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Effects of Extraction on Wettability and Gluability of Apitong (*Dipterocarpus Grandiflorus Blanco*)

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An investigation was made of the effects of extraction and various chemicals applied on veneer surface on the wettability and gluing properties of apitong, *Dipterocarpus grandiflorus* Blanco, using urea formaldehyde resin. Wettability was determined by measuring contact angles with distilled water.

It was found that extraction with methanol-benzene greatly improved the wettability and gluability of apitong veneer. Likewise, surface treatment with methanol-benzene significantly increased the wettability of the veneer as well as the dry and wet shear strengths of the resulting bond. Treatment with sodium hydroxide increased both wettability and dry shear, but decreased the wet shear strength of the bond. Acetone did not have a significant effect on both wettability and dry shear, but decreased wet shear strength. On the other hand, ether had adverse effects on the wettability and gluability of apitong veneer.

A positive linear correlation was found between wettability and gluability of apitong veneer.

INTRODUCTION

The measurement of contact angle of a liquid on a solid surface is becoming an important tool in the study of adhesion. Contact angle has been used as a measure of wettability and gives an indication of the attraction between the molecules of the liquid and the solid surface. Wettability is defined as

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the cosine of the contact angle of a liquid on a solid surface, or the ratio of the difference between the surface tension of the solid and the solid-liquid interfacial tension to the surface tension of the liquid.¹

The wetting properties of plastics, glass, and metals have been extensively studied. However, little work has been conducted on wood along this line. Collett² made an excellent review of this subject.

Presumably, the first attempt to relate the wettability of wood to bond quality was made by Freeman.³ His results and those of Bodig⁴ and Chen⁵ showed a high correlation between wettability and glue bond quality. Gray⁶ and Herczeg⁷ demonstrated the validity of Zisman's relationship for critical surface tension in their studies on the wettability of wood using a series of liquids of varying surface tensions. They pointed out the probability that bond quality is dependent upon wetting, spreading, and surface tension of the adhesive. Bryant⁸ showed that bond quality is influenced by the wettability of the wood and the interactions between the adhesive and the wood surface.

One of the factors which appears to affect wettability and adhesion is the amount and type of extraneous components present in the wood. Chen⁵ reported that extractive removal treatment improves the wettability and increases the pH of some tropical woods. In many species a difference in gluing properties has been observed between sapwood and heartwood, and this has been attributed to the nature and amount of extractives found in the latter.⁹ Extractives may interfere with the spread and penetration of adhesives and hence with the formation of bonds. Dadswell and Millis¹⁰ noted that extractives affect the curing of adhesives in teak. Truax¹¹ reported that treatment with a 10% solution of caustic soda before gluing improves the joint strength in certain species.

Apitong is one of the commercial timber species in the Philippines. Its utilization for plywood manufacture and related products has been limited by the difficulty in obtaining a good adhesive bond.¹² This may be attributed to the fact that said species contains a high percentage of extractives which interferes with gluing.

The present investigation reports on the effects of extraction and surface chemical treatments on the wettability and gluability of apitong veneer. Gluability was evaluated by dry and wet shear tests, while wettability was determined by the cosine of the contact angle.

EXPERIMENTAL

Apparatus

The veneer dryer used was a COE roller-type mechanical dryer. It is heated with steam supplied from a boiler at a pressure of 140 psi. It is operated at

an air velocity of 1,200 ft/min. The soxhlet extractors used had an inside diameter of 50 mm; the capacity to the top of the siphon was approximately 100 ml while the siphon tube was approximately 55 mm in height.

The oven used was a laboratory model with forced air circulation operated at 300 ft/min. It was provided with thermostatically controlled heaters.

The pressing machine used was a William hydraulic press with only one daylight opening. Its platens measured 12 × 12 inches.

The set-up for the wettability measurements consisted of a microscope equipped with an improvised goniometer eyepiece made of transparent plastic and an improvised stage for specimen mounting. The goniometer was graduated from angles 0° to 270°, with divisions of 2.5°.

The joint testing device used was a Riehle standard plywood shear machine (automatic motor driven) equipped with jaws designed to grip the shear specimen tightly so that no slippage occurs during testing.

