Tool wear properties of five extender/ fillers in adhesive mixes for plywood

Terry Sellers, Jr.✳
George D. Miller✳
Wade Smith

Abstract

Southern pine veneers were bonded into plywood with phenol-formaldehyde adhesives containing one of five different extender/fillers. The five fillers were 1) a 65/35 ratio blend of English walnut shell flour/oat hull furfural residue; 2) an oat hull furfural residue; 3) an imported Asian furfural residue; 4) an English walnut shell flour; and 5) a pecan shell flour. The fillers were analyzed for 16 chemical elements, ash, pH, and moisture content. An alder bark flour (West Coast, U.S. source) was also analyzed for elemental analysis for comparison. The plywood was tested for glueline durability by an accelerated-aging (vacuum-pressure water) test and with a two 4-hour boil test regime. The knife wear at the plywood gluelines was tested for each filler-type adhesive. Differences in knife wear were evident depending on the filler-type adhesive. The plywood with the 65 percent walnut shell flour/35 percent oat hull furfural residue filler had the lowest knife wear, and the plywood bonded with the Asian furfural residue had the highest knife wear. The Asian furfural residue had a low pH and a high presence of sulfur, chloride, iron, aluminum, and organic-type silicon (and generally ash) compared to the other fillers tested for knife wear.

The term "filler" in this report refers to relatively nonadhesive substances added to an adhesive binder to improve working properties, permanence, strength, or other qualities (ASTM and ISO vocabulary). Usually, fillers are either lignocellulosic or inorganic in nature and are often added to reduce costs. Proteinaceous and amylaceous materials are most often called extenders because they can enter into the matrix of the binders. Fillers in North American structural-plywood adhesives often contain up to 80 percent carbohydrates; therefore, they are often referred to as "extenders." Fillers are extensively utilized in adhesives and sealants, coatings, plastics, medicines, and cleaning compounds (Sellers 1994).

Stone and Robitschek (1978) reported how furfural extenders (fillers) differ principally in their ability to build viscosity and hold glues on the surface of veneers. As applied in phenol-formaldehyde (PF) resin-adhesive formulations, the work found that various physical characteristics of the fillers were not as significant as the inherent chemical nature of the materials in the filler. Sellers (1985) wrote a chapter on fillers as applied to the plywood industry, outlining the wetted mix and caustic dispersion mix characteristics of various filler types.

Stevens (1974) describes an apparatus to monitor the "abrasiveness" of wood panel products. Stewart (1985) describes a turning method for defining knife wear. Stewart (1992) and Sellers (1997) described how cutting knife wear was related to metal corrosion and accelerated at high temperatures, and showed that mechanical wear mechanisms may remove the corrosion materials. Therefore, abrasion is only one of several wear mechanisms. Knife wear of plywood bond (glue) lines containing a furfural residue or a nutshell/mineral (inorganic) blend have been described by Sellers and Gardner (1989). In this work, substantial wear occurred after 230 m of plywood passed the cutter knives. Sellers (1989) demonstrated that pecan nut shell flour as filler resulted in less knife wear than two types of furfural residue fillers. Similar results compar-

The authors are, respectively, Professor Emeritus and Research Associate, Mississippi State Univ. Forest Products Laboratory (MSFPL), Forest and Wildlife Research Center, Box 9820, Mississippi State, MS 39672-9820; and Quality Assurance Manager, Bates and Company, Orange, TX. This project was partially funded by Bates and Company. The use of trade, firm, or corporation names in this article is for the information and convenience of the reader. This use does not constitute an official endorsement or approval by MSFPL or Mississippi State Univ. to the exclusion of others that may be suitable. This paper was received for publication in November 2003. Article No. 9798.
✳Forest Products Society Member.

Forest Prod. J. 55(3):27-31.

Table 1. — Sieve analysis of six filler types.[a]

U.S. Standard Sieve		65% E. walnut shell / 35% oat hull furfural residue	Oat hull furfural residue	Asian furfural residue	E. walnut shell flour	Pecan shell flour	Alder bark flour
(No.)	(μm)	(% retained)					
50	300	0	0	0	0	0	0
100	150	< 1	< 1	1 to 3	< 1	< 1	< 1
200	75	4	3 to 9[b]	9 to 25[b]	< 8	~ 5	~ 10

[a]Sieve analysis determined by jet air classification (Hosokawa Micron System).
[b]Two different tests.

