Laboratory manufacture of high moisture southern pine strandboard bonded with three tannin adhesive types

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Abstract

Three types of tannin-based adhesives (wattle, quebracho, and radiata pine) were examined as binders for strandboard panels at two binder solids (10% and 13%) application levels. The strand moisture content (MC) was 6 percent or higher with an objective of having an out-of-press panel MC of 6 percent or higher. Characteristics (viscosity, stability, and gel time) of the tannin solutions and final tannin adhesives with cross-linking compounds are described. The strandboard panels were tested for internal bond, bending properties, durability after an accelerated aging test, and thickness swell after exposure to water. The preliminary panels bonded with 10 percent binder solids and 5.5 to 6.5 percent MC strands showed some promising test results, with some exceptions meeting or exceeding Canadian standards for strandboards. The final series of panels had varied physical/mechanical test results, due in part to strand MCs that were as high as 8.6 percent. Further work is suggested on fine-tuning the cross-linking compounds and the press processing parameters for tannin adhesives for strandboard manufacture.

Tannin has been voluminously reported in the literature and only selective comments are reported in this article. Tannins are generally classified into two chemical compounds of mainly phenolic nature: hydrolizable tannin and condensed tannins (Pizzi 1983, Hergert 1989). The hydrolizable tannins have had medicinal and cottage applications since antiquity but there is little mention in the literature of their application for adhesives (Hergert 1989). Hills (1979) reported that the vicinal trihydroxy phenolic moiety of hydrolyzable tannin is not readily amenable in the preparation of adhesives, but it may form a useful extender for phenolic resins, as in the case of chestnut-wood extracts (Kulvik 1976, 1977). The historic preservative treatment (tanning) of leather with tannin is well known. Pizzi (1983) stated that 90 percent of the total world production (~ 350 kt) of commercial tannin is of the condensed type, and the condensed tannins are both chemically and economically more viable for the preparation of resin adhesives. Condensed tannins are known for their wide distribution in nature and for their substantial concentration in the wood and bark of various softwood and hardwood trees, including various acacia (South African wattle of mimosa bark extract), *Schinopsis* (Argentina quebracho wood extract), *Tsuga* (hemlock bark extract) and *Pinus* (pine bark extracts) species. Mangrove (*Rhizophora* spp.) bark is another source of tannin (Teck et al. 1993).

Herbert (1989) reported about 10 kt of condensed tannins (bark extracts of *Acacia mearnsii*) were utilized as adhesives in comparison to 300 to 400 kt of phenol consumed for the same purpose. Herbert (1989) further stated that 30 to 50 percent tannin replacement of the resin solids in amino (urea-formaldehyde [UF] and melamine-formaldehyde [MF]) and phenolic (phenol-formaldehyde [PF] and resorcinol-formaldehyde [RF]) resins have been formulated into wood adhesives. Research conducted in North America with bark tannin extracts of hemlock, Douglas-fir, and redwood began as early as 1945 (Herbert 1989),

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and some limited utilization of the tannins occurred for about two decades. Commercial tannin production has been successful in South Africa, Brazil, and Argentina, with most utilization for adhesives occurring in South America, South Africa, Finland, Russia, Australia, and New Zealand. Commercial tannin products in small quantities have been intermittently produced in New Zealand (*Pinus radiata* bark extract, U.S. Patent 5,417,888 by Collins and Yazaki), Chile (*Pinus radiata* bark extract), and the United States of America (pecan shell inner lining, Tannex®, or Resorcinex®) without sustained economic success as a wood adhesive. Worldwide, wattle tannin has achieved more commercial success than other tannins.

Dalton (1950) discussed tannin adhesives for wood in Australia. Then, a flurry of tannin activity as wood adhesive occurred and was reported during the early to mid-1970s, due to the high price of phenol (Saayman and Oatley 1975). In 1975 (Roux), described wattle tannin as:

a. wattle tannin is water soluble while quebracho has to be bisulfited to be water soluble;
b. wattle tannin is renewable from an 8-year tree rotation resource versus quebracho which requires trees more than 100 to 150 years old;
c. wattle tannin requires less cross-linking agent (e.g., formaldehyde) than other tannins;
d. wattle tannin has a molecular weight range of 300 to 3,000, with an average of about 1,250;
e. tannins, including wattle tannin, increase glueline brittleness; and
f. the 1975 wattle tannin price for leather tanning was quite high but the surplus tannin available for adhesives has been "artificially kept approximately one cent below the phenol market price."

