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Spent Tea Leaves as Extender and Scavenger for Urea Formaldehyde-Bonded Plywood

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In lieu of wheat flour, spent tea leaves (STL) from the manufacture of tea-flavored drink was mixed with urea formaldehyde (UF) resin to produce an adhesive for 5-mm-thick plywood. Three glue mixes (GM1, GM2, and GM3) were formulated using three STL levels (3.4%, 6.8%, 10.2% by mass). GM1 had no catalyst and coconut shell flour/filler; GM2 had no catalyst but had a filler; while GM3 had a catalyst but no filler. The glue manufacturer's recommended formulation was used for making the control plywood. All glue mixes were formulated with the same total resin solids. The effect of the STL on the various glue mixes was evaluated in terms of the plywood's shear strength & wood failure (PNS ISO 12466-1:2016 & PNS ISO 12466-2:2016), and formaldehyde emission (PNS ISO 12460-4:2016). Results indicated that GM3, or complete replacement of wheat flour and coconut shell flour with STL both as extender and filler in the plywood glue mix, was the best formulation. It did not adversely affect the panel's strength properties. Moreover, formaldehyde emission was significantly reduced by 36 to 60%.

Key words: extender, formaldehyde emission, plywood, shear strength, spent tea leaves

INTRODUCTION

In the late-1970s, substituting tree foliage to wheat flour as extender in plywood and particleboard glue formulations was reported to obtain similar or even enhanced results compared to completely using wheat flour. This was attributed to the protein present in the tree foliage (Chow 1978). There was no mention of the formaldehyde scavenging activity of the tree foliage then. Formaldehyde was much later classified as carcinogenic to humans in a meeting in Lyon, France on 2-9 Jun 2004 by the International Agency for Research on Cancer – World Health Organization (IARC –WHO 2006).

In the quest for means to reduce formaldehyde emission (FE) from wood-based panels bonded with formaldehyde-based resins, various plant parts rich in polyphenolic compounds like tannin and protein have been studied. Found to significantly reduce FE from particleboard were mimosa bark extracts (Nemli et al. 2004), spent tea leaves (STL) (Shi et al. 2006), stone pine (*Pinus pinea* L.) cones (Buyuksari et al. 2010), and walnut/almond shells (Pirayesh et al. 2013). For plywood, Chan (1996) reported a significant FE reduction using giant ipil-ipil [*Leucaena leucocephala* (Lam.) de Wit] seed flour as glue extender.

In the Philippines, production of tea-flavored drinks using *Camellia sinensis* (L.) Kuntze by one company generates about 18 metric tons of STL per day (wet basis) or about 3.8 metric tons per day (dry basis). Presently without any large-scale uses, these tons of STL are dumped as waste or given away for composting or research. STL is generated after simple hot water extraction and is expected to retain much of its fiber, protein, phenolic compounds, and other components, which indicates its potential as extender for formaldehyde-based glues and as a possible formaldehyde

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scavenger. To prove the material's potential in plywood manufacture, glue formulations with varied proportions of the STL were used in the production of three-ply panels. The objective of the study was to evaluate the STL as a glue extender to completely replace wheat flour and to serve as formaldehyde scavenger. This was made in the absence or presence of other components i.e., catalyst and filler (coconut shell flour), to observe possible synergism among them. The performance of the glue mixes with varying amounts of STL was tested based on the shear strength, wood failure, and formaldehyde emission of the panels.

MATERIALS AND METHODS

Collection, Chemical Analysis, and Preparation of Spent Tea Leaves

Freshly generated STL was collected from the manufacturing plant of Universal Robina Corp. in Cabuyao, Laguna. The STL came from a discharge bin at the end of the production line of tea-flavored drinks. The STL was immediately sun-dried to atmospheric moisture content. Further drying was made in the oven at 50°C to about 10% moisture content (MC) then ground in the Wiley mill. Particle fraction passing the 100-mesh screen was used for the glue mixes. The pH was determined by soaking 25 g of STL in a beaker with 80 mL of distilled water. After 20 min, the pH of the solution was read using a pH meter.

A portion from the dried STL was analyzed for crude protein, crude fiber, and ash contents as follows:

Crude Protein. Semi-micro Kjeldahl method (AOAC 2005b) determines total nitrogen and the amount of protein is calculated by a protein factor (general factor = 6.25). The STL sample was digested with sulfuric acid to convert organic nitrogen to ammonium ions. Ammonia was liberated by addition of alkali in the digested sample and distilled into a solution of boric acid. The distillate was then titrated with hydrochloric acid to determine the amount of H_3BO_3, which is equivalent to the amount of NH_3 present in the sample.