Table 2. — PF adhesive mix characteristics (low shear mixer) for laboratory gluing of two fillers.

Ingredient	Amount	Non-volatile solids	Adhesive mix solids
	(%)		
Water	18	- -	- -
Extender (wheat flour)	6.5	~ 95	6.2
Filler	6.5	~ 95	6.2
Mix 5 min. until smooth			
PF resin (GP 5779)	21 to 26	43	9.0 to 11.2
Mix 3 to 5 min. until smooth			
Sodium hydroxide (NaOH)	3	50	1.5
Mix 20 min. (paddle mixer)			
PF resin (GP 5779)	40 to 45	43	17.2 to 19.4
Mix 3 to 5 min. until smooth, add in two additions			
Total resin solution	66		
Resin solids	28.4		
Total mix solids	42.3		
Total mix water	57.7		
Total mix	100.0		

ing pecan nut shell with two furfural types were exhibited in another study by Sellers et al. (1990), after only 152 m of plywood bondlines passed the shaper knives.

This study involved evaluating the knife wear of five filler types in commercially produced 4-ply, 12.5-mm (0.5-in.) southern pine plywood bonded with PF resin adhesives. Certain physical characteristics and chemical elemental analyses were performed for each filler to identify which chemical element(s) may be influencing the knife wear.

Materials and methods

Industrial plywood and filler types

Four panels, approximately 610 by 610 mm in size and 12.5-mm thick, were supplied from commercial southern pine plywood manufacturers for each filler-type PF resin-adhesive formulation. The panels were 4-ply, thus having three bondlines. The filler types in the PF formulations were: 1) a 65 percent English (E.) walnut shell flour/35 percent oat hull furfural residue blend; 2) an oat hull furfural residue; 3) an Asian source furfural residue; 4) an E. walnut shell flour; and 5) a pecan shell flour residue.

Filler physical/elemental analysis

Each filler was evaluated for certain physical characteristics, i.e., color, moisture content, pH, ash, and sieve size (**Table 1**). The sieve analysis was determined with an air jet sieve over a range of sizes (Hosokawa Micron Systems, Summit, NJ). Each filler was also analyzed for content of 16 chemical elements. A sixth filler, an alder bark flour (West Coast, USA source), was only evaluated for physical/chemical analysis for comparison.

Laboratory plywood

Four panels each of southern pine plywood utilizing the Asian source furfural residue and oat hull furfural residue were re-glued at Mississippi's Forest Products Laboratory for knife wear re-verification. This exercise was fostered by the initial higher tool wear results of plywood containing Asian furfural filler versus a U.S. standard oat hull furfural filler. The PF adhesive mix (**Table 2**) contained 28.4 percent PF binder solids (Georgia-Pacific Resins code GP 5779 Plywood PF Resin), which is a typical formulation for southern pine plywood. The plywood conditions for gluing were as follows:

- Veneer: regional southern pine, 3 to 4 percent moisture content, 3.2 mm thickness;
- Panel construction: 4-ply, 12.7 mm thick, 25 mm wide, 50 mm long;
- Adhesive spread: 214 g/m^2, single glueline basis, rubber roll coated applied;
- Assembly time: 15 minutes closed (< 1 min. open) and no cold prepress;
- Hot press: 141°C temperature, 1379 kPa pressure, 5.5 minutes.

Bondline knife wear tests

The knife wear procedure (MSFPL 1987) involved a shaper with a mechanical feed mechanism. In this study, 152 linear m of plywood were fed into the machine uniformly in 3.2-mm increments for each pass of a single cutter knife, which was rotating about 607 rad/sec. (5,800 rpm). The knives passed by the end grain of the face and back plies, which means the two parallel inner plies are treated as a single core. The knife wear was determined by measuring the weight loss of the freshly polished knives and observed by photographing the depth of notches on the knives at the three bondlines (gluelines). The bondlines near the two faces (front and back) have veneers perpendicular to each other and the center bondline has veneers parallel.