Saayman (1975) reported wattle tannin utility in resole-type PF resins for bonding wood veneers requiring up to 30 percent PF fortification to obtain a fully water-resistant bond (boil test criteria, presumed). In 1976, Saayman's (1975) comments were cursory verified by testing southern pine plywood by a two 4-hour boil test procedure that had been bonded with a PF/tannin adhesive (Sellers 1994). Scharfetter et al. (1977) discussed in some detail pine (South Africa *Pinus patula*, etc.) bark tannins, along with wattle type for wood adhesives.

Miller (1999) reported on extensive work with tannin:PF resin solids blends (80:20 ratio, respectively) for strandboard panels. Miller's study involved tannin from *Acacia mearnsii* (Brazil and South African via Australia sources) and *Pinus radiata* (Chile source) with much learned about tannin analysis techniques. The strandboard results were not as successful as desired, in part due to the low resinated strand moisture content (MC) (~ 9% ± 3%, ovendry fiber weight basis) that is typical in current strandboard manufacture with thermoset resins but is insufficient for flow of tannin-based adhesives.

Teck et al. (1993) evaluated mangrove bark tannin in particleboard made of rubberwood (*Heava* spp.) and compared it to board made with three synthetic resins (UF, PF, and polymeric diphenyl methane diisocyanate [pMDI]) as well as with blends of tannin and synthetic resins. The test results were compared to the requirements of British Standard BS 5669-1979, Type I Bond. The particleboards containing 14 percent tannin binder solids (dry fiber weight basis) passed the standard for modulus of rupture (MOR), internal bond (IB), screw withdrawal, and thickness swell (TS). Panels bonded with all synthetic resins passed the standard as did the tannin blended with 10 percent UF resin and tannin blended with 8 to 10 percent pMDI, tannin solids basis. Panels bonded with the tannin/10 percent PF blend did not pass the standard (low MOR, high TS, and borderline IB).

Pizzi (1999) reviewed the current status of technology on phenolic and tannin adhesives for panel products. He demonstrated old and new technology for cross-linking mechanisms as related to wood adhesive chemistry. Roll (1999) reviewed European tannin adhesives for 50:50 surface:core oriented strandboard (OSB) that passed fully exterior, marine-grade requirements. The pine strands (presumed to be European pines) in Roll's work were 6.5 percent initial MC and the resinated strands (before hot-press curing) were 24 percent MC, ovendry fiber weight basis. The industrial board size was 2,440 by 1,200 by 12 mm, density 650 to 700 kg/m^3, press temperature 205°C, and press time at 12 s/mm. Panel MC after hot-press curing was 11.5 to 12.3 percent MC. The pine tannin adhesives were prepared in a 40-percent solution with one of two types of hardener added (paraformaldehyde or a low molecular weight PF resin with a higher than usual free formaldehyde). Berg et al. (1999) reviewed Chilean efforts in utilizing tannin (*Pinus radiata* bark extracts) adhesives for commercial particle board panels and OSB laboratory panels. Since 1995, commercial particleboard for exterior applications (tannin binder) had been produced in Chile and sold principally in Europe.

The objective of this project was to re-examine three condensed tannins (wattle, quebracho, radiata pine) as a renewable resource adhesive for southern pine strandboard panels. Another objective was to realize out-of-press panel MCs of more than 6 percent in panels bonded with the tannin adhesives to enhance panel dimensional stability prior to product shipment.

Materials and methods

Tannins

Three tannins with specification descriptions as follows were examined.

Quebracho tannin
Fintan® 737
Flavonoid polymers, pH 7.0 to 8.5
(Bakelite 2002)

Wattle tannin
Bondtite® 645
Tannins, modified, pH 6.5 to 7.5
(Bakelite 2002)

Pinus radiata tannin
Indutan®
Bark tannin, pH 5.8
(DITECO Ltd. 2003)

The tannins were evaluated for physical/chemical characteristics (Iowa Testing Laboratories, Eagle Grove, IA) per Association of Official Analytical Chemists (AOAC) test procedures and in part with an inductive plasma coupling (ICP) instrument. The physical characteristics included moisture, protein, carbohydrate, fat, fiber, ash, and pH. Sixteen chemical elements were determined for each tannin.

