Evaluation of Forestry Residue-Source Oil-Tea Cake as an Extender for Phenol-Formaldehyde Plywood Adhesive

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Abstract

Forestry residue-source oil-tea cake (consisting of plant protein, tannin, carbohydrate compounds, and tea saponin) is a major by-product from the pressing process of Camellia (*Camellia oleifera* Abel) seeds. In this study, we evaluated oil-tea cake as a potential extender to replace the current industry extender, wheat flour, for applications with phenol-formaldehyde (PF) plywood adhesives. We evaluated mixing properties, curing characteristics, and adhesion performance of the adhesive containing oil-tea cake as compared with those of the standard wheat flour–based adhesive. The oil-tea cake–based adhesive showed excellent mixing properties and working life, and its viscosity met the recommended value for the sprayline coater. The adhesive containing oil-tea cake was comparable in wet shear strength and wood failure values of plywood to the wheat flour–based adhesive. Thermal analysis showed that adhesives containing oil-tea cake had significant curing reactions because of the relatively large curing enthalpy (ΔH) value of oil-tea cake. These results indicate that oil-tea cake is suitable as a low-cost and effective plywood adhesive extender. Therefore, using oil-tea cake is economically beneficial because it not only makes use of what would otherwise be forestry residue, but it is cheaper to use as a plywood adhesive than wheat flour.

Phenol-formaldehyde (PF) thermosetting resins are used extensively as exterior-grade adhesives for the manufacture of fiberboard and oriented strand board, especially for structural plywood bonding (Pizzi 1994). For practical production of wood panels, extenders are added to the adhesive formulations to improve its workability. One of the functions of extenders is to reduce the resin cost, since they replace resin solids without reducing total solid content. Furthermore, they promote bonding by holding the adhesive on the veneer surface (Sellers 1985). They can also increase viscosity, limit penetration of the resin into porous substrates, and improve adhesive performance. Extenders in adhesives are defined as materials having some adhesive function. Most extenders added to PF resin in plywood manufacture are proteinaceous and amylaceous materials, such as soy meal, wheat flour, furfural residue, because they can enter into the matrix of the binders (Gardner et al. 1990, Oh and Sellers 1999, Sellers et al. 2005, Hojilla-Evangelista 2010). Cereal grain–derived (CDA) extenders have been used as adhesive additives for many years to partially replace liquid PF adhesives for plywood production (Johnson and Kamke 1994). CDA-type extenders in adhesives can therefore reduce the plywood resin consumption and enhance panel properties, thereby reducing production costs. Stone and Robitschek (1978) discussed how furfural extenders principally affect the performance of adhesives, as applied in PF resin formulations. More recently, Hojilla-Evangelista (2010) presented results of a study evaluating soybean meal as a protein extender in plywood adhesives intended for

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sprayline coaters. The results indicated that soybean meal is a viable extender in plywood glue. Given the cost of the extenders, agricultural or forestry residues are typically used as low-cost and abundant extenders for the plywood adhesive. Fermentation residues obtained by anaerobic cellulolytic bacteria can serve as a coadhesive for plywood PF resin because of their components of protein and glycocalyces (Weimer et al. 2005). Geng et al. (2007) showed that paper mill sludge (secondary sludge) could serve as an effective extender for adhesive formulations. Methyl glucosides (MeG), which are a by-product of corn syrup production, were also found to be good extenders that belong to carbohydrate-based materials (Sellers and Bomball 1990). Commercial MeG adhesive extenders have been successfully applied to partially replace plywood adhesives such as PF resin and urea-formaldehyde (UF) resin. In addition, recycled micronized polyurethane powders can also be used as active extenders of UF and PF wood panel adhesives (Geng et al. 2007). In China, wheat flour is most often used as a wood adhesive extender designed for construction and industrial plywood panels, and its consumption per year is continually increasing. Wheat flour in adhesives serves not only as an extender, but also as a good filler. In general, wheat flour is added to synthetic resin adhesives in ratios of 15 to 30 parts of extender to 100 parts of resin solution. However, the output of wheat flour in China is not sufficient to meet the consumption requirement in the food industry, so large quantities of wheat flour need to be imported from other countries to meet the requirement every year. This limits wheat flour as a commonly used extender in the application of plywood adhesive.

