ROLE OF PRETREATMENTS IN THE PROTECTION OF WOOD SURFACE AND FINISHES IN THE WEATHERING OF *PTEROCARPUS MARSUPIUM* WOOD

N. K. Upreti* & K. K. Pandey

Institute of Wood Science and Technology, 18th Cross Malleswaram, Bangalore – 560 003, India

Received December 2002

UPRETI, N. K. & PANDEY, K. K. 2005. Role of pretreatments in the protection of wood surface and finishes in the weathering of *Pterocarpus marsupium* wood. Wood samples of *Pterocarpus marsupium* (bijasal) treated with aqueous solutions of ammoniacal copper ferricyanide, acid copper chromate, acid copper chrome arsenate, ammoniacal copper chromate and ammoniacal copper chrome ferricyanide were studied for outdoor weathering. The treated and untreated wood samples were coated with three type of commercially available wood finishes, viz. polyurethane finish, synthetic enamel and acrylic emulsion paints, before exposing to the outdoor. Paint performance was studied for different periods of outdoor exposure. Results indicated that pretreatment of wood surface with the inorganic salt solutions helps in imparting water repellency and dimensional stabilization to the wood, resulting in increased service life of opaque and transparent coating over it. These treatments also slow down the photo-degradation of wood surface and inhibit fungal growth over it. Out of the five treatments used, the acid copper chromate provided the best protection to outdoor exposed wood surface.

Key words: *Pterocarpus marsupium* – weathering – FTIR – photodegradation – polyurethane finish – pretreatment – delignification

UPRETI, N. K. & PANDEY, K. K. 2005. Peranan prarawatan dalam perlindungan permukaan kayu dan kemas siapan dalam luluhawa kayu *Pterocarpus marsupium*. Sampel kayu *Pterocarpus marsupium* yang dirawat dengan larutan akuas kuprum ferisianida berammonia, asid kuprum kromat, asid kuprum kromat arsenat, kuprum kromat berammonia, kuprum krom ferisianida berammonia dikaji untuk lulu hawa luar. Sampel kayu yang dirawat dan yang tak dirawat disalut dengan tiga jenis kemas siapan kayu komersial iaitu polyuretana, enamel tiruan dan cat emulsi akrilik sebelum didedahkan di luar. Prestasi cat dikaji untuk masa dedahan yang berlainan. Keputusan menunjukkan bahawa prarawatan permukaan kayu dengan larutan garam tak organik menyebabkan kayu tak telap air dan menjadikan kayu lebih stabil dari segi dimensi. Ini dapat meningkatkan khidmat hayat lapisan legap dan lutsinar pada kayu. Rawatan ini juga melambatkan degradasi foto pada permukaan kayu dan menghalang pertumbuhan kulat. Antara kelima-lima rawatan, asid kuprum kromat menawarkan perlindungan terbaik kepada kayu yang didedah di luar.

Introduction

Outdoor wood undergoes photodegradation due to sunrays (Fiest & Hon 1984). As a result of photodegradation the wood surface becomes uneven and consequently unbalanced forces are developed on the paint coating over it, which starts cracking. With time, due to exposure to rain water and air, the cracked coating flakes out. If

*E-mail: nkupreti@hotmail.com

the wood surface beneath a paint coating is treated to protect it from photodegradation the durability of paint coating improves. Similarly, clear coatings on exposed wood have very short life (Chang *et al.* 2002). The primary cause of the failure is photodegradation of wood surface to which the coating loses its adhesion. Treatment of wood surface by inorganic salt solutions plays an important role on imparting some useful properties to wood surface (Williams & Fiest 1985). The properties like water repellency and dimensional stability can be infused in wood by surface treatments using inorganic salt solutions.

The service life of paint coatings over outdoor exposed wood can also be enhanced by pretreating the wood with certain water repellent formulations. Fiest (1987) has reported the benefits of such pretreatments on outdoor weathering of yellow poplar. Water-repellent pretreatment was shown to be very beneficial in improving the performance of painted yellow poplar siding, especially in decay control.

The emphasis in this work is on performance of various inorganic pretreatments for *Pterocarpus marsupium* wood surface that may inhibit fungal growth on it, protect it against photodegradation and improve its dimensional stability.

