant weight. Weight loss was determined for individual sample using eqn. 4,

$$\% \text{ Weight loss} = [(W_o - W_f) / W_o] \times 100 \quad (4)$$

where  $W_o$  is oven dry weight of sample prior to exposure, and  $W_f$  is the oven dry weight of samples after exposure to fungus.

### Evaluation of photostability of Modified Wood Surfaces

The photostability of wood surfaces modified with phenylisothiocyanate was assessed using artificial accelerated weathering in a weather-o-meter (ATLAS CI 4000). The modified wood specimens measuring  $135 \times 45 \times 4$  mm (longitudinal by radial by tangential) along with unmodified control specimens were exposed continuously to a 6500 watt water cooled xenon arc lamp at operating black panel temperature of  $63^\circ\text{C}$  and relative humidity of 70%, with water spray cycle of 18 minutes rain for every 102 minutes at the rate of 0.2 L/minute. The samples were fixed to a steel holder and rotated around the fixed xenon light source. The samples were removed from weather-o-meter after exposure of 100 and 200 hours and analyzed using FTIR.

## RESULTS AND DISCUSSION

The average weight percent gain (WPG) increased with increasing reaction time. A WPG up to 16.5% was obtained after 24 hours of reaction at  $85^\circ\text{C}$ .

### Chemical Structure Changes of Wood after Modification

The FT-IR spectra illustrate the chemistry of esterification (Fig. 3). The assignments of major FTIR absorption bands of unmodified and modified wood are summarized in Table 1. The appearance of a strong absorption band at  $1375 \text{ cm}^{-1}$  in modified wood

may be due to C=S asymmetric and symmetric stretching vibrations. The increase in the intensity of band at  $1550\text{ cm}^{-1}$  is due to NH deformation. No drastic change, as is usually observed in case of other esterification reactions, was observed in the intensity of the OH stretching peak at  $3400\text{ cm}^{-1}$ . This is due to introduction of new NH stretching bonds, which absorb in the same region. However, a slight narrowing of band was observed with respect to control wood.

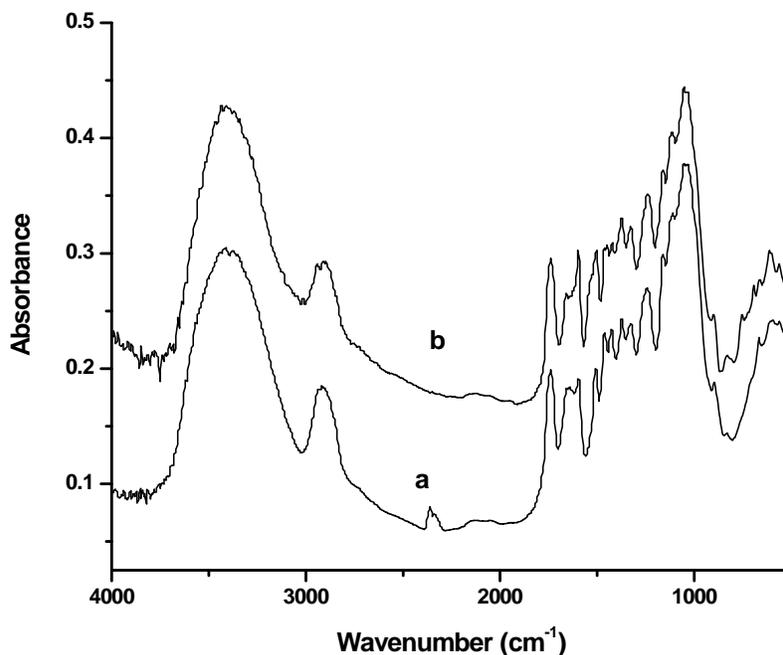


Fig. 3. FTIR spectra of (a) unmodified and (b) modified rubberwood

Table 1. Assignments of FTIR Peaks (Dyer 1991; Chen 1992)

Band position ( $\text{cm}^{-1}$ )	Assignment
1375	C=S asymmetric and symmetric stretching
1511	C=C aromatic stretching
1550	NH bending vibration
1740	C=O stretching
3400	OH and NH stretching

The NMR spectra of unmodified and modified wood are shown in Fig. 4. The appearance of signals at 62.1ppm and 84.1ppm in CP/MAS  $^{13}\text{C}$  NMR spectra of unmodified wood are due to  $\text{C}_6$  and  $\text{C}_4$  amorphous cellulose, respectively (Chang and Chang 2006). The decrease in intensity of above said signals indicates that esterification occurred at the  $\text{C}_6$  and  $\text{C}_4$  positions of amorphous cellulose. Results from  $^{13}\text{C}$  NMR and FTIR spectral analyses indicate formation of carbamate ester bond during modification.