Crude Fiber. In the Weende method (AOAC 2005c), cellulose, lignin, pentosans, and a little nitrogenous matter comprise the crude fiber in an organic sample. The STL sample was hydrolyzed with boiling sulfuric acid, followed by boiling in alkali to dissolve remaining minerals and saponify lipids in the sample. The residue was filtered, dried, weighed then ignited at 500°C, cooled and weighed again. The loss in weight is the crude fiber.

Ash. In the gravimetric method (AOAC 2005a), ash serves as an indicator of inorganic matter in a sample. The STL sample was placed in a porcelain crucible and ignited to 550°C for 2 h, cooled, and then weighed. It was then reignited for another 30 min and re-weighed until there was no more loss in weight.

Formulation of Glue Mixes

Commercial urea formaldehyde resin (Ures® R21-030, 50% resin solids, viscosity 110-300 cP, specific gravity 1.20, pH 7.6, and 1.1% free formaldehyde) were obtained from RI Chemical Corporation, Pasig City. Initial glue mix formulation of the control as per the glue manufacturer's suggested formulation of up to 1.35% by mass catalyst (NH_4Cl) resulted to a very high viscosity above 1500 cP after 30 min stand of the glue-mix. Hence, new formulations of the control with only 0.34% by mass of catalyst were used in preliminary formulations. Based on the viscosities of the preliminary glue-mix formulations, the final glue-mix formulations were decided (Table 1).

Production of Three-ply Panels

Rotary-cut *Falcataria moluccana* (Miq.) Barneby & J.W. Grimes and *Shorea sp.* veneers were obtained from Mt. Banahaw Wood Industries, Inc. (MBWI), a plywood manufacturing plant in Sariaya, Quezon. These two species are commonly used for plywood production in

Table 1. Various glue-mix formulations using spent tea leaves as replacement for wheat flour (control) in % by mass.

Components		**GM1 (Zero catalyst & CSF)**			**GM2 (Zero catalyst with CSF)**			**GM3 (With catalyst zero CSF)**		
	Control	STL-a	STL-b	STL-c	STL-a	STL-b	STL-c	STL-a	STL-b	STL-c
UF resin (50% solids)	68.03	68.03	68.03	68.03	68.03	68.03	68.03	68.03	68.03	68.03
Catalyst (NH_4Cl)	0.34	0.00	0.00	0.00	0.00	0.00	0.00	0.34	0.34	0.34
Wheat flour (WF)	13.61	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Coco shell flour (CSF)	3.40	0.00	0.00	0.00	3.40	3.40	3.40	0.00	0.00	0.00
Spent tea leaves (STL)	0	3.40	6.80	10.20	3.40	6.80	10.20	3.40	6.80	10.20
Water	14.63	28.57	25.17	21.77	25.17	21.77	18.37	28.23	24.83	21.43
Glue mix total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Glue mix resin content (%)	34	34	34	34	34	34	34	34	34	34

the Philippines (Jimenez et al. 2015). MBWI does not produce its own veneers but simply buys them either from Mindanao or abroad. Outer ply veneers were made of *Shorea sp.*, while core veneers were from *F. moluccana* – both were defect-free and straight-grained. Veneers were cut to dimensions of 60 cm x 60 cm. Outer ply veneers were 0.5 mm thick, while core veneers measured 3.8 mm thick. These were dried to about 7% MC using an oven dryer. Using the prepared glue mixes at 200 g/m^2 glue spread and 15 min total assembly time, three-ply panels were produced under specific pressure of 10 kg/cm^2, pressing temperature of 115°C for 2 min. Four replicate panels were made for each treatment.

Properties Testing

After two-week conditioning of the boards, specimens for shear strength/wood failure (bonding quality) tests were prepared and evaluated following PNS ISO 12466-1: 2016 procedures. Shear test specimens (25 mm x 83 mm) grooved one third of its length from the end were cut from each panel. These were soaked in water maintained at 23°C for 24 h. After soaking, excess water was allowed to drain for 15 min, then the specimens were towel-dried and tested while wet. The tension shear strength of each specimen was tested using a 5 kN Universal testing machine (AGS-X, Shimadzu, Japan) at crosshead speed of 5 mm/min. Load was continuously recorded using data acquisition software (Shimadzu Trapezium X version 1.4.2 software) running in a personal computer. Percentage of apparent cohesive wood failure on broken shear specimens was estimated following PNS ISO 12466-1.