Materials

The veneer specimens used were cut from two bolts, 21 inches in diameter and 54 inches long, taken from the middle portion of an apitong tree trunk at the University of the Philippines Land Grant, Quezon province. Rotary cutting was done at the Forest Products Research and Industries Development Commission (FORPRIDECOM), College, Laguna. The veneer specimens were $\frac{1}{8} \times 12 \times 12$ inches taken from the heartwood portions of the bolts with a green specific gravity of 0.64.

The resin used was liquid urea formaldehyde (CR-5H). It was provided by Borden Chemical Company.

Methods

Eighteen sheets were used in the experiments involving the effects of extraction. Nine sheets were used for each treatment, i.e., unextracted (control) and extracted. Veneer extraction was carried out in soxhlet extractors. It consisted of 16 hours immersion in 150 ml of methanol-benzene solution (50:50 parts by volume). After 8 hours of extraction, the veneers were inverted since they were not completely immersed in the solvent. Two veneer sheets were rolled and extracted together at a time in the same soxhlet apparatus. The extraction was followed by three days of soaking in distilled water. The veneer sheets were dried in an oven at 340°F to a moisture content of 6%. Drying times were 7.3 and 2.0 minutes for the unextracted and extracted veneers, respectively. They were stored in a plastic bag for a day and then conditioned for a week in an air-conditioned room at 62°F and 64% relative humidity.

The veneers for surface chemical treatment were initially dried in a veneer dryer to an average moisture content of 6% after which they were piled on stickers for one week in an air-conditioned room maintained at a temperature of 62°F and 64% relative humidity. Nine sheets each were wiped for 1 minute with cloth saturated with the following solutions: 10% sodium hydroxide, methanol-benzene (50:50 parts by volume), acetone, and ether. The sheets treated with sodium hydroxide were wiped with a clean dry cloth to remove excess solution and then neutralized with a solution of 10% acetic acid. After each treatment, the veneer surfaces were immediately wiped dry with a clean white cloth. Nine untreated sheets were also set aside to serve as control. All the veneer sheets were again conditioned in the same air-conditioned room for a week to an average moisture content of 10%. They were then cut to dimensions of 10 × 10 inches prior to gluing.

Glue mixing was done with an electrically-operated glue mixer. A fresh mix of glue was uniformly double-spread on the veneers at 50 lb per MSGL by a rubber roller spreader. A closed assembly time of 20 minutes was used. Each assembly (three-ply) was then cold pressed at room temperature (82°F) and specific pressure of 150 psi for 24 hours. The pressed panels were stacked in an air-conditioned room at 62°F and 64% relative humidity for a week before preparing specimens from them.

Three panels were made for each treatment from which plywood shear specimens were prepared. Half of the specimens were used for dry shear and the other half for wet shear (hot and cold soak) tests. The specimens were stored at room temperature for three days prior to testing. The dry shear test was conducted in accordance with the American Society for Testing Materials,¹³ and the wet shear test with the Japanese standard for Type II plywood.¹⁴ Testing was carried out at room temperature (82°F) and 85% relative humidity. The load at failure and the percentage of wood failure were recorded for each specimen.

Wettability measurements were made on the trimmings ($\frac{1}{2} \times 2$ inches cut along the grain) of the core veneers used for gluing. They were carried out in an air-conditioned room maintained at $62^\circ \pm 2^\circ\text{F}$ just before gluing. The specimen was mounted on an improvised stage which moved the former up and down. A drop of distilled water was placed on the tight side of the specimen by means of a syringe. The contact angle was measured by rotating the goniometer so that the hairline passed through the point of contact between the drop and veneer surface and tangent to the drop at that point. Measurements were made 5 seconds after the distilled water had been dropped to allow the drop to attain equilibrium on the surface of the specimen. There were three specimens for each treatment and five measurements for each specimen.

An additional experiment was carried out to determine the amount of

extractives soluble in alcohol-benzene, acetone, ether, and sodium hydroxide. Eight veneer sheets were ground into wood flour with a Wiley mill. The flour that passed through a 40-mesh but retained on a 60-mesh sieve was used for extractive determination using the ASTM standards.¹³ Three determinations were made for each chemical.

