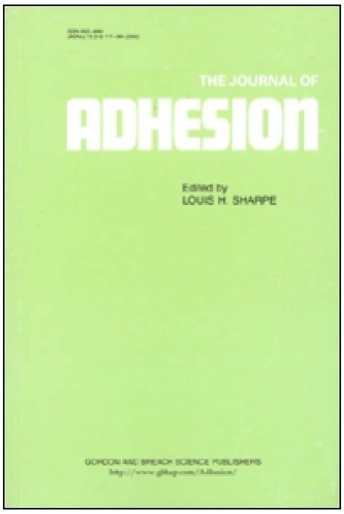

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The Present Status and Potential of Kraft Lignin-Phenol-Formaldehyde Wood Adhesives

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The Present Status and Potential of Kraft Lignin-Phenol-Formaldehyde Wood Adhesives

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Kraft lignin (KL), a phenolic polymer formed during the kraft pulping process, is presently burned as a low value fuel. For decades, researchers have attempted to use KL as an inexpensive substitute for phenol in phenol-formaldehyde (PF) resins, but no one has produced a commercially satisfactory KL–PF resin. This paper reviews the literature on the present status of KL–PF adhesives and makes recommendations on needed research.

Kraft lignin solutions are complex mixtures which have broad molecular weight distributions, high viscosities, relatively low reactivities, and low solubilities. Attempts to overcome these inherent problems include methylolation of lignin to improve reactivity, the use of co-solvents to improve solubility, and ultrafiltration to yield more homogeneous molecular weight fractions. Future research efforts need to focus on understanding the fundamental chemical and physical properties of kraft lignin and its resins. The search for an economic lignin-based wood adhesive should continue.

Key Words: Adhesives, Kraft-lignin, Lignin, Phenol-formaldehyde resins, Review, Wood Adhesives.

INTRODUCTION

Phenol-formaldehyde (PF) wood adhesives are used in panels intended

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for water-resistant, exterior applications. The wood-based panel industry would find it desirable to replace the phenol component in PF resins with a less expensive substitute that has similar physical properties. Kraft lignin (KL) is an available, relatively inexpensive, polyphenolic waste material that can be used as a reactive phenol substitute to form a composite kraft lignin-phenol formaldehyde (KL–PF) wood adhesive. Numerous attempts have been made to incorporate kraft lignin into PF resins but no commercially satisfactory resin has been synthesized.[1–4] Research efforts in this field need to be continued and new directions explored.

During the kraft pulping process, wood is contacted with a solution of sodium hydroxide and sodium sulfide at an elevated temperature (150–170°C) and pressure. Under these pulping conditions, wood is converted into fibers that can be used to form paper. During this process, lignin is degraded into a lower molecular weight, alkaline soluble material, as shown in Figure 1. Approximately 24 million tons of kraft lignin are produced annually in the U.S. and Canada.[5] This lignin is efficiently burned as a fuel in the kraft chemical recovery cycle, but valuable water-resistant adhesives can likely be produced from this waste material. Since most kraft mills are limited by their capacity to burn lignin in the chemical recovery process, the removal of some organic material from the black liquor stream could conceivably increase pulp mill production without significantly upsetting the energy balance.[6]

Kraft black liquor is a complex mixture of kraft lignin, hydroxy sugar acids, and inorganic salts.[7,8] These compounds are difficult to separate and purify economically. Lignin can be crudely precipitated from black liquor with sulfuric acid or flue gases.[9–11] Ultrafiltration, a more selective separation method, is currently being investigated by several pulp and paper companies as a way to separate lignin from the low molecular weight organic and inorganic salts.[12] The method chosen for isolating kraft lignin will directly affect its suitability for adhesive applications, as lignin preparations can vary considerably in molecular weight, solubility, and reactivity.