Oil-tea cake is the main by-product produced by extracting tea oil from Camellia (*Camellia oleifera* Abel, Theaceae) seeds during the processing of cooking oil. It is estimated that more than 400 million kg of oil-tea cake was generated in 2009 from the tea oil industry (You 2008). Most oil-tea cake is either burned in power boilers, where it contributes to the energy demands of the industry, or is directly discarded as forestry residue. The oil-tea cake is mainly composed of plant protein, tannin, carbohydrate compounds, and tea saponin, and thus it is a potential protein resource (Qi and Peng 1989). This forestry residue-source oil-tea cake thus has the potential to display extender features.

The objective of this study was to find a new use for the forestry residue-source oil-tea cake that is usually disposed of, with the goal of increasing its economic value. We explored the potential of completely or partially substituting wheat flour with oil-tea cake as an extender for PF plywood adhesives. In order to examine the efficacy of oil-tea cake as an extender, we investigated the mixing properties, curing characteristics, and adhesion performance of the adhesive containing oil-tea cake and compared these properties with those of the standard wheat flour–based adhesive.

Materials and Experimental Methods

Materials

Phenol, formaldehyde aqueous solution (37%), and sodium hydroxide were supplied by Beijing Chemical Industries (Beijing, China) and were used directly as raw materials without further purification. We purchased a commercial PF resin with code number P961 from Dynea (Beijing) Co., Ltd. Wheat flour was purchased from Beijing Guchuan Food Co., Ltd. (Beijing, China) with the following nonstarch contents: 13.4 percent water, 9.3 percent proteins, and 0.45 percent ash (wt/wt of flour mass). Oil-tea cake supplied from the Research Institute of Subtropical Forestry (Fuyang, China) was composed of 16 percent plant protein, 8 percent tannin, 41 percent carbohydrate compounds, 13 percent plant cellulose, 10 percent tea saponin, and 12 percent other compounds including ash, water, etc. (wt/wt of total oil-tea cake).

Laboratory PF resin preparation

A laboratory PF resin with a F:P:NaOH molar ratio of 2.0:1:0.7 was prepared following the procedure in Fan et al. (2009b). The calculated amount of phenol, sodium hydroxide solution, and the first portion of formaldehyde (F1) were charged in a three-neck reactor equipped with a condenser according to the formula in Table 1. An appropriate amount of distilled water was also added to the reaction system. After that, the reaction temperature was gradually raised to reflux (90°C) within 40 minutes and maintained at this temperature for 30 minutes. The second portion of formaldehyde (F2) was then added to the reactor, and the temperature was kept at 85°C until the viscosity (measured at 20°C) reached between 400 and 500 mPa·s.

Measurement of PF resin properties

The nonvolatile solid content was measured by heating about 4 g of sample in a small aluminum pan in an oven at 120°C for 2 hours. The final free formaldehyde content in the PF resin was determined by the hydroxylamine method (Manfredi et al. 1999). Gel times were measured at 145°C according to the method in Fan et al. (2009a). Viscosity and pH values were respectively obtained with a rotational viscometer (NDJ-5S, Shanghai Changyi Gealogical Instruments Co., Ltd., China) and pH meter (PHS-3B, Shanghai Precision & Scientific Instrument Co., Ltd., China) at 20°C. Each value of the above property is an average of five test results. These properties of commercial and laboratory PF resins are shown in Table 2.

Extender and adhesives preparation

The oil-tea cake was used as an alternative adhesive extender to completely or partially substitute for wheat flour (the control extender). The oil-tea cake was dried to approximately 8 percent moisture content and then ground with a laboratory Wiley mill into a 100-μm (150-mesh) powder. In this work, wheat flour, oil-tea cake, and different weight ratios of oil-tea cake/wheat flour (20/80, 40/60, 60/40, and 80/20) were used as PF adhesive extenders.

Commercial and laboratory PF resins were used for adhesive formulations and were prepared following identical procedures. First, 18 kg of extender was mixed with 15

Table 1.—Synthesis of laboratory phenol-formaldehyde resins.

Ingredient	Amount (kg)
Phenol	94
Formaldehyde (37%)	
F1	122
F2	41
Sodium hydroxide (40%)	70

Table 2.—Properties of phenol-formaldehyde (PF) resins.

Property	Laboratory PF resin	Commercial PF resin
Nonvolatile solids (%)	43.4	43.1
pH	11.8	12.1
Viscosity (mPa·s)	462	435
Free formaldehyde (%)	0.16	0.11
Gel time (min)	14.2	13.7

kg of water with a rapid stirring mixer for 10 minutes, and the pH value of the mixture was adjusted to 10 with 30 percent sodium hydroxide solution. Then, 100 kg of PF resin was gradually added to the rapidly stirring mixture at an average rate of 20 kg/min and stirred for about 10 minutes. After that, appropriate amounts of distilled water and sodium hydroxide solution were added to adjust the solid content and the pH of adhesive to 43 percent and the same value as original resin, respectively. Each adhesive was measured for viscosity stability after a 24-hour period.