Materials and methods

Wood specimens of *P. marsupium* of size 6 x 4 x 1 inch and moisture content less than 14 % were brush coated with five different inorganic salt solutions by keeping them in horizontal position. The following chemical compositions of the treating solutions are in accordance with the formulations of Black and Marz (1974):

Treatment 1.	Ammoniacal copper ferricyanide	Percentage by weight
	$CuSO_4.5H_2O$	7.9
	$K_3Fe(CN)_6$	6.0
	NH_4OH	14.0
	Water	72.1
Treatment 2.	Acid copper chromate	
	$CuSO_4.5H_2O$	10.82
	$Na_2Cr_2O_7.2H_2O$	6.51
	Water	82.67
Treatment 3.	Acid copper chrome arsenate	
	$CuSO_4.5H_2O$	3.0
	$Na_2Cr_2O_7.2H_2O$	4.0
	As_2O_5	1.0
	Water	92.0

Treatment 4. Ammoniacal copper chromate

$CuSO_4.5H_2O$	10.81
$Na_2CrO_4.4H_2O$	10.13
NH_4OH	21.62
Water	57.44

Treatment 5. Ammoniacal copper chrome ferricyanide

$CuSO_4.5H_2O$	1.85
$Na_2CrO_4.4H_2O$	4.73
$K_3Fe(CN)_6$	1.28
NH_4OH	3.70
Water	88.44

Seven replicates were taken for each treatment solution. The cross-sectional ends of the specimens were coated with bituminous paint to slow down the moisture movement through the ends. The treated specimens were air dried for ten days in open racks in the laboratory. These specimens were brush coated with three different types of commercially available wood finishes, viz. synthetic enamel paint, acrylic emulsion paint and polyurethane finish. The first two paints are opaque and white in colour, the third one is transparent. All the paints were applied following the recommendations provided by the manufacturers.

All the treated and painted specimens along with the controls were screwed from the back side on marine grade plywood sheets of 12 mm thickness and were exposed to the outdoor on a rooftop without any shadow falling over the panels. The samples were kept at 15° inclination from the horizontal, facing the southeast direction to get maximum solar radiation.

Pretreatment and paint performance ratings

The evaluation methods used in this study were based on the American Society for Testing and Materials (ASTM) Standards (Table 1). These standards use pictorial standards to evaluate the coating defects.

Determination of overall performance of pretreatment was based on the average of the pretreatment performance, fungal attack, erosion of wood surface and its general appearance. A 10 to 1 rating was used for its evaluation. A 10 value represented the original condition of the pretreatment; a 5 represented that reapplication of the pretreatment was required without much surface preparation and a 1 represented total failure, i.e. the finish was completely eroded and the surface was covered with fungus.

Similarly for paint coatings the evaluation was based on the performance of paint coatings and their general appearance. The performance of paint was evaluated for checking, flaking, cracking and discoloration. General appearance was based on surface disfigurement of paint films/substrate by microbial growth.

Table 1 Inspection criteria and evaluation methods

Inspection criterion	Evaluation method
Paint checking	ASTM D 660-87
Paint cracking	ASTM D 661-86
Paint flaking	ASTM D 772-86
Surface disfigurement	ASTM D 3274-88
General appearance	Subjective visual assessment
Wood surface photodegradation	FTIR study

For convenience of presentation, the various performance criteria were combined into a single value called the overall performance rating. The overall performance of paint coatings was based on average of its evaluation for checking, flaking, cracking and general appearance rating. Each inspection criterion was evaluated on a scale of 10 to 1. The overall rating of 10 indicated no change in the original unweathered condition, 5 indicated the condition in which repainting would be required, and 1 indicated total failure of the paint coating. The duration of outdoor exposure until checking or cracking of paint coating starts appearing is a convenient measure of paint durability and the effectiveness of the surface treatments.

FTIR spectra of wood surfaces of weathered and unweathered controls were measured by direct transmittance using the KBr pellet technique. Spectra were recorded using a Nicolet Impact 400 FTIR spectrometer equipped with a DTGS detector. The top of the weathered surfaces (thickness $< 50\ \mu m$) was removed using a razor blade and mixed with potassium bromide (for IR spectroscopy, MERCK) to about 0.5–1 % concentration. All the spectra were measured at a spectral resolution of 4 cm^{-1} and 32 scans were taken per sample. The assignment of different bands is described elsewhere (Pandey & Theagarajan 1997).