PHYSICAL AND CHEMICAL PROPERTIES OF KRAFT LIGNIN

The physical properties of kraft lignin solutions[13–19] are very

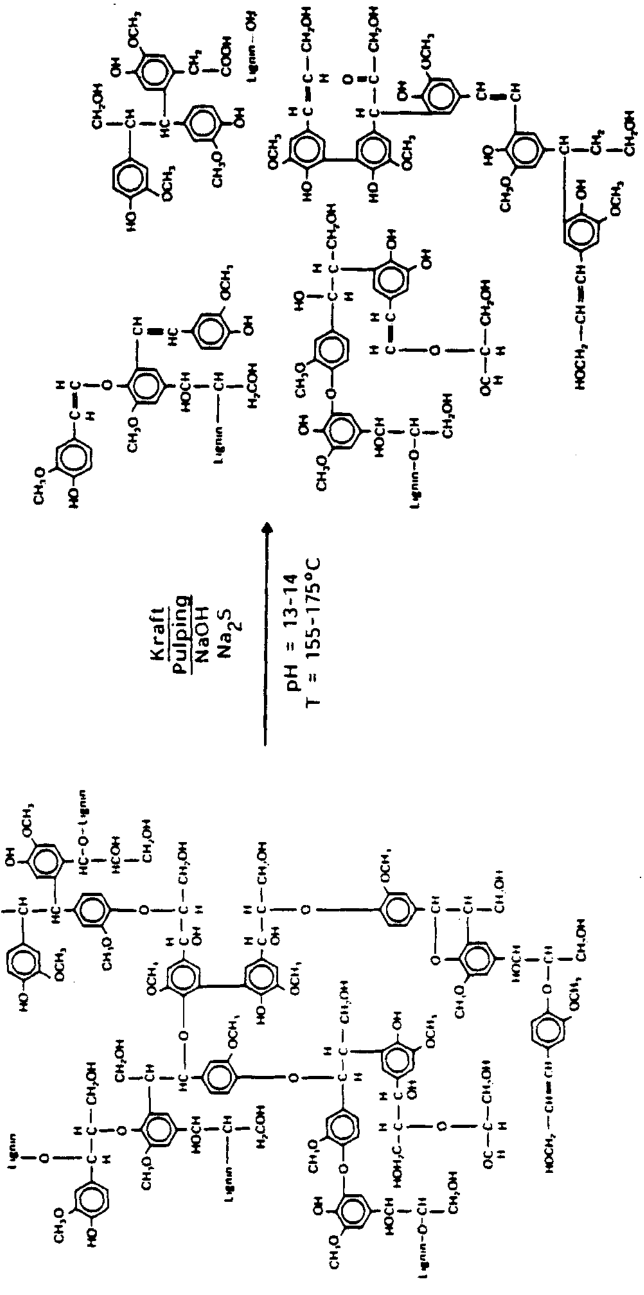


FIGURE 1 A structural representation of softwood lignin being decomposed during kraft pulping into kraft lignin segments.[1]

TABLE I
Functional groups in kraft lignin (after Ref. 11)

Functional Group	Pine Kraft Lignin (no. per 100 C_9 units)		Relative Percentage
1. Total OH	120		
Guaiacyl OH		60	50%
Catechol OH (2 ×)		12	10%
Aliphatic OH		48	40%
2. Total COOH	16		
3. Total CO	15		
Coniferyl aldehyde		—	
Alpha carbonyls		5	33%
Beta and/or other carbonyls		10	67%
4. Benzyl alcohol and ether	<6		
5. Carbon-Carbon double bonds	7		
6. Free 5-positions	25–40		

important to adhesive characteristics. Kraft lignin has limited solubility in water but can be dissolved in alkaline solutions. Lignin molecules are strongly associated through intermolecular hydrogen bonds. This fact probably accounts for the wide diversity in apparent molecular weights reported in the literature.[14] A typical pine kraft lignin has a number average molecular weight of 1600 and a weight average molecular weight of 3500.[11] The lignin polymer is thermoplastic with a glass transition temperature that is both moisture and molecular weight dependent.[13] The solubility, molecular weight, and degree of association of lignin critically affect the rheological properties of KL–PF adhesive systems.