Differential scanning calorimetry

The differential scanning calorimetry (DSC) analysis was made using a DSC-60/60A (Shimadzu, Japan) instrument and TA60 thermal analysis software. About 5 to 6 mg of adhesive sample was weighed and then sealed in a high-pressure stainless steel crucible capable of resisting up to 5 MPa pressure. Dynamic scans were carried out from 30°C to 300°C at a heating rate of 10°C/min. Both the temperature and enthalpy calibrations were conducted with indium. Laboratory PF adhesives with wheat flour, 60/40 oil-tea cake/wheat flour weight ratio, and oil-tea cake were characterized. All experiments were done in duplicate, and the average values were reported.

Preparation and test of plywood

Aspen veneers with dimensions of 1,220 by 2,440 by 1.6-mm thickness were used for this study. Three-layer laboratory plywood panels were bonded using the prepared PF resin adhesives. The adhesive was applied to both faces of the veneer using a sprayline coater. The adhesive-coated veneer was stacked between two uncoated veneers with the grain directions of two adjacent veneers perpendicular to each other. The plywood panels were prepared under the following conditions: moisture content of veneer, 8 to 10 percent; glue spread, 340 to 360 g/m^2 (double line); cold-press under 1.0 MPa for 20 minutes; hot-press temperature, 145°C; hot-press pressure, 1.0 MPa; hot-press time, 5 min.

The bond performance testing of the plywood panels was conducted in accordance with US Voluntary Product Standard PS1-95 for Construction and Industrial Plywood. The wet shear strengths of plywood bonded with PF adhesives were measured with a tension testing machine (Jinan Shijin Co. Ltd., China) under a crosshead speed of 5 mm/min.

Results and Discussion

Adhesive stability

The chemical components of the oil-tea cake and wheat flour described above showed that the oil-tea cake had more plant protein and more complex chemical components such as tannin, tea saponin, carbohydrate compounds, etc., that were not present in the wheat flour. The plant protein, tannin, and carbohydrate compounds were able to serve as an extender since they can enter into the adhesive matrix during curing due to their high reactivity with PF resin. The PF adhesives with different extenders blended well but required some amounts of distilled water for dispersing the extenders prior to adding to PF resin. From the appearance of the adhesive mixtures, it was observed that the oil-tea cake and oil-tea cake/wheat flour were uniformly distributed in the PF resin, and the resulting adhesives had a similar flow behavior to the standard wheat flour–based adhesive. This was supported by the fact that they had lower viscosity values than wheat flour–based adhesive, both in the initial mix and after sitting overnight (Table 3). However, the adhesive containing oil-tea cake presented a much darker color than the one with wheat flour because of the inherent dark brown color of the oil-tea cake. In general, a highly mobile adhesive may easily penetrate deep into wood through the porous surface, so that little adhesive is left on the surface to adhere to the wood blocks. On the other hand, if highly viscous adhesives are applied to the wood substrate, excessive amounts of resin are left at the surface with little penetration into the pores. Thus, the effects of both mechanical interlocking and efficient interfacial contact between the adhesive and wood are decreased due to highly viscous resins, leading to a much weaker bonding strength. The recommended viscosity of PF adhesive is around 1,000 to 2,000 cp for sprayline coaters.

As Table 3 shows, both adhesives with oil-tea cake and oil-tea cake/wheat flour had satisfactory initial mix viscosity and a working life that met the required viscosity for sprayline coaters, as compared with the wheat flour–based adhesive. For commercial and laboratory PF adhesives, wheat flour resulted in a marked increase in the viscosity of the adhesive because of its higher absorption capacity; the oil-tea cake with lower absorption capacity had a minor viscosity enhancement effect. The viscosity enhancement's influence on the adhesive increased gradually as the proportion of wheat flour in oil-tea cake/wheat flour increased. When the oil-tea cake/wheat flour weight ratio was increased to 20/80, its adhesive viscosity showed a similar value to that of the adhesive with wheat flour. The viscosities of the adhesives increased significantly from 991 to 1,063 cp to 2,173 to 2,565 cp for commercial PF resin and from 962 to 1,034 cp to 1,989 to 2,417 cp for laboratory PF resin, respectively, after 24 hours of sitting at room temperature. It was also noted that for the same extender used, the laboratory PF adhesive had a much higher initial

Table 3.—Viscosity changes of phenol-formaldehyde (PF) adhesives with different extenders (initial and after 24 h).