Adhesive bond assessment

While not a requirement of the study, the plywood bond was assessed. Part of each test panel was cut into 40 plywood tension-shear specimens (USDC 1995). Twenty test specimens were submitted to a standard vacuum-pressure test and 20 to two 4-hour boil test (PS 1-95) (USDC 1995). The shear strengths on the breaking load of wet specimens were recorded, and the wood failures on the sheared and dried specimens were observed and estimated to the nearest 5 percent wood failure (range 0% to 100%).

Figure 1. — Appearance of six filler flours for bonding plywood in PF resin-adhesives. Top row, left to right: 65 percent E. walnut shell flour/35 percent oat hull furfural residue; oat hull furfural residue; Asian furfural residue. Bottom row, left to right: E. walnut shell flour; pecan shell flour; alder bark flour.

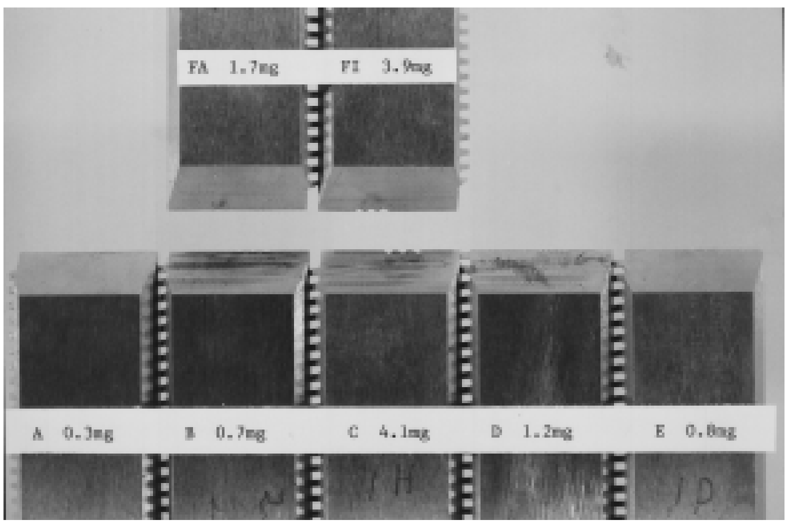

Figure 2. — Photograph showing knife edge recession and knife weight loss after cutting 152 linear m of 4-ply, 12.7-mm plywood (three bondlines) bonded with PF adhesives containing five different fillers. (A = 65% E. walnut shell/35% oat hull furfural residue; B [FA] = oat hull furfural residue; C [FI] = Asian furfural residue; D = E. walnut shell flour; and E = pecan shell flour.)

Results and discussion

Physical/chemical filler characteristics

The ash content (600°C) of the six fillers examined ranged from 1.1 to 15.0 percent, with the Asian furfural residue being the highest and the E. walnut shell flour being the lowest (**Table 3**). The Asian furfural and alder bark flour residues had substantially more iron and aluminum than the other filler types (**Table 3**). The furfural residues were less than 3.0 pH, while the nut shell flours were about 5.0, or higher, pH. The sieve analysis showed similar mesh size characteristics among the filler types (**Table 1**). The filler color ranged from light tan (E. walnut shell flour) to very dark brown (Asian furfural residue flour) (gray to black in **Fig. 1**).

Knife wear results

Since all adhesives mixes contained similar ingredients except for different fillers, the knife wear at the bondlines reflected the effect of the different fillers. This correlation implies that the amount and types of chemical/physical material and the ash in the fillers of the bondlines cause wear of the knives after the knives cut plywood. Therefore, possible correlations between the knife wear of the bondlines and the elemental analyses of the five fillers are discussed. (The wood veneers did not cause measurable wear in this procedure.)