Cross-linking agents

Hexamethylenetetramine (hexa, $C_6H_{12}N_4$, Sigma Chemical Co., St. Louis, MO) was one cross-linking agent. A second cross-linking agent was a liquid PF resin (code SS-101, Dynea Resins, Moncure, NC) described as a

face resin binder for OSB with the following specifications:

PF specification	Reported	At time of application
Viscosity	120 to 180 cP or mPa•s	183 to 208 cP or mPa•s
Nonvolatile solids	49.5% to 50.5%	--
pH	10.1 to 10.5	--
Alkalinity	3.5% to 3.7%	--
Free formaldehyde	< 0.10 %	--
Gel time (at 100°C)		18 minutes

Wood strands

The wood strands were obtained from a regional OSB mill (Nexfor, Guntown, MS) and consisted of southern pine (principally, *Pinus taeda* with possibly some *P. echinata*). The strands were about 115 mm long, variable widths (up to 60 mm), and about 0.7 mm thick. The strands received had a MC of 3 to 4 percent and were allowed to equalize in the laboratory to 6 to 7 percent, ovendry fiber weight basis. Southern pine grown in the United States are considered to be some of the highest density softwoods in the world in comparison to European pines presumed to be utilized by Roll (1999).

Tannin adhesive preparation

Aqueous solutions of each tannin extract powder (40% tannin at 10% MC, 60% water) were prepared and observed for stability and pot life over 2-, 4-, and 24-hour periods. Viscosity measurements were made with a Brookfield RVTD digital viscometer, and solution solids contents were determined with a digital moisture balance (CSC Scientific Company, Fairfax, VA). The PF resin was analyzed for viscosity and gel time. Tannin adhesives were made by mixing ~36 percent tannin solutions at 90 parts by weight with five parts cross-linking PF resin and five parts hexa. The tannin adhesives were observed for stability and pot life with procedures similar to the tannin solutions. In addition, each tannin adhesive was tested for gel time (Sunshine Gel Timer) at 100°C and for viscosity.

Board manufacture

The tannin adhesives were applied with a Concord EL-2 atomizer (Coil Manufacturing Ltd., Surry, B.C. Canada) at about 1,047 rad/s (10,000 rpm) in a 1,830-mm diameter tumbling blender. A Dieffenbacher hot press with PressMAN® computer controls and monitoring system was utilized to make the strand panels. The PressMAN system provides a color-coded printout of each hot-press cycle, showing internal (core) and surface (platen) panel temperatures, mat pressure, core gas pressure, core temperature, and mat thickness.

Initially, two strandboards (864 by 864 mm in size, 11 mm thick) per tannin formulation were made at one binder solids application (10%, dry fiber weight basis) and at one wax-emulsion application (1.5% wax solids, dry fiber weight basis). The panel mats were hand felted and were homogenous. The mat (resin/wax coated strands) MC ranged from 17 to 23 percent. The hot-press cycle at 200°C was 3.5 and 4.0 minutes (one panel each time), with the platen pressure computer-controlled for the desired panel thickness (11 mm). (Due to the high mat MC, lower hot-press pressures are required.) Decompression time was extended to 40 seconds. The target panel density was 560 to 700 kg/m^3 (40 to 44 lb./ft.^{3}). The objective out-of-press panel MC was 6 percent or higher.

With no panel ruptures (864 by 864 mm size) experienced with the initial panels (11 mm thick) and panel mechanical/physical properties appearing satisfactory, the project panels were processed. To conserve tannin raw materials, the final project panels were made 560 by 610 mm in size, 11 mm thick. Four panels per condition were made for the final experiment (three tannin formulations and a control PF resin, two resin solids application levels [10% and 13%, dry fiber weight basis], and four replications). The binder application rates in this study are substantially higher than current practice with synthetic thermoset binders. The reasons for the high tannin solids levels were related to past documented industrial experience (e.g., South Africa) and the desire to elevate the resinated strand MC as well as to elevated the final panel out-of-press MC.