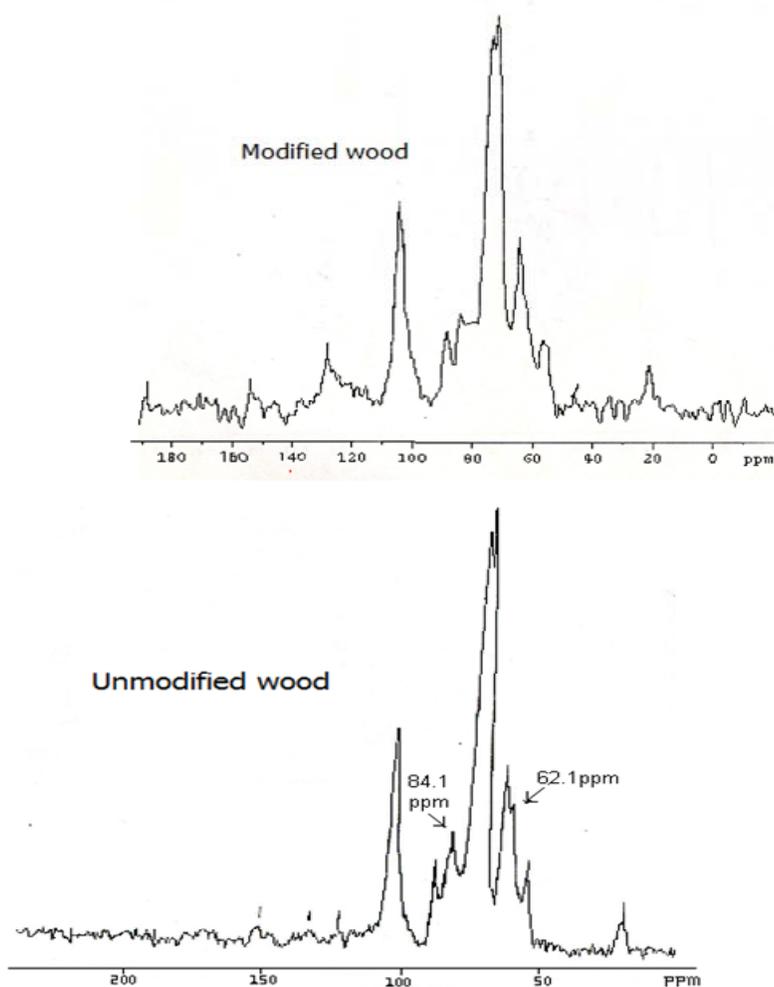


Fig. 4. NMR spectra of (a) unmodified and (b) modified rubberwood

### Dimensional Stability of Modified Wood

The values of volumetric swelling ( $S_1$  and  $S_3$ ) and shrinking coefficients ( $S_2$  and  $S_4$ ) and anti-swelling efficiency ( $ASE_1$  and  $ASE_3$ ) and anti-shrinking efficiency ( $ASE_2$  and  $ASE_4$ ) were calculated. Average values of  $S_1$ ,  $S_2$ ,  $S_3$ , and  $S_4$  along with standard deviations are shown in Table 2. The volumetric swelling coefficient of unmodified wood was  $12.6 \pm 1.11$  and  $12.0 \pm 1.23$  for the first and second soaking cycles; while the values of volumetric shrinking coefficients were  $12.33 \pm 1.18$  and  $11.90 \pm 1.22$  for first and second drying cycles, respectively. The minor decrease in value of  $S$  in subsequent cycles may be due to leaching of extractives. After modification, the volumetric swelling coefficient of esterified wood was reduced significantly. The value of  $S_1$  decreased with increase in degree of modification and attained a value of  $4.72 \pm 0.47$  for  $\sim 13.23$  WPG. This has been attributed to the bulking of cell wall and reduction in hydrophilicity of the wood after modification. This indicates that the dimensional stability of wood was greatly improved by modification with phenylisothiocyanate. ASE values based on average values of swelling coefficients are also summarized in Table 2. The anti-swelling

efficiency depends upon the extent of modification (WPG), and its value increased with increasing the WPG of modified wood. The maximum value of anti-swelling efficiency of esterified wood ( $ASE_1$ ), corresponding to  $13.23 \pm 1.01$  WPG, was 62.53%. Its value decreased to 56.41% ( $ASE_3$ ) in the second soaking cycle. This shows some leaching of phenylisothiocyanate groups during first water soaking cycle. The value of anti-shrinking efficiency was 53.85 ( $ASE_2$ ) and 56.38 ( $ASE_4$ ) during first and second drying cycles, respectively. This indicates the stable nature of bonding. These values of ASE are comparable to the values reported for acetic anhydride, methyl isocyanate, and alkylene oxides (propylene and butylene oxide) (Rowell 2005; 2006).