Kraft lignin is a three-dimensional polymer containing a broad array of functional groups (Figure 1 and Table 1) which determine its reactivity in adhesive applications. Approximately 120 hydroxyl groups per 100 phenyl propane units are present in lignin. Of these hydroxyls, 60% are attached to the aromatic ring and 40% are attached to the side chain. Approximately 16 carboxylic acid groups and 15 ketone or aldehyde carbonyl groups are present per 100 phenyl propane units. Five of the carbonyls are located on the alpha carbon and the remaining 10 are on the beta or other carbons (see Figure 2 for specific positional nomenclature). There are less than 6 benzyl alcohol groups and approximately 7 nonaromatic carbon-carbon double bonds per 100 phenyl propane units. The number and type

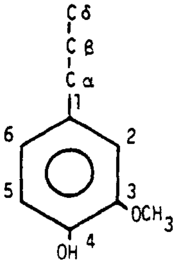


FIGURE 2 Numbering system for C_6C_3 subunits composing kraft lignin.

of functional groups become important when considering KL reactions with formaldehyde or when considering possible sites for condensation.

KRAFT LIGNIN REACTIONS WITH FORMALDEHYDE

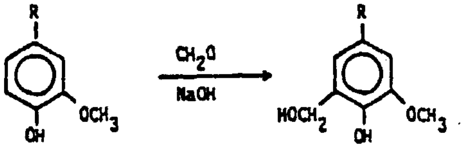
Kraft lignin has a limited ability to condense with itself and with PF resins. Therefore, it is often methyolated with formaldehyde to increase its reactivity prior to resin formulation. Essentially two types of reactions can occur between lignin and formaldehyde in alkaline solution. Formaldehyde can react with the phenolic ring, predominantly at the 5-position, or it can react with the side chain. Approximately 33% of the phenol units have a free 5-position[11] which is reasonably reactive to methylolation or condensation. The side chain reaction with formaldehyde typically occurs next to a carbonyl or at the beta carbon of an alpha-beta double bond conjugated to a free phenol. Alpha-beta double bonds and alpha carbonyls deactivate the ring toward methyolation.[20]

Representative types of alkaline-catalyzed reactions that can occur between lignin and formaldehyde are shown in Table 11.[1,20] As shown in reaction A, guaiacyl units can react with formaldehyde to yield methylol groups at the 5-position. Catechol units can yield methylol derivatives at the 2, 5, and/or 6-positions as shown in reaction B. Mixed aldol reactions can occur between formaldehyde and the carbon next to a carbonyl on the side chain as shown in reaction C. Methylolation can also occur by a Prins reaction in which formaldehyde reacts with the beta carbon of a double bond adjacent to the ring (reaction D).

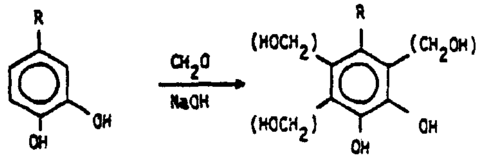
TABLE II

Representative reactions that can occur between lignin and formaldehyde in alkaline solutions

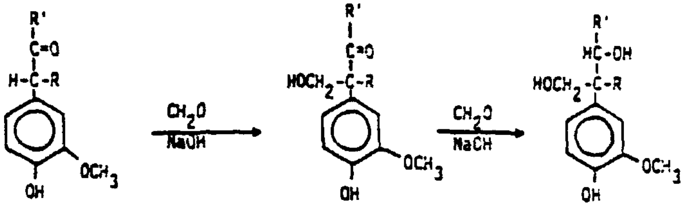
A. Methylolation of guaiacyl units at the 5-position (Lederer-Manasse Reaction).



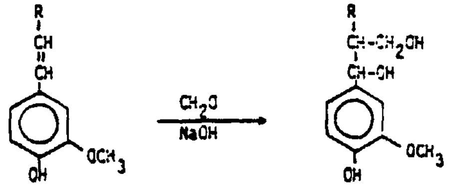
B. Methylolation of catechol units at 2, 5, and/or 6-position (Lederer-Manasse Reaction).



C. Methylolation of the side chain alpha to a carbonyl (Tollens Reaction) followed by reduction of the carbonyl (Crossed Cannizzaro Reaction).



D. Methylolation of the side chain at the beta position (Prins Reaction).



In a typical lignin methylolation reaction, approximately 40 methylol groups are added per 100 phenyl propane units.[20] Approximately 75% of these methylol groups are on the aromatic ring, and 25% are on the side chain. These methylol groups can condense with other lignin molecules or with PF prepolymers in the adhesive mixture to form methylene bridges during hot-pressing.