Formaldehyde Emission

Specimens were prepared and quantity of formaldehyde emitted was determined according to the 24-h desiccator method specified by PNS ISO 12460-4:2016. The procedure is a simple and an inexpensive way of measuring FE suited for quality testing of experimental plywood or composite panels. Ten specimens (50 mm x 150 mm) per panel were cut at the middle portion. These were secured in wire holders and allowed to be conditioned to constant mass in a room at 21°C and 65% relative humidity for seven days. Distilled water (300 ml) in glass dish was placed at the bottom of each 11 L glass desiccator. The wire holder with the specimens were placed above the glass dish. The set up was maintained at 20°C for 24 h to allow formaldehyde released from the boards to get absorbed in the water. The formaldehyde solution contained in the glass dish was treated with acetylacetone-ammonium acetate solution prior to the determination of formaldehyde using a UV spectrophotometer (UV-1700, Shimadzu, Japan). Three replicate desiccators were used for each glue-mix formulation. Desiccators containing no specimens were used as control.

Evaluation of Data using SAS® Analytics Pro

Three by three factorial experiment in completely randomized design was used to analyze the variation contributed by the three levels of glue-mix (GM1, GM2, GM3) and three levels of STL (a, b, c for 3.4%, 6.8%, and 10.2% by mass, respectively) on the bond properties (shear strength and wood failure) and FE of plywood. The nine treatment combinations were also compared against the control. Means of tested properties were separated using Tukey's Honest Significance Difference (HSD) test (α=0.05).

RESULTS AND DISCUSSION

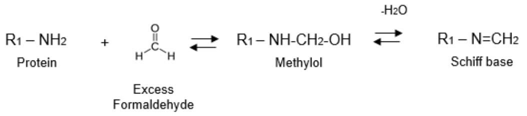
Chemical Composition of STL

Analysis of the air-dried samples was limited to crude protein, crude fiber, ash, and pH, the results of which are shown in Table 2. The chemical composition analyzed were important in characterizing the behavior of the glue-mix, as well as the bond performance of the UF glue mix with added STL. STL's crude protein and fiber were found to be within the values reported for most commercial plywood glue additives (Robertson & Robertson 1977; Sellers et al. 2005). In comparison, wheat flour is reported with 11.0% protein, 0.5% crude fiber, and 0.7% ash (Chan & Dionglay 1996). STL's ash content (3.85%) was lower than most of the commercial fillers i.e., oat hull (8.0-10.8%) and Asian furfural (4.9-15.0%) residue used to bond Southern pine plywood in America (Sellers et al. 2005). Previous work of Sellers and co-authors (2005) showed that the high amount of ash in plywood contributes to knife wear.

Table 2. Chemical composition and pH of spent tea leaves.

Parameter	Method	Content, g/100g
Crude Protein	Semi-micro Kjeldahl - AOAC	20.84
Crude Fiber	Weendee – AOAC	20.62
Ash	Gravimetric – AOAC	3.85
pH	pH meter	5.8

A comparison of the chemical composition before and after processing could not be made as samples of tea leaves before processing were not provided. Duke (1983) presents varying data as follows: fresh *C. sinensis* leaves contain 17.2% protein, 27.0 % crude fiber, and 5.6 % ash; and per 100 g, protein ranges from 24.1 to 28.3 g, fiber 8.7-9.7 g, and ash 5.3-5.9 g. *C. sinensis* is grown in several countries. The varying data – particularly on crude fiber – may be due to the differences in the location's soil and climatic conditions, as well as possible differences in methods of analysis. Nonetheless, it is noted that the protein content of STL (20.84 g/100 g) is still relatively high compared to that of fresh tea leaves (24.1-28.3 g/100 g) (Duke 1983). It is this protein content that is of particular interest in this study as indicator of the STL's potential as both glue extender and formaldehyde scavenger. According to Evangelista (2012), proteins can enhance a strong linkage with the adhesive, thus increasing bond strength. On the other hand, Pervaiz and Sain (2011) showed that protein from secondary sludge of a kraft paper mill can be utilized as a wood adhesive. Further, protein can scavenge excess formaldehyde from UF (Chan 1996). The basic chemical reaction for this is illustrated below (Hoffman et al. 2015):



(1)

It is also notable that the pH of STL (5.8) is mildly acidic and therefore expected to also act as catalyst to UF resin (pH 8). This was considered in the glue formulations (GM1 and GM2) where the catalyst for the UF resin was removed.