RESULTS AND DISCUSSION

Effect of extraction

Results of the experiments on the effects of extraction on wettability and bond quality are presented in Table I. It is evident that extraction with methanol-benzene showed great improvements in the wettability and gluability of apitong veneer. Analyses of variance indicated that the wettability and breaking loads, determined by the dry and wet shear tests, differed significantly (at 0.01 probability level) between the unextracted and extracted veneer.

TABLE I

Wettability and gluability of extracted and unextracted apitong veneer

Treatment	Wettability ^a (cosine of contact angle)	Dry shear test ^b		Wet shear test ^b	
		Breaking load (psi)	Wood failure (%)	Breaking load (psi)	Wood failure (%)
Extracted	0.4683	523	100	445	94
Unextracted	0.0260	377	60	297	46

^a Each value is the average of 15 observations.

^b Each value is the average of 24 and 9 observations for the unextracted and extracted treatments, respectively.

The results suggest that the presence of methanol-benzene soluble extractives affects the wettability and gluability of apitong veneer. This was probably due to the low polar characteristics of the methanol-benzene soluble extractives which have the effect of lowering the surface free energy of the wood. It was observed that the extracted veneers wetted more easily than the unextracted ones. The lower bond strength observed with the unextracted veneer was probably due to a thin surface layer of waxy constituents of the wood. This if of low strength, and hence acts as a "weak boundary layer". Subsequent extraction removes more or less this boundary layer resulting in

alteration of the constitution and properties of the surface layer of the wood. Extraction gave increases of about 39 and 50% for dry and wet shear strengths, respectively.

The results obtained in this study agree with those of other workers.^{6, 15} Gray⁶ attributed the poorly wettable surfaces of European oak, Parana pine and beech to low molecular weight fatty acids, and the poor wettability of even sanded greenheart, ekki and afrormosia to the high extractive contents of the wood species. Likewise, the poor bond quality of determa glued with phenolic resin was attributed to the wax substances present in the wood.¹⁵ Marian and Wissing¹⁶ also showed that the free energy of wood, as found by wetting angle measurements, was reduced by silane treatment.

Surface chemical treatment

Results of the experiments on the effects of veneer surface chemical treatments on wettability and bond quality are presented in Table II. Wettability, breaking load, and wood failure in the dry shear test were highest for veneer treated with sodium hydroxide and lowest with ether. In the wet shear test,

TABLE II

Wettability and gluability of apitong veneer surface treated with various chemicals

Treatment	Wettability ^a (cosine of contact angle)	Extractives ^b (%)	Dry shear test ^c		Wet shear test ^c	
			Breaking load (psi)	Wood failure (%)	Breaking load (psi)	Wood failure (%)
Control	0.0377		390	80	267	64
Methanol- benzene	0.2016	8.74	415	86	319	64
Acetone	0.0429	5.24	389	82	242	33
Ether	-0.3471	2.12	341	62	152	1
Sodium hydroxide	0.6850	27.39	489	93	200	4

^a Each value is the average of 15 observations.

^b Each value is the average of 3 observations based on the oven-dry weight of the wood flour.

^c Each value is the average of 24 observations.

breaking load and wood failure were highest for methanol-benzene and again lowest for ether. Statistical analyses made on the wettability and shear strength values for both the dry and wet shear tests showed that there were significant differences among the treatments. Comparison of means by

Duncan's multiple range test showed that the application of sodium hydroxide and methanol-benzene gave significant improvements in wettability and dry shear strength of the resulting bond. On the other hand, ether decreased both wettability and dry shear strength, while acetone did not have any significant effect on either parameter. Wet shear strength also increased by treatment with methanol-benzene, and decreased by treatments with acetone, sodium hydroxide, and ether.