KRAFT LIGNIN-PHENOL-FORMALDEHYDE (KL–PF) ADHESIVES

Researchers attempting to synthesize KL–PF adhesives[21–37] have taken several different approaches as shown in Table III. In general, crude kraft lignin and black liquor have been methylolated with formaldehyde to yield a reactive prepolymer intermediate. The methylolated kraft lignin (MKL) or KL have been mixed with a PF resole or with phenol and formaldehyde to form plywood, particleboard, and waferboard adhesives. However, the limited reactivity, heterogeneity, low solubility, high viscosity, and relatively high cost of lignin resins have thus far deterred commercial development.

TABLE III
Research approaches to the synthesis of kraft lignin-PF adhesives (KL–PFs)

A. Production of methylolated pre-polymer intermediates

1. Kraft black liquor + $CH_2O \xrightarrow{NaOH}$ Methylolated kraft lignin
2. Kraft lignin + $CH_2O \xrightarrow{NaOH}$ Methylolated kraft lignin
3. Phenol + $CH_2O \xrightarrow{NaOH}$ PF resole

B. Production of KL–PF adhesives from pre-polymer intermediates

1. Methylolated kraft lignin + PF resole ⟶ KL–PF
2. Kraft lignin + PF resole ⟶ KL–PF
3. Kraft lignin + Phenol + CH_2O ⟶ KL–PF

A. Problems

The limited reactivity of KL is a significant problem in substituting it for phenol in PF resins. For example, lignin adhesives typically require longer press times, higher press temperatures, and have poorer water-resistance than pure PF resins. Steric effects related to the size and shape of KL molecules also reduce the reactivity of lignin. Future lignin adhesives will likely require the addition of some PF, isocyanate, or other cross-linking resin to improve the reactivity and physical characteristics of lignin resins.

The heterogeneity of lignin preparations is also a significant problem. Reproducible, standard lignin resins are difficult to prepare because kraft lignin samples vary according to wood species, pulping conditions, and isolation method.[28] The broad molecular weight distribution of KL also is a critical problem in adhesive formulations as it can affect the solubility, viscosity, reactivity, wood penetration, wet and dry strength, and the amount of PF that can be substituted in KL–PF resins.

The low solubility and high viscosity of lignin solutions are persistent problems. Even at high alkali concentrations (10% based on lignin weight) lignin solubility is still below an acceptable 35–45% solids level for a lignin adhesive formulation.[26,28] The high alkali concentrations required for dissolution cause additional problems as control over the intermediate condensation reactions is reduced. The

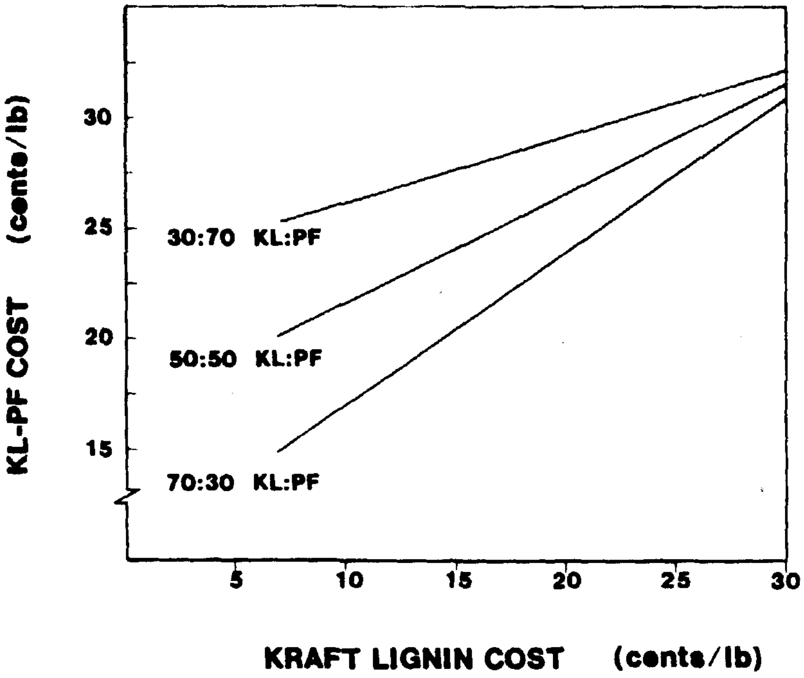


FIGURE 3 The effect of kraft lignin cost on KL–PF cost at three different substitution ratios. A PF resin solids cost of 33 cents/lb was assumed.

high viscosity of lignin resins can cause application problems, decrease pot-life, and limit the addition of low cost fillers and extenders.