Panel testing

Panels were trimmed for density calculation and tested for MC immediately out of the hot press. The mechanical/physical tests included bending strength (MOR) and stiffness (modulus of elasticity, MOE), tension perpendicular to the surface (IB), MOR strength after an six-cycle accelerated aging tests (PS 2-92, Moisture Cycled Breaking Load) with the percent strength retention calculated, relative density, and TS and water absorption (WA) after a 2-hour and 24-hour water soak test. These properties were compared to Canadian standards for strandboard and waferboard (CSA 1993, Lowood 1997).

Results and discussion

Tannins

The elemental analysis showed some similar (nonsignificant) elemental results among the three tannins, but the radiata pine bark tannin had the lowest pH (4.8) and the greatest content of sulfur, manganese, iron, aluminum, and organic silicon (**Table 1**). The equalized MC of the tannin extract powders was about 10 percent. These data are not analyzed to add up to 100 percent, and not all test techniques are perfect for tannins (i.e., carbohydrates).

Tannin solutions

Generally, the technique was to mix the tannin in a 40 percent aqueous solution and let stand about 24 hours at 20°C (**Table 2**) before making the final adhesive formulations. All powdered tannin extracts have some MC (**Table 1**), thus the actual nonvolatile solids of the initial wetted tannin solutions are nearer to 36 percent. The initial viscosity of the water/tannin solutions varied from 38 to 516 mPa•s and were fairly stable over the 24-hour period in an open container (**Table 2**).

Tannin adhesives

Generally, the final tannin adhesives were made with the 24-hour-old tannin solutions by mixing five percent of PF resin and "hexa", and sprayed quickly after mixing. The wattle and radiata tannin adhesives were fairly stable over a 2- to 24-hour period, but the quebracho tannin adhesive was stable only for a few hours (**Table 3**), indicating some reactivity of the adhesive ingredients. At elevated temperatures (100°C), the gel time of tannin adhesives was very fast (2 to 3 min.) (**Table 3**). The reactivity resulted from the cross linking of the tannin adhesive with hexa and/or the interaction of the highly alkaline PF (pH 10+) with the tannin solution (pH < 7).

Preliminary strandboard panels

The tannin adhesive formulations were atomized on 5.5 to 6.5 percent MC

Table 1. — Physical/chemical analysis of three tannin raw materials.[a]

Physical/chemical property	Tannin type		
	Wattle	Quebracho	Radiata
% Moisture	9.60	10.60	9.80
% Nitrogen	0.36	0.21	0.13
% Protein	2.23	1.29	0.80
% Fat	0.39	0.28	0.22
% Fiber	0.21	0.04	0.28
% Ash	7.43	8.87	9.33
% NFE[b]	89.73	89.52	89.37
% Carbohydrates	89.94	89.56	89.65
% TDN[c]	92.36	91.09	90.39
Dig. energy (Kcal/lb.)	1,685.98	1,660.45	1,646.48
Met. energy (KCal/lb.)	1,611.00	1,589.76	1,577.99
% Calcium (Ca)	0.15	0.25	0.12
% Phosphorus (P)	0.02	0.03	0.07
% Potassium (K)	0.42	0.16	0.80
% Magnesium (Mg)	0.12	0.13	0.14
% Sulfur (S)	0.03	0.40	1.02
% Sodium (Na)	2.36	2.82	2.21
ppm Zinc (Zn)	7	22	33
ppm Manganese (Mn)	25	4	131
ppm Copper (Cu)	4	3	11
ppm Iron (Fe)	45	29	736
ppm Cobalt (Co)	<1	<1	2
ppm Aluminum (Al)	58	45	1,768
% ADF[d]	1.19	1.77	48.47
% Chloride (Cl)	0.25	0.11	0.42
Molybdenum (Mo)	1	1	<1
ppm Silicon (Si)[e]	87	62	225
pH	5.8	6.5	4.8

[a] Test procedures were run per AOAC and in part with an inductive plasma coupling (ICP) instrument.
[b] NFE = nitrogen free extract.
[c] TDN = total digestible nitrogen (protein).
[d] Acid detergent fiber (ADF) values are typically greater than crude fiber values, but are relative.
[e] The silicon values are organic-type silicon and do not reflect inorganic silicon values.