Formulation of Glue Mix with STL

Initial attempt to use the catalyst at the prescribed 1.35% by mass resulted in quick gelling of the glue mix, particularly that with STL. Dramatic increase in viscosity was also observed with an increase in the amount of STL to 13.6% in lieu of wheat flour (WF). Indeed, STL also acts as catalyst as its mildly acidic pH indicates. Thus, only a maximum of 0.34% by mass of catalyst and 3.4-10.2% by mass of STL were used in the glue-mix formulations used in bonding the experimental plywood. Variations without catalyst and or filler were also considered. Total glue mix resin solids were, however, maintained at 34%.

Performance of Plywood Glue Mix with STL

Shear Strength and Wood Failure. Tables 3 and 4 show summaries of the ANOVA on the shear strength and wood failure, respectively, of the plywood bonded with STL-extended UF resin. Both sets of data indicate that neither glue mix composition nor amount of STL and their interaction showed any statistically significant effect on the shear strength and wood failure of the experimental plywood panels. This means there was neither remarkable adverse effect nor improvement on these properties by switching from wheat flour to STL as glue extender, thus indicating that STL is comparable with wheat flour.

Table 3. ANOVA on the shear strength of plywood bonded with STL-extended UF resin.

Source of Variation	*dF*	*SS*	*MS*	*F*	*Pr>F*
Treatments	9	0.1765			
Control vs. Treated	1	0.0151	0.0151	0.6448	0.0895
Among Treated	8	0.1615			
A-Glue mix composition	2	0.0014	0.0007	0.0292	0.1087
B-Spent tea leaves (% by mass)	2	0.0215	0.0108	0.4608	0.1013
A*B	4	0.1385	0.0346	1.4816	0.0856
Error	20	0.4676	0.0234		
Total	29	0.6441			
CV=22.025 R^2=28.22					

Table 4. ANOVA on the wood failure of plywood bonded with STL-extended UF resin.

Source of Variation	*dF*	*SS*	*MS*	*F*	*Pr>F*
Treatments	9	4684.3519			
Control vs. Treated	1	107.0370	107.0370	0.3428	0.0927
Among Treated	8	4577.3148			
A-Glue mix composition	2	565.4321	282.7160	0.9055	0.0938
B-Spent tea leaves (% by mass)	2	1568.0556	784.0278	2.5111	0.0666
A*B	4	2443.8272	610.9568	1.9568	0.0734
Error	20	6244.4444	312.2220		
Total	29	10928.7963			

CV=23.8602 R^2=43.00

Figures 1 and 2 show the treatment means for both shear strength and wood failure, respectively. While the average values for both properties appear to be lower than the control for most of the STL-extended glue mix, the differences are not statistically significant as indicated by their *Pr>F* values in Tables 3 and 4. This implies that complete replacement of wheat flour with STL may be possible.

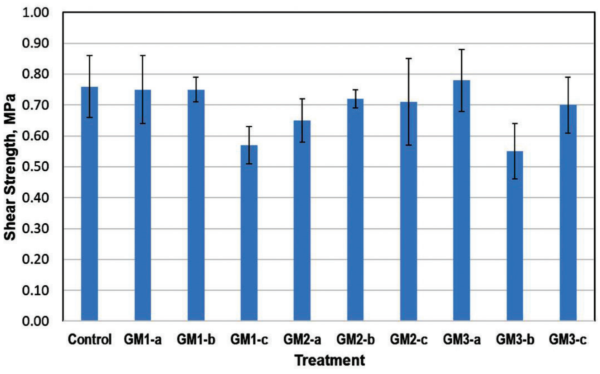


Figure 1. Shear strength of various glue-mix with STL.

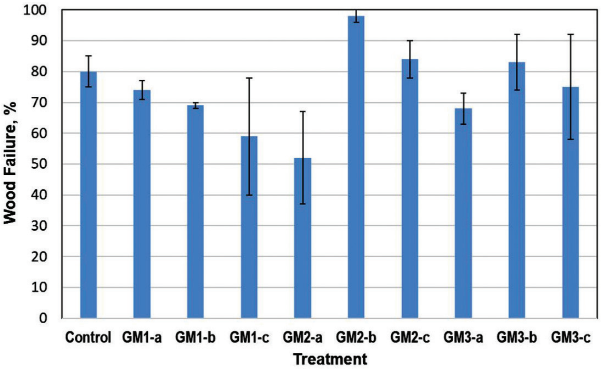


Figure 2. Wood failue of various glue-mix with STL.

The patent by Chow (1978) also claims similar performance by tree foliage powder as plywood glue extender or extender-plus-filler. With UF glue, however, Chow replaced wheat flour up to 70% only. In another development, plywood adhesive extended with freeze-dried protein from corn germ meal demonstrated higher tensile strength than the wheat flour-extended adhesive (Evangelista 2012). Bark flour from walnut, chestnut, fir, and spruce trees were also found suitable to replace wheat flour by up to 10 parts by weight in the UF glue-mix due to its rich polyphenol content (Aydin et al. 2017).