	Viscosity change (mPa·s) at 20°C			
	Commercial PF resin		Laboratory PF resin	
Filler type	Initial	After 24 h	Initial	After 24 h
None	462	446	435	465
Wheat flour	1,063	2,417	1,034	2,565
Oil-tea cake/wheat flour				
20/80	1,042	2,378	1,018	2,481
40/60	1,026	2,236	997	2,317
60/40	1,014	2,182	983	2,253
80/20	998	2,073	975	2,197
Oil-tea cake	991	1,989	962	2,173

viscosity and higher increasing viscosity value after 24 hours than the commercial PF adhesive. This indicated that, compared with the commercial PF adhesive, the extenders used in this study had a more viscosity-reinforcing effect on the laboratory PF adhesive, regardless of initial viscosity value or after 24 hours. Although the 24-hour viscosity of oil-tea cake–based adhesive was more than twice its initial viscosity, it still had reasonable viscosity (1,000 to 2,000 cp), which thus makes it applicable as a new extender in plywood adhesive for sprayline coaters.

Curing characteristics

The characteristic temperature range of the DSC curves gives valuable information on the curing reaction of resin adhesives. In this study, laboratory PF adhesives with oil-tea cake, with an oil-tea cake/wheat flour weight ratio of 60/40, and with wheat flour were used as examples for DSC analysis in order to discuss the effects of oil-tea cake as an extender on the curing characteristics of adhesives. Figure 1 shows the DSC curves obtained using a heating rate of 10°C/min. The cure characteristics of each adhesive are summarized in Table 4 in terms of characteristic curing temperatures (onset temperature, peak temperature, and endset temperature) and curing enthalpy (ΔH). The DSC curve of the wheat flour–based adhesive showed an exothermic peak with one broad signal at the peak temperature of around 150°C, which was similar to the curing peak of the pure PF resin in previous research (Fan et al. 2010). Moubarik's reports indicated that there was no reaction at all between cornstarch and PF resins and that cornstarch seemed to function just as an extender (Moubarik et al. 2009). Thus, this peak was associated with the condensation of methylol groups and phenolic units or wheat flour protein. Both adhesives with oil-tea cake and oil-tea cake/wheat flour presented two well-separated exothermic peaks at 148°C to 151°C and 188°C to 200°C, respectively. The plant protein component of oil-tea cake was easily dissolved in the alkaline water solution and was able to react with active groups of PF resin. Thus, the first peak was similar to that of the wheat flour–based adhesive. Previous research reported that tannin can incorporate into PF resin to form co-condensed resins due to its high reactivity (Pizzi and Scharfetter 1978, Vázquez et al. 2005). However, it needs a higher curing temperature and it has

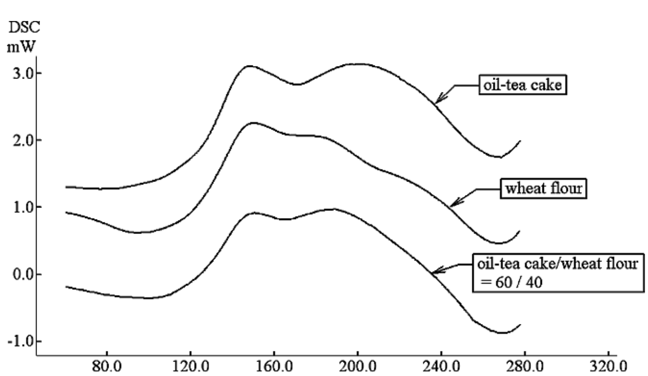

Figure 1.—Differential scanning calorimetry curves of laboratory phenol-formaldehyde adhesives with three different extenders: wheat flour; oil-tea cake/wheat flour, 60/40; oil-tea cake.

Table 4.—Characteristic curing temperature and curing enthalpies of laboratory phenol-formaldehyde adhesives with three different extenders.

Filler type	Temperature (°C)			Enthalpy (J/g)
	Onset	Peak	Endset	
Wheat flour	119.4	150.3	165.6	26.9
Oil-tea cake/wheat flour = 60/40				
Peak 1	121.4	150.6	163.1	41.6
Peak 2	187.5	188.8	258.4	
Oil-tea cake				
Peak 1	118.6	148.7	166.4	58.3
Peak 2	171.3	199.6	257.9	

higher activation energy values. Therefore, taking into account that the wheat flour–based adhesive had only one exothermic peak, the second peak at 188°C to 200°C in the curve of both adhesives with oil-tea cake and oil-tea cake/wheat flour was possibly caused by the reaction of laboratory PF resin units with active compounds of oil-tea cake, such as tannin and tea saponin.