One additional problem that has not been adequately addressed in the literature is the economics of KL–PF adhesives. It is difficult to produce a good KL adhesive at a competitive price because several separation and processing steps are typically required. Figure 3 shows the effect of kraft lignin cost on KL–PF cost at three different substitution ratios. The economics of KL–PF adhesives appear marginal when lignin costs are above 15 cents/lb and current raw phenol prices are 36 cents/lb.[39]

B. Partial solutions

The present status of KL–PF adhesive science can be summarized with a review of several pertinent articles and patents dealing with partial solutions to KL–PF problems. It should be pointed out that the KL–PF literature is contradictory and difficult to interpret. The problem has been compounded by in-house studies which have not been reported in the literature.

1. Reactivity The limited reactivity of KL can be increased by methylolating lignin or black liquor as a first step in adhesive synthesis. Methylolated KL–PF adhesives appear to have much better wet strength and reactivity than unmethylolated KL–PFs.[23] The methylol groups provide reactive sites that increase the amount and rate of cross-linking, but the rate of MKL condensation is still less than that of PF resins.

Enkvist[27] methylolated kraft lignin by heating black liquor and formalin at 70°C for 3 hours. The MKL black liquor solution was then combined with a PF resole and 35% formalin to form a plywood adhesive. In another typical methylolation reaction, Dolenko and Clarke[23,24] reacted black liquor with formaldehyde for three days at room temperature. The MKL was then precipitated with acid and combined with a PF resole under high-speed mixing to form a dispersion of fine particles that could be used as an adhesive. This adhesive mixture was spread on poplar veneers and pressed at 177°C and 200 psi (1380 kPa) for 10 minutes to make plywood. The panels had good strength characteristics at these relatively high pressing temperatures and long times.

In a similar study, Rosenberg[36] methylolated kraft lignin in a black liquor solution. The MKL was acid-precipitated and filtered from the solution. In one example, the MKL and PF were mixed as liquids, spray-dried, and then ball-milled to produce a powdered adhesive. In a second example, the MKL was dissolved in a PF resole at a 60:40 ratio and then applied to wafers at a level of 2% (resin solids to wafers). The mats were pressed at 210°C and 400–500 psi for 5–6 minutes to yield satisfactory waferboards.

Sudan and Berchem[29] made a powdered adhesive in a similar fashion without acid-precipitating the lignin. They mixed black liquor with a PF resin at ambient temperature and then spray-dried the solution. The resin coated wafers (3% resin by weight) were then pressed for 6 minutes at an unstated temperature. Even without methylolation or lignin purification, the waferboard adhesive was capable of meeting the Canadian Standards Association (C.S.A.) criteria for use in construction. An adhesive composed of 91% black liquor solids also met the C.S.A. M.O.R.-Dry criteria while an adhesive containing 83% black liquor solids met the C.S.A. M.O.R.-Boiled criteria.

Dolenko and Clarke[23] attempted to enhance KL–PF reactivity and wet strength using an acidic, rather than a basic, catalyst. Wet strength was observed to increase as the pH decreased. The low solubility of lignin in acidic solutions is a problem with this approach.

2. Low Solubility and High Viscosity The high molecular weight of KL makes it difficult to produce high solids, low viscosity KL solutions. Lignin will dissolve in alkali to yield solutions that contain approximately 35% solids, but at higher solid contents the lignin solutions become too viscous for practical use.[28] Adams and Schoenherr[28] were able to produce high solids, low viscosity lignin solutions by dissolving kraft lignin in phenol-water or phenol-water-alkali solutions. These lignin solutions had viscosities of 10,000 cps or less and were suitable for use in KL–PF resins.