In this experiment, the ash content correlated with the tool wear, with lower ash content fillers experiencing the least knife wear (**Table 3**). The Asian furfural residue had 2 to 14 times more knife wear (as measured by knife weight loss) than the other fillers (**Fig. 2**, **Table 3**). Nut shell flours, such as pecan shell and walnut shell, are known for having low knife wear. Furfural residues may have high iron and aluminum chemical elements present unless the processing equipment (reactors, etc.) that makes the furfural is stainless steel. Furfural residues often have low pH (< 3.0) due to the sulfur-type acids utilized to produce furfural. The Asian furfural residue filler had an ash content of 15 percent in the original samples and 4.9 percent in the resubmittal sample, with about 1 percent iron, 1 percent aluminum, and minor amounts of other elements, indicating the presence of other nonburning inorganic materials. The other ash materials were likely dirt/sand-type and organic silicon materials. Also, the chemical elements and ash of furfural residues depend on the initial raw materials, e.g., corn cobs, oat hulls, rice hulls, bagasse, etc. Even with a resubmittal (repeat) and care taken to reduce the ash contributing factors for the Asian furfural residue filler, iron, and aluminum remained high and the pH low (< 3.0) due to high S and Cl corrosive agents (**Table 3**).

Laboratory adhesive mix

The adhesives mixed in the laboratory (**Table 2**) for two of the fillers (repeat evaluation) blended well but required differing amounts of resin added prior to the sodium hydroxide dispersion. The Asian filler required more pre-NaOH resin addition to have a workable mix viscosity. Both filler-type mixes had satisfactory initial mix viscosity and mix working life as shown in **Table 4**.

Table 3. — Physical/chemical characteristics of six fillers and knife-weight loss for five fillers in PF adhesives used to bond southern pine plywood.

Physical/elemental property[a]	Unit	Filler type							
			Oat hull furfural residue		Asian furfural residue				
		65% E. walnut shell/ 35% oat hull furfural residue	Original	Repeat	Original	Repeat	E. walnut shell flour	Pecan shell flour	Alder bark flour
Moisture	%	7.2	6.1	10.3	6.9	4.4	7.2	10.2	8.4
pH (10% solution)	No.	3.71	2.62	2.50	2.98	2.40	5.76	4.99	4.65
Ash (600 °C)	%	5.1	8.0	10.8	15.0	4.9	1.1	1.5	5.2
Nitrogen free extracts	%	33.2	31.7	36.1	39.0	46.8	33.1	33.3	35.2
Carbohydrates	%	88.7	87.1	84.1	80.3	92.1	92.3	93.3	89.1
Protein	%	3.9	3.6	4.0	3.9	2.6	3.3	2.7	3.5
Fat	%	2.2	1.1	1.0	0.7	0.5	3.2	3.1	2.0
Fiber	%	55.5	55.4	48.1	41.2	45.1	59.1	59.9	53.9
Nitrogen (N)	%	0.62	0.59	0.64	0.64	0.41	0.54	0.33	0.57
Calcium (Ca)	%	0.14	0.12	0.13	0.15	0.27	0.15	0.63	1.09
Phosphorus (P)	%	0.10	0.11	0.13	0.06	0.04	0.05	0.02	0.03
Potassium (K)	%	0.58	0.83	0.58	0.72	0.62	0.34	0.19	0.23
Magnesium (Mg)	%	0.09	0.14	0.13	0.04	0.07	0.04	0.04	0.08
Sulfur (S)	%	0.29	0.63	0.72	0.53	0.49	0.0	0.02	0.04
Sodium (NA)	%	0.03	0.04	0.07	0.02	0.55	0.02	0.02	0.01
Chloride (Cl)	%	0.05	0.06	0.12	0.03	0.18	0.03	0.03	0.02
Zinc (Zn)	ppm[b]	12	17	17	10	60	6	9	41
Manganese (Mn)	ppm	24	42	17	26	30	10	50	114
Copper (Cu)	ppm	4	3	3	11	14	5	6	5
Iron (Fe)	ppm	101	148	169	1005	1256	34	41	999
Cobalt (Co)	ppm	< 1	< 1	14	< 1	12	< 1	1	1
Aluminum (Al)	ppm	42	21	32	1009	778	138	18	703
Molybdenum (Mo)	ppm	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Silicon (Si)[c]	ppm	402	600	519	126	988	41	29	429
Knife weight loss (knife wear)	mg	0.3	0.7	1.7	4.1	3.9	1.2	0.8	--

[a] Test procedures were run per the Association of Official Analytical Chemists (AOAC) and in part with an inductive plasma coupling (ICP) instrument. (The elemental data are not analyzed to add up to 100%, and not all test procedures are perfect for fillers.)