The ΔH values of the adhesives in Table 4 are based on the mass of liquid resin. As a result of more reactive components of oil-tea cake with PF resin units, the ΔH value generated during the curing of the adhesive with oil-tea cake was much higher than those of wheat flour and oil-tea cake/wheat flour. This indicated that the PF adhesive reacted more with the oil-tea cake than with the other extenders.

Adhesion performance

The adhesion performance of adhesives with different extenders was evaluated according to US Voluntary Product Standard PS1-95. The wet shear strength values of plywood panels bonded with adhesives are shown in Table 5. The wet shear strength of the adhesive containing oil-tea cake was slightly lower than that of the wheat flour adhesive. This result appeared to be inconsistent with our hypothesis that the components of oil-tea cake, such as protein, tannin, and tea saponin, have the ability to react with PF resin, thus resulting in an improvement in bond strength. This was also not in agreement with our hypothesis that the larger ΔH value of the adhesive would generate higher bonding strength. These results have the following probable causes. (1) Although the oil-tea cake powder was small enough to disperse uniformly in the adhesive, we also detected the

Table 5.—Wet shear strength values of phenol-formaldehyde (PF) adhesives with different extenders.

Filler type	Plywood wet shear strength, mean ± SD (kgf/cm^2)[a]	
	Commercial PF resin	Laboratory PF resin
Wheat flour	1.71 ± 0.12	1.68 ± 0.16
Oil-tea cake/wheat flour		
20/80	1.69 ± 0.10	1.67 ± 0.14
40/60	1.67 ± 0.16	1.63 ± 0.10
60/40	1.63 ± 0.12	1.58 ± 0.12
80/20	1.61 ± 0.08	1.56 ± 0.17
Oil-tea cake	1.60 ± 0.14	1.55 ± 0.19

[a] Fifteen replicates of each resin.

presence of some particulates of oil-tea cake in the adhesive, thus leading to more pores in cured adhesive and further decreasing the wet shear strength of plywood. (2) Some compounds of oil-tea cake, such as tannin and tea saponin, might not react fully with the active groups of PF resin under the hot-pressing temperature of 145°C, which was much lower than its actual curing temperature (188°C to 200°C) shown in Table 4. (3) Amylaceous, the main component of wheat flour, had better adhesion properties when compared with some unreacted compounds of oil-tea cake such as tea saponin, carbohydrates, plant cellulose, etc.

It is noted in Table 5 that the addition of wheat flour to the oil-tea cake did lead to a gradual increase in adhesive shear strength, and the wet shear strength was almost equal to that of the wheat flour–based adhesive when the wheat flour/oil-tea cake weight ratio increased to 80/20. The commercial PF adhesives gave slightly higher wet shear strength than the laboratory resins after the boiling water test. Generally speaking, the more oil-tea cake that substitutes for wheat flour in the adhesive, the lower the cost of the adhesive. Although the total replacement of wheat flour with oil-tea cake led to a slight decrease in wet shear strength, the adhesive still had a relatively high bonding strength (close to that of the wheat flour–based adhesive), which means it meets the Product Standard PS1-95 requirement. This indicates that the oil-tea cake, a forestry residue material, can be used in place of wheat flour (that can be better used as food) for the production of plywood panels. In addition, the Product Standard PS1-95 requires a minimum average of 85 percent wood failure for exterior-type plywood constructions. All wood failure percentages of the plywood bonded with PF adhesives after the wet shear strength test were higher than the minimum value. Therefore, this study showed that oil-tea cake has strong potential to serve as an effective and low-cost extender for PF plywood adhesives.

Conclusions

Both PF adhesives with oil-tea cake and oil-tea cake/wheat flour as extenders had satisfactory mixing viscosity and working life that were comparable to that of the standard wheat flour–based plywood adhesive. Oil-tea cake–based adhesives had a much stronger curing reaction than the adhesive with wheat flour as determined by the larger ΔH released during curing. Although the bonding strength of adhesive with oil-tea cake was slightly lower when compared with the wheat flour–based adhesive, it remained relatively high and was comparable to that of the wheat flour–based adhesive. These results indicate that forestry residue-source oil-tea cake is a promising extender to substitute for wheat flour in plywood adhesive applications.

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