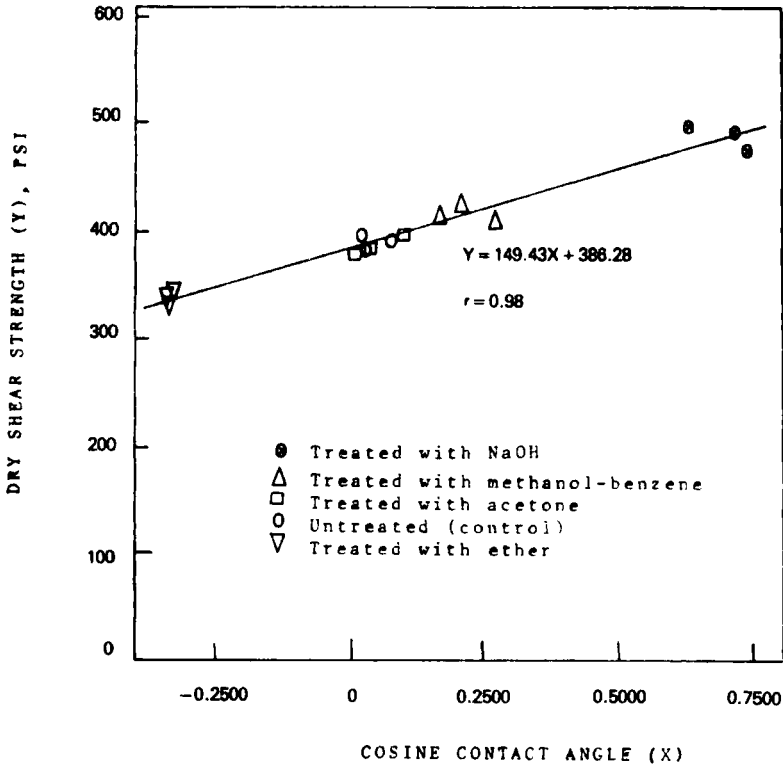


FIGURE 1 Relationship between wettability and gluability of apitong veneer surface-treated with chemicals.

The wettability of untreated apitong veneer with distilled water was low. This was probably due to the presence of extractives on the veneer, particularly those soluble in alcohol-benzene and sodium hydroxide. Veneers treated with these solvents (alcohol-benzene and sodium hydroxide) showed significant improvements in wettability due probably to the large amounts of extractives soluble in them which were 8.7 and 27.4%, respectively. These substances are probably the fatty acids in the wood. Apitong contains about 0.62% fatty acids based on the oven-dry weight of the wood.¹⁷

Ether and acetone were not effective in improving the wettability and gluability of apitong veneer. In fact, ether gave significant reductions in both these properties. This may be due to the method of applying the chemicals on the surface of the veneer. Wiping the surface with cloth might have damaged the wood cells and moved particles on the surface layer closing the cavities or openings resulting in the decrease in wettability and gluability of the veneer. Furthermore, only a small amount of extractives in the wood is soluble in ether, about 2.1%. This wood species has been found to contain about 1.32% of ether-insoluble extractives, based on the oven-dry weight of the wood.¹⁷

The wet shear strength of apitong veneer treated with the four chemicals more or less followed the same trend as that determined by dry shear test, with the exception of sodium hydroxide (Table II). Treatment with this chemical affected the water resistance of the panels glued with urea formaldehyde resin. This may be due to the dispersion effect of sodium hydroxide on the resin when the shear specimens were subjected to hot water as evidenced by the low wood failure value obtained. This phenomenon was reported by Louis and Drennen¹⁸ on wet strength of paper.

Results of breaking loads, determined by hot and cold soak shear test, of the five treatments met the requirements of the Japanese standard.¹⁴

A regression analysis made on the wettability and dry shear strength values showed a significant correlation between these two properties. Dry shear strength increased linearly with wettability or cosine contact angle as shown in Figure 1. This corroborates the findings of Bodig⁴ and Gray.⁶

CONCLUSIONS

Extraction of apitong veneer with methanol-benzene solution greatly improves its wettability and gluing properties. The improvement is probably due to the removal of extractives from the wood which interfere with bond formation.

Surface treatment with chemicals affects the wettability and gluing properties of apitong veneer. Among the four chemicals used, methanol-benzene is the most effective in improving the wettability and gluability of this species, followed by sodium hydroxide, although the latter decreases the water resistance of the bond produced with urea formaldehyde resin. Acetone does not have a significant effect on wettability and gluability while ether adversely affects both properties.

There is a high correlation between wettability and gluability of apitong veneer. Bond strength increases linearly with increasing wettability.

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