[b] ppm = mg/kg.

[c] The silicon values are organic-type silicon and do not reflect inorganic silicon values.

Table 4. — Initial mix viscosity and mix working life of both filler types.

	Furfural residues	
	Asian source	Oat hull
Resin percent prior to NaOH dispersion: Mix viscosity, (Brookfield, Model RV, mPa·s at 25 to 29°C)	26	21
Initial	6400	2700
After 1 hr.	6880	2980
After 4 hr.	7600	4460
After 24 hr.	10,500	7380

Table 5. — Bondline performance per PS 1-95 of southern pine plywood glued with PF resin-adhesives containing five different filler types.

	Vacuum-pressure test		Two 4-hr. boil test	
Plywood by filler type	Strength	Wood failure	Strength	Wood failure
	(kPa)	(%)	(kPa)	(%)
65 percent E. walnut shell / 35 percent oat hull furfural residue	519	96	533	59
Oat hull furfural residue				
Industrial panels	865	81	546	69
Laboratory panels	1065	95	765	92
Asian furfural residue				
Industrial panels	790	94	727	89
Laboratory panels	799	95	706	91
E. walnut shell flour	838	94	504	96
Pecan shell flour	658	79	656	82

Adhesive bond assessment

The product standard PS 1-95 requires an 80 percent wood failure average for interior-type plywood bonded with exterior PF adhesives. The industrial plywoods in this study generally met these requirements when tested via the vacuum-pressure test, but the presence of dryout bondlines occurred in two types, resulting in low test values (59% and 69%) when tested via the two 4-hour boil test (**Table 5**). These two low bondline test results were judged (observations of veneer and bondlines) to be due to manufacturing variables and not related to the adhesive. This conclusion was reinforced in part because laboratory-glued plywood with one of the fillers in question yielded excellent bond properties (90%+ wood failure).

Conclusion

Knife wear tests are an important "tool" in deciding the quality of additives for plywood adhesives.

Tool wear when cutting construction and industrial plywood of southern pine bonded with PF adhesive formulations is often due to the filler quality, including filler elemental content variations.

Factors important to quantify filler quality for tool wear are ash (high metallic and silicon values) and pH (S, Cl, and other corrosive elemental agents).

Literature cited

Mississippi State University Forest Products Lab. (MSFPL). 1987. CR 49: Knife wear test for plywood gluelines. MSFPL Standard Analytical Test Procedures. MSFPL, Mississippi State, MS.

Sellers, Jr., T. 1985. Chapter 20: Fillers. In: Plywood and Adhesive Technology. Marcel Dekker, New York. pp. 471-493.

__________. 1989. Knife wear due to filler type in plywood adhesives. Forest Prod. J. 39(4):39-41.

__________. 1994. Technological and market trends of fillers used in wood adhesives. Panel World 35(3):22-26.

__________. 1997. Cutting tools wear: corrosion, adhesion, abrasion. Panel World 38(3):60-61.

__________ and D.J. Gardner. 1989. Variables affecting the suitability of filler raw materials for adhesives used to bond structural plywood. Forest Prod. J. 39(3):34-38.

__________, G.D. Miller, and W.L-S. Nieh. 1990. Evaluation of three fillers in PF adhesives used to bond intermediate moisture content plywood: Glueline durability and knife wear. Forest Prod. J. 40(10):23-28.

Stevens, R.R. 1974. Apparatus to facilitate comparison of abrasiveness of wood panel products. Forest Prod. J. 24(8):37-39.

Stewart, H.A. 1985. A turning method for monitoring tool wear when machining reconstituted wood products. Forest Prod. J. 35(11/12):41-42.

__________. 1992. High-temperature halogenation of tungsten carbide-cobalt tool material when machining MDF. Forest Prod. J. 42(10):27-31.

Stone, J.B. and P. Robitschek. 1978. Factors affecting the performance of plywood glue extenders. Forest Prod. J. 28(6):32-35.

U.S. Department of Commerce (USDC). 1995. Voluntary product standard: PS 1-95 Construction and Industrial Plywood. National Institute of Standards and Technology, U.S. Dept. of Commerce, Washington, DC. 40 pp.