Results and discussion

Weathering of treated wood

The overall responses to the pretreatments of wood surfaces exposed to the outdoor are summarized in Table 2. Among the pretreatments treatment No. 2 provided the best protection, up to 60 months followed by treatment No. 5 and then treatments No. 1 and 4. Treatment No. 3 was found the least effective at the end of 60 months of outdoor exposure in protecting the wood surface and performed well only up to 15–28 months of exposure. Treatment No. 2 was found fairly effective even after 48 months of exposure. In the controls many surface cracks developed compared with treated samples which may be due to frequent wetting and drying of wood surface. The treatment chemicals worked as water repellents, hence surface cracks were found negligible up to 54 months of outdoor exposure in the pretreated wood. At 60 months of outdoor exposure, the wood surfaces were found eroded in

Table 2 Overall performance ratings of pretreatments on *Pterocarpus marsupium*

	04 M	08 M	15 M	28 M	48 M	60 M
Control (no treatment)	09	08	07	05	04	03
Treatment 1	10	10	10	08	07	06
Treatment 2	10	10	10	10	09	08
Treatment 3	10	10	09	08	06	03
Treatment 4	10	10	09	08	07	06
Treatment 5	10	10	10	09	08	07

M = months

all the pretreated samples showing the wearing off of the protection afforded by the pretreatments. This might have been due to of leaching of chemicals.

FTIR studies

The FTIR spectra of the control and untreated *P. marsupium* wood surfaces weathered for 15 and 48 months are shown in Figure.1. Severe delignification and hemicellulose breakdown in the weathered wood are evident from the significant reduction in the intensity of bands at 1505, 1600, 1463, 1268, 1240 and 1737 cm^{-1} after 15 months. Longer exposure showed depolymerization of cellulose which is indicated by the reduction at 898 and 1160 cm^{-1} bands after 48 months.

Figure 2 shows the FTIR spectra of wood treated with ammoniacal copper ferricyanide (treatment No. 1) and weathered for 15 and 48 months, and of the treated controls. It can be seen that this treatment checks delignification very effectively up to 15 months, which is indicated by only slight reduction in 1509 cm^{-1} bands after exposure. Treatment with solution No. 4 results in reduction at 1737, 1509, 1240 cm^{-1} and slight increase at 1120 cm^{-1}. This treatment checks delignification up to 15 months and is effective up to 48 months to some extent. Similarly, treatment No. 5 results in considerable reduction of bands at 1737 cm^{-1} and a slight reduction at 1509, 1462 and 1240 cm^{-1}. The FTIR spectra indicate that treatment No. 5 is very effective in reducing and checking delignification up to 15 months of outdoor exposure and is also effective up to 48 months to a considerable extent as compared with the untreated wood (Figure 3). This treatment performed best among all the five in reducing delignification.

FTIR studies revealed that treatment No. 2 is ineffective in reducing delignification and hemicellulose degradation whereas treatment No. 3 provides only a small protection from photodegradation of these polymeric constituents of wood. This is in contradiction with the overall performance of treatment No. 2 as discussed earlier (refer Table 2). Though this solution is ineffective in restricting delignification of wood, the overall appearance of the treated samples was best even after 48 months of exposure. The overall performance of the treatment under outdoor exposure is a combination of its property of water repellency, dimensional stabilization, effectiveness against photodegradation of wood constituents (mainly lignin) and against microbial attack.

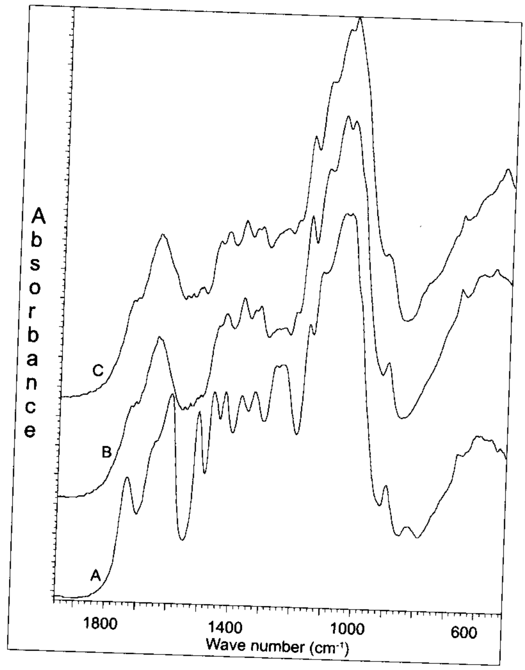

Figure 1 FTIR spectra of untreated wood weathered for (A) 0 days, (B) 15 months and (C) 48 months