Table 2. — Characteristics of three tannin solutions.

Tannin type	Nonvolatile solids[a]	Brookfield viscosity @ 20°C	
		Initial	After 48 hours
	(%)	- - - - - - (mPa•s) - - - - - -	
Wattle	35.2	38	42
Quebracho	36.0	388	577
Radiata	35.6	516	795

[a] The tannin solutions contained 40 parts tannin at ~10 percent MC and 60 parts water.

strands at 10 percent binder solids, and a 11-mm board of about 672 to 704 kg/m^3 (42 to 44 lb./ft.^{3}) was made. The time from resin adhesive application to hot pressing ranged from 11 to 24 minutes. One panel was hot-press cured at 4 minutes and one panel at 3.5 minutes for each tannin type (**Table 4**). The mat MC before entering the hot press was 15.5 to 16.1 percent. The test results of the preliminary panels, with some exceptions, generally met, or exceeded, the criteria of the Canadian Standard 0437-Series 93 for (random orientations) OSB and waferboard (CSA 1993) (**Table 4**). The values that did not meet the standard involved IB, MOR strength retention after an accelerated aging test, and TS, and generally involved panels hot-press cured at the longer press time (4.0 min.). However, it was decided to hot-press cure the final test panels at the longer press time.

Final strandboard panels

The final test run involved hot-press curing four panels at two binder solids levels (10% and 13%), all at 4-minute hot-press time (**Table 5**). The process lay up time (before hot pressing) was about the same as with the preliminary panels (11 to 24 min.). The period of eight weeks that this gluing occurred was very humid (about 250 to 380 mm of rain per month), and the laboratory is not climate controlled. The equalized MC of the strands prior to binder application ranged from 6.0 to 8.6 percent, and the resultant mat MCs were 13.1 to 17.6 percent for the 10 percent binder applications and 16.4 to 21.6 percent for the 13 percent binder applications, showing the impact of environmental conditions. The lower mat MCs were those of the control resin which had the lower strand MCs. The internal panel gas pressure and temperatures are shown in **Table 5**, showing the magnitude of the higher MC panels influences. The hot press was programmed for ~11 mm thickness base. The actual thickness ranged from 11.7 to 12.2 mm. Some panels delaminated around the edges at the 13 percent binder solids application (quebracho and PF control), indicating under cure at the higher MC levels (**Table 5**). The test results varied compared to Canadian Standard 0-437 for random orientation OSB and waferboard. While the panels exceeded the minimum MOR test criteria, none of the panel averages for MOR strength retention after accelerated aging reached the 50 percent minimum standard (**Table 5**). All panel MOE averages exceeded the MOE standard, except the panels that ruptured on the edges (**Table 5**). All but one of the TSs after the 24-hour soak test did not meet the Canadian Standard (< 15%), and 50 percent of the values were unacceptable by U.S. standards (< 25%) (U.S. Dept. of Commerce 1992). Three of eight conditions met or exceeded the IB

standard (345 kPa). Selected panel MCs out of the hot press ranged from 6.0 to 8.6 percent.

Conclusions

This work with U.S. southern pine (one of the world's most dense softwoods) strands bonded with three tannins did not result in similar exterior-grade panels suggested by Roll (1999) with some of the same tannins and at some of the same conditions (i.e., 10% to 13% binders solids, 6.5% strand MCs and up to 24% MC resinated strands [mat MC]). In some cases, Roll (1999) utilized a low molecular weight PF resin with higher than usual free formaldehyde and/or paraformaldehyde cross linker, other lower density, European pine species, and a hot-press schedule that resulted in out-of-press MC of 11.5 to 12.3 percent.

The results in this study are sufficiently promising to continue pursuing research of bonding southern pine strandboard products with tannin-based adhesives. Future work should concen-

Table 3. — Characteristics of three tannin adhesives.