UF-bonded panels are readily categorized under Class 1 for bonding quality i.e., for use in normal interior climates excluding any extended direct exposure to weather (PNS ISO 12466-2:2016). The standard glue line requirements for any class, however, are the same. In this study, the average shear strength (τ) values due to the treatments, ranging 0.55-0.78 MPa, come with apparent cohesive wood failure (WF) ranging 52-98%. The results remarkably surpassed the PNS ISO glue line requirements for τ (MPa) and WF (%) as follows: $\geq$ 80% WF for $0.2\leq\tau<0.4$, $\geq$60% WF for $0.4\leq\tau<0.6$, and $\geq$40% WF for $0.6\leq\tau\leq1.0$.

Formaldehyde Emission. Table 5 shows highly significant effects of glue mix composition, amount of STL, and their interaction on the amount of formaldehyde emitted by the experimental panels.

Tukey's HSD Test on the interaction treatment means (Fig. 3) revealed that the high 13.8 mg/L FE by the control panel was significantly reduced by about 36 to 60% FE by the panel with GM3, or complete replacement of wheat flour and coconut shell flour, but retaining the catalyst. This indicates that in the presence of the catalyst for the adhesive, STL works effectively as an extender and formaldehyde scavenger (attributed to its protein and fiber contents) and as filler (probably due to its ash content).

Table 5. ANOVA on the FE of plywood bonded with STL-extended UF resin.

Source of Variation	*dF*	*SS*	*MS*	*F*	*Pr>F*
Treatments	9	511.2319			
Control vs. Treated	1	64.2652	64.2652	43.8527	<0.0001
Among Treated	8	446.9667			
A-Glue mix composition	2	283.1457	141.5729	96.6053	<0.0001
B-Spent tea leaves (% by mass)	2	46.5458	23.2729	15.8808	<0.0001
A*B	4	117.2752	29.3188	20.0063	<0.0001
Error	20	73.2739	1.4655		
Total	29	584.5057			

CV=11.367 R^2=86.23

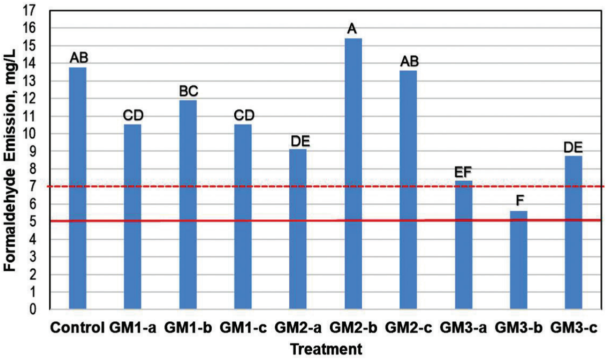


Figure 3. Formaldehyde emission of various glue-mixes with STL. Note: horizontal bar in red indicates the average limit for FE* Performance Class of plywood (PNS 2103:2017). A maximum of 7 mg/L (in dash bar) is also indicated in the standard. Letters above the bars are the means separation – similar letters indicate not significantly different at α=0.05

Without a little catalyst, as in GM1 and GM2, only minimal up to no significant reduction in FE was achieved. This needs further investigation, however, into reaction mechanisms between and among the components, including the quality/wood species (mixed species, in this case) of the veneers used.

In this experiment, however, the initial (control) emission values from the supposedly usual gluing conditions using wheat flour were way above the average limit for FE as provided for by PNS 2103:2017. Results indicate that the use of STL in the presence of the catalyst, as in the GM3 treatment and with further improvement, has better chances of meeting the strict standards for FE in plywood.

Nonetheless, further investigation is needed to better understand the rheological and other gluing properties of STL to improve conditions with its use. Moreover, since STL imparts its green color in the glue mix (though glueline is colorless when hardened), it would be of further advantage to try it also with phenolic resins, as Chow (1978) did with tree foliage. In this case, the pH of STL may need to be adjusted to adapt for curing with phenol resins.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of this study, it is found that complete replacement of wheat flour with STL as extender in UF glue-mix for plywood does not adversely affect the strength properties of the resulting plywood panel. Results also indicate that STL not only works as an extender but also as filler and formaldehyde scavenger. Investigations into reaction mechanisms involved including the rheological and other gluing properties of STL are recommended. Similar investigations using STL with phenol formaldehyde resin are also recommended.

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