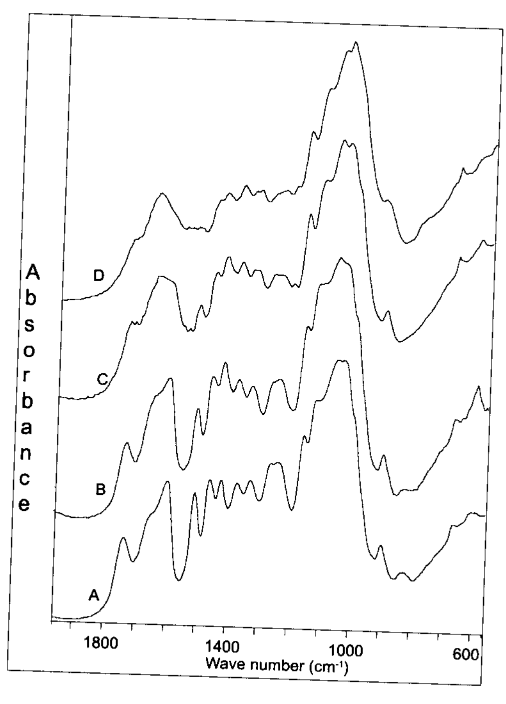

Figure 2 FTIR spectra of wood treated with ammoniacal copper ferricyanide and weathered for (B) 0 days, (C) 15 months and (D) 48 months. Curve (A) corresponds to untreated and unweathered wood.

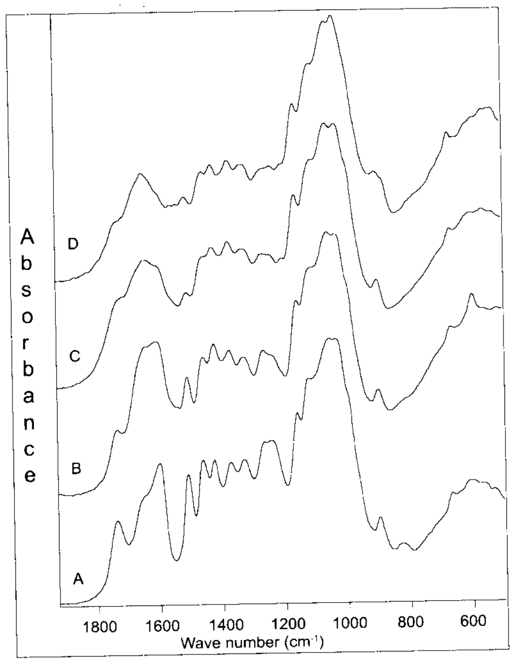

Figure 3 FTIR spectra of wood treated with ammoniacal copper chrome ferricyanide and weathered for (B) 0 days, (C) 15 months and (D) 48 months. Curve (A) corresponds to untreated and unweathered wood.

Weathering of painted wood pretreated with inorganic salt solutions

The overall performance of the three paint systems is shown in Table 3. The polyurethane paint is transparent and the two other paints used in the study are opaque and white in colour.

Polyurethane finish

Table 3 shows that up to 8 months of outdoor exposure the transparent polyurethane coating performed well in all the samples with and without treatments. It failed completely after 15 months of outdoor exposure in the untreated controls. Overall , the transparent coating performed well up to 15 months of exposure, with treatment No. 4 being the best. The coating started losing its gloss at longer exposure and required refinishing after 21 months of exposure and failed completely after 28 months of exposure. These results are in accordance with the FTIR study of weathered pretreated wood, i.e. reduction in photodegradation of lignin and hemicellulose reduces cracking and flaking out of transparent coating as compared with the coatings without pretreatment. These results clearly show the beneficial effects of surface pretreatment on the outdoor performance of transparent coatings provided the coating does not photodegrade itself before the wood surface beneath it.

Table 3 Overall performance ratings of paint coatings on *P. marsupium*

	04 M	08 M	15 M	21 M	28 M	48 M	60 M
Polyurethane finish							
Control (no treatment)	10	10	03	01	01		
With treatment 1	10	10	06	05	01		
With treatment 2	10	10	06	05	01		
With treatment 3	10	10	06	04	01		
With treatment 4	10	10	07	05	01		
With treatment 5	10	10	06	05	01		
Synthetic enamel paint							
Control (no treatment)	10	10	10		08	07	05
With treatment 1	10	10	10		09	09	08
With treatment 2	10	10	10		09	09	08
With treatment 3	10	10	10		10	09	08
With treatment 4	10	10	10		09	09	08
With treatment 5	10	10	10		09	09	08
Acrylic emulsion paint							
Control (no treatment)	10	10	09		05	01	00
With treatment 1	10	18	04		03	02	01
With treatment 2	10	10	10		09	08	07
With treatment 3	10	10	10		09	08	06
With treatment 4	10	10	10		09	08	07
With treatment 5	10	10	10		08	05	04

M = months

Synthetic enamel paint

All the pretreated as well as untreated samples coated with white synthetic enamel paint performed well up to 15 months of outdoor exposure. Slight cracking of paint coating was observed in the untreated controls after 28 months of exposure. Until 60 months of exposure all the coated samples with pretreatment were very effective whereas the paint coating cracked and flaked out in the untreated controls after the same exposure. The results show the usefulness of pretreatments in prolonging the paint life over wood surfaces. As the paint coating is opaque here, it seems the dimensional stability imparted by these pretreatment plays an important role. The performance of the paint coating is equally good for all treatments even after outdoor exposure of 60 months. Longer exposure times are necessary to determine the superiority of a treatment.