Viscosity time	Tannin adhesive type		
	Wattle	Quebracho	Radiata
	- - - - - - - - - (viscosity, mPa•s or cP, Brookfield) - - - - - - - - -		
Initial	99	114	1,035
After 2 hours	91	168	1,080
4 hours	106	450	908
24 hours	295	settled out of solution	1,002
Gel time @ 100°C (min.)	190 s	291 s	116 s
Nonvolatile solids (%)	39.7	39.5	38.2
Adhesive appearance	smooth	foam	foam

Table 4. — Preliminary strandboard (11 by 834 by 834 mm) test results bonded with 10 percent tannin binder solids, dry fiber weight basis.

Adhesive type	Press time[a]	Mat moisture	Core (peak)		Panel density	IB		MOR	MOR strength retention	MOE	2 hour		24 hour	
			Gas pressure	Temperature		Avg.	Range				TS	WA	TS	WA
	(min.)	(%)	(kPa)	(°C)	(kg/m^3)	- - - (kPa) - --		(MPa)	(%)	(MPa)	- - - (%) - - -		- - - (%) - - -	
--	CAN Std. (Random):				(610 to 710)	345	--	17.2	50	3,100	--	--	15[b]	--
Wattle	4.0	16.1	434	315	705	379	(152 to 703)	26.1	50	3,405	4.7	6.6	14.9	26.0
Wattle	3.5	16.1	434	348	710	248	(41 to 703)	29.7	52	3,712	5.1	8.1	16.3	38.1
Quebracho	4.0	16.5	503	336	726	331	(269 to 476)	24.6	55	3,342	5.5	5.9	19.6	27.9
Quebracho	3.5	16.1	434	312	726	386	(228 to 455)	33.2	25	4,531	6.9	7.5	24.0	42.2
Radiata	4.0	15.5	427	336	718	469	(303 to 586)	22.7	50	3,300	6.1	6.6	21.0	34.6
Radiata	3.5	15.5	512	372	705	310	(131 to 420)	20.9	50	3,107	6.1	9.0	23.4	50.4

[a] Press time at 200°C temperature.
[b] The U.S. Standard PS-2 (1995) thickness swell maximum is 25 percent. Strand moisture ranged from 5.5 to 6.5 percent for resination. Two panels per condition were made, thus the specimens for the values reported are 16 for IB, 4 for MOR and all other values.

Table 5. — Test results[a] of 11-mm-thick strandboards (560 by 610 mm) bonded with three tannin binders and a PF control resin, pressed at 200°C for four minutes.

Adheisve type	Applied binder solids[b]	Mat moisture[c]	Core (peak)		Panel density	IB	MOR	MOR strength retention	MOE	2 hour		24 hour	
			Gas pressure	Temperature						TS	WA	TS	WA
	- - - (%) - - -		(kPa)	(°C)	(kg/m^3)	(kPa)	(MPa)	(%)	(MPa)	- - - (%) - - -		- - - (%) - - -	
--	Can. Std. (Random):				(610 to 710)	345	17.2	50	3,100	--	--	15[d]	--
Wattle	10	17.6	276	310	688	345	26.9	39	3,802	9	13	23	57
Wattle	13	20.6	359	324	704	234	23.1	29	3,724	5	7	13	26
Quebracho	10	17.5	227	303	704	248	21.3	15	3,735	16	17	42	78
Quebracho	13[e]	18.6	276	293	688	159	18.5	--	2,754	10	14	26	56
Raidata	10	15.9	314	282	608	289	21.2	12	3,221	12	13	39	73
Radiata	13	21.6	358	300	704	345	24.1	34	3,629	10	11	29	61
PF control	10	13.1	186	286	672	490	28.9	26	4,498	7	11	20	46
PF control	13[e]	16.4	--	--	688	124	--	--	3,039	7	16	19	51

[a] Each property is an average of four panels (four specimens for density, 24 for IB, eight for MOR, eight for MOR and all other values).
[b] Binder solids based on dry fiber weight basis.
[c] Stand moisture average 7.4 percent; range = 6.0 to 8.6 percent.
[d] The U.S. Standard PS-2 (1995) for thickness swell is 25 percent.
[e] Panels had edge delaminations.

trate on cross-linker compounds and process parameters (MC and hot press factors). A particular need is more accurate data on internal hot-press panel temperatures and pressures (gaseous) as relates to tannin adhesive cure rates.

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