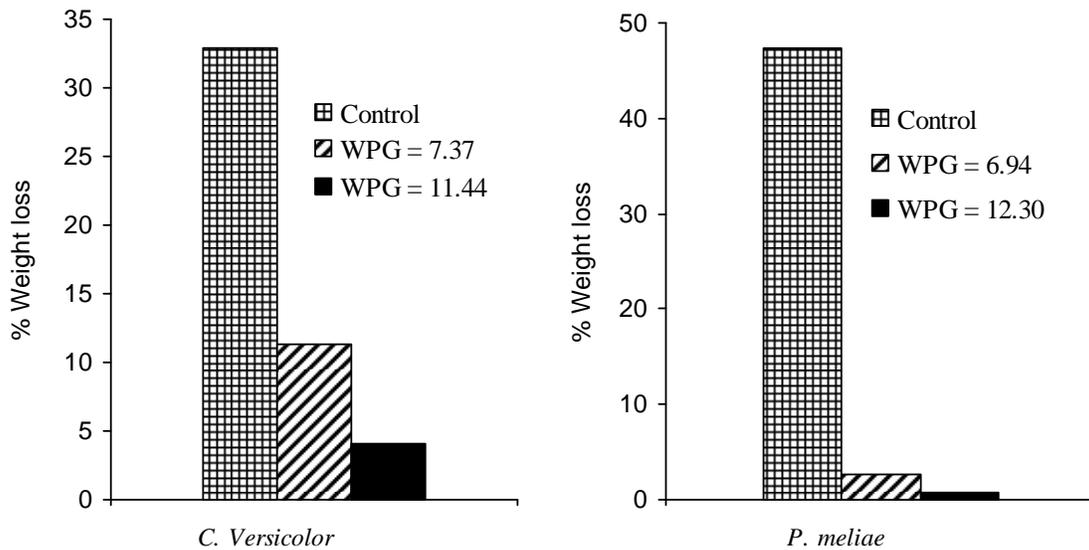
**Table 2.** Volumetric Swelling and Shrinking Coefficients (S) and Anti-Swell/ Anti-Shrink Efficiency (ASE) of Rubberwood Modified with Phenylisothiocyanate

WPG	$S_1$	$ASE_1$	$S_2$	$ASE_2$	$S_3$	$ASE_3$	$S_4$	$ASE_4$
Control	12.6 $\pm 1.11$	-	12.33 $\pm 1.18$	-	12.0 $\pm 1.23$	-	11.90 $\pm 1.22$	-
8.49 $\pm 1.24$	6.01 $\pm 0.98$	52.30	6.83 $\pm 0.81$	44.61	6.7 $\pm 0.95$	44.16	6.43 $\pm 0.77$	45.96
11.05 $\pm 0.58$	5.13 $\pm 0.43$	59.28	6.2 $\pm 0.43$	49.71	5.86 $\pm 0.50$	51.16	5.57 $\pm 0.56$	53.19
13.23 $\pm 1.01$	4.72 $\pm 0.47$	62.53	5.69 $\pm 0.39$	53.85	5.23 $\pm 0.56$	56.41	5.19 $\pm 0.43$	56.38

WPG - Weight percent gain,  $S_1$  - Volumetric swelling coefficient determined from initial OD volume and first water swollen volume,  $ASE_1$  - Anti-swelling efficiency based on  $S_1$ ,  $S_2$  - determined from first water swollen volume and re-oven dry volume,  $ASE_2$  - Anti-shrink efficiency based on  $S_2$ ,  $S_3$  - Determined from re-ovendry volume and second water-swollen volume,  $ASE_3$  - Anti swell efficiency based on  $S_3$ ,  $S_4$  - Determined from second water swollen volume and second re-ovendry volume,  $ASE_4$  - Anti-shrink efficiency based on  $S_4$ .