Acrylic emulsion paint

The results of white acrylic emulsion paint coating are interesting. The paint coating over treatment No. 1 failed badly within 12 months of exposure. The coating condition over the controls was good up to 15 months of exposure and required refinishing after 28 months of exposure. The coating required refinishing after 48 months of exposure in samples treated with solution No. 5. The coating condition was maintained very well after 28 months of exposure and showed very little deterioration even after 48 months of exposure in samples treated with treatments No. 2, 3 and 4. At 60 months of exposure treatments No. 2 and 4 showed superiority over treatment No. 3. Here also longer exposure times are necessary to determine the superiority of a treatment.

Pterocarpus marsupium is a wood species described to have natural durability of more than 120 months (Anonymous 1982). Other than the usual surface staining fungi on the controls, there was no sign of decay fungi on any untreated/treated test specimens. This wood leaches dark coloured extractives when it comes in contact with water which can be controlled by surface treatments (Upreti *et al.*1999).

Conclusions

Pretreatments of *P. marsupium* wood surface with inorganic salt solutions help in imparting photoprotection, water repellency and dimensional stabilization to the wood. These pretreatments also inhibit fungal growth over the wood surface. Out of the five pretreatments used, acid copper chromate provided the best overall protection to the outdoor exposed wood surface. The FTIR study showed that ammoniacal copper chrome ferricyanide provided the best protection against photodegradation of lignin in the wood surface. The life of transparent polyurethane paint also increased in the pretreated wood. Ammoniacal copper chromate seems to be the most effective in prolonging the life of polyurethane coating. In the case of opaque synthetic enamel paint, the pretreatments imparted water repellency and dimensional stabilization to the wood ultimately increasing the life of paint coating over it. The ammoniacal copper ferricyanide and ammoniacal copper chrome ferricyanide pretreatments failed to give any promising results whereas the other three pretreatments, viz. acid copper chromate, acid copper chrome arsenate and ammoniacal copper chromate, were found effective in wood coated with opaque acrylic emulsion paint.

Acknowledgements

The authors express their thanks to the Director of the Institute of Wood Science and Technology, Group Co-ordinator (Research) and Head, Wood Seasoning & Preservation Division, for their support and encouragement.

References

ANONYMOUS. 1982. *IS 401. Code of Practice for Preservation of Timber.* Indian Standards Institution, New Delhi.

BLACK, M. J & MARZ, E. A. 1974. *Inorganic Surface Treatments for Weather-resistant Natural Finishes.* USDA Forest Service Research Paper, FPL 232.

CHANG, H. T., YEH, T. F. & CHANG, S. T. 2002. Comparisons of chemical characteristic variations for photodegraded softwood and hardwood with/without polyurethane clear coatings. *Polymer Degradation and Stability* No. 77: 129–135.

FIEST, W. C. 1987. Weathering performance of finished yellow-poplar siding. *Forest Products Journal* 37 (3): 15–22.

FIEST, W. C & HON, D. N. S. 1984. Chemistry of weathering and protection. Pp. 401–451 in Rowell, R.M. (Ed.) *Chemistry of Solid Wood.* Advances in Chemistry Series No. 207. American Chemical Society.

PANDEY, K. K. & THEAGARAJAN, K. S. 1997. Analysis of wood surfaces and ground wood by diffuse reflectance (DRIFT) and photoacoustic (PAS) Fourier transform infrared spectroscopic techniques. *Holz als Roh- und Werkstoff* 55(6): 383–390.

UPRETI, N. K. , PANDEY, K. K. & ANANTHANARAYAN, A. K. 1999. Prevention of extractive leaching by chemical treatments of wood surface. *Holzforschung* 53(6): 675–676.

WILLIAMS, R. S. & FIEST, W. C. 1985. Wood modified by inorganic salts: mechanism and properties. I. Weathering rate, water repellency, and dimensional stability of wood modified with chromium (III) nitrate versus chromic acid. *Wood and Fiber Science* 17: 184–198.