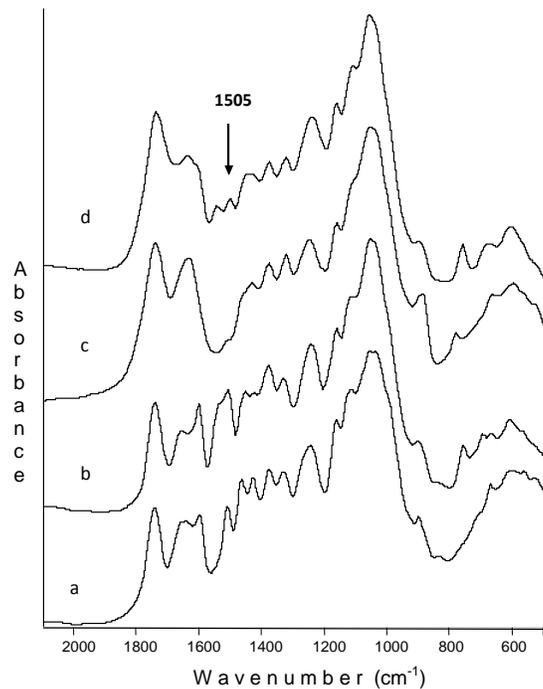
### Decay Resistance of Modified Wood

Figure 5 (a, b) shows fungal decay resistance for 12 weeks of exposure to a brown rot (*P. meliae*) and a white rot (*C. versicolor*) fungus. 12 weeks exposure of unmodified wood to *C. versicolor* resulted in a weight loss of 32.9%, while modified samples suffered weight loss of only 11.34% (corresponding to 7.37% WPG specimen) and 4.02% (corresponding to 11.44% WPG specimen). Similarly, weight loss in samples exposed to *P. meliae* was reduced from 47.42% in unmodified samples to 2.7% (for 6.9% WPG) and 0.68% (for 12.3% WPG) in modified samples. This shows drastic reduction in weight loss due to fungal decay of esterified wood. These observations are in accordance with the findings of Chen (1992) on phenylisothiocyanate modified wood against brown rot fungus. The biological resistance of modified wood is increased because of increased hydrophobicity induced by replacement of accessible hydroxyl groups in wood polymers. The chemical modification results in reduction in moisture content as compared to unmodified wood. The fungicidal thiocarbonyl groups from the modified wood may also contribute to the decay resistance (Chen 1992). Several chemicals have been shown to exhibit excellent resistance against brown and white rot fungi (Rebecca and Rowell 2000; Chang and Chang 2005; Rowell 2005; 2006).



**Fig. 5.** Weight loss of unmodified (control) and modified rubberwood blocks due to exposure to *C. versicolor* and *P. meliae*.

### Photo Stability of Modified Wood



**Fig. 6.** FTIR spectra of: (a) unmodified wood, (b) modified wood, (c) unmodified wood irradiated for 100h, and (d) modified wood irradiated for 100h

The effect of irradiation on the FT-IR spectra of the unmodified and modified wood is shown in Fig. 6. Analysis of FT-IR spectra of unmodified wood showed a

significant reduction in the intensity of the aromatic C=C band in lignin at 1506  $\text{cm}^{-1}$ , which disappeared almost completely after 100 hours exposure. The reduction in the intensity of the C=C band indicated photodegradation of the lignin structure. This was accompanied by an increase in the intensity of the bands at 1740  $\text{cm}^{-1}$  and around 1650  $\text{cm}^{-1}$ . The bands at 1740 and 1650  $\text{cm}^{-1}$  are characteristic absorption of non-conjugated and conjugated carbonyl stretching vibrations. This has been attributed to the formation of quinone type structures during irradiation (Hon 2001; Williams 2005; Pandey 2005; Pandey and Vuorinen 2008). A similar effect of irradiation was also observed in modified wood (curve d in Fig. 6). After 100 hours of exposure, there was a significant decrease in lignin C=C band at 1506  $\text{cm}^{-1}$  and increase in intensity of band at 1740  $\text{cm}^{-1}$  and 1650  $\text{cm}^{-1}$ . The intensity of C=S asymmetric stretching vibration band at 1375  $\text{cm}^{-1}$  also decreased upon irradiation. This indicates that modification of wood with phenyliso-thiocyanate is not effective in reducing photodegradation of wood surfaces.

## CONCLUSIONS

FTIR and  $^{13}\text{C}$  NMR spectral analysis of wood modified with phenylisothiocyanate indicated formation of an ester. Esterified wood exhibited very high resistance to brown- and white-rot fungi and also improved its dimensional stability. However, it was not effective in reducing photodegradation of wood surfaces.

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