Hollis and Schoenherr[26] used this phenol solvent system to produce lignin adhesives which had good wet strength. Acid-precipitated lignin was dissolved in phenol and then mixed with a typical resole. The mixture was refluxed at 95°C for 20 minutes and then heated for approximately one hour at 80°C. The resin had a solids content of 37%, a pH of 11.4, and a viscosity of 460 cps at 25°C. The resin was formulated into an adhesive by adding fillers, a defoaming agent, sodium hydroxide, and water. A typical three-ply panel made with the adhesive was pressed at 140°C and 175 psi for 6 minutes and exhibited 94% wood failure following a 120 minute vacuum pressure soak.

3. Broad Molecular Weight Distribution Two research groups have investigated ultrafiltration as a method for producing homogeneous lignin molecular weight fractions for use in adhesives.[21,22,38] Ultrafiltration is also of current interest to the pulp and paper industry for purifying waste streams and concentrating components.[12,40–42] In ultrafiltration of black liquor, the high molecular weight lignin is retained on one side of a porous membrane while the low molecular weight hydroxy sugar acids and inorganic salts pass through. Removal of these water-soluble salts should increase the water resistance of lignin adhesives. Specific lignin molecular weight fractions can be obtained by varying the pore sizes of the ultrafiltration membranes.

Forss and Fuhrmann[21,22] obtained several lignin molecular weight fractions by ultrafiltration and formulated the fractions into adhesives. These were applied to veneers, pre-pressed at 100 psi for 6 minutes, and then hot-pressed at 135°C and 230 psi for 2 or 4 minutes. The adhesive (known commercially as Karatex) gave results comparable to a PF resin as shown in Figure 4. Panels made with Karatex

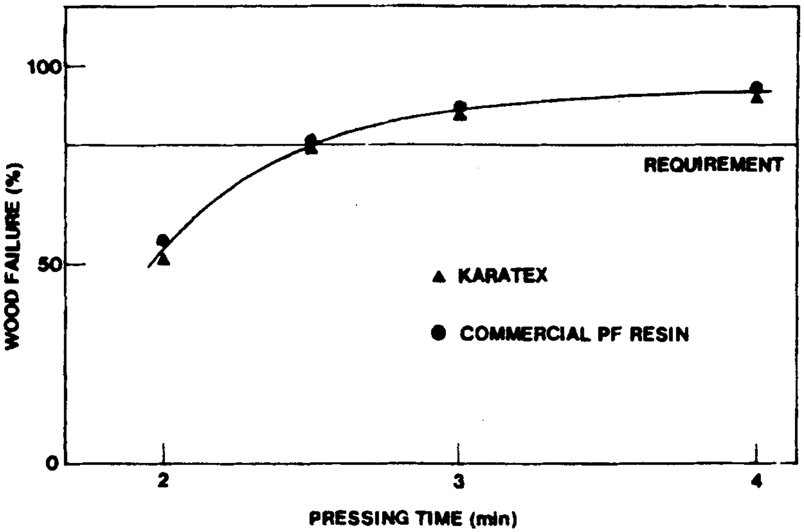


FIGURE 4 Properties of plywood glued with Karatex adhesive compared with PF-glued plywood.[21]

met Finnish standards for exterior-grade plywood and German standards for weatherproof particleboard. Karatex was evaluated on a full plant scale, but apparently is not being used as a commercial wood adhesive.

Forss and Fuhrmann[21] concluded from their study that the adhesive strength increased as the percentage of high molecular weight lignin in the adhesive was increased. They attributed the poor lignin reactivity observed in earlier adhesive research to the broad lignin molecular weight distribution. Forss and Fuhrmann suspect that large amounts of PF resin are required to link the low molecular weight lignins whereas smaller amounts of PF resin are required to link the high molecular weight lignins. The trends observed in their study cannot be conclusively attributed to molecular weight effects because the inorganic salt concentration varies in each ultrafiltered fraction.[2]

A similar ultrafiltration, molecular weight study was performed by Shen and Calve[43] on sulfite lignin adhesives. Their results, in direct contrast to Forss and Fuhrmann's work, indicated that panels made from the low molecular weight lignin fractions had better strength, required shorter press times, and were more water-resistant than those made from the high molecular weight fractions.

C. Research needs

The macromolecular properties of kraft lignin solutions are poorly understood, and additional fundamental research is needed. More information on methods for improving the chemical reactivity of lignin resins is also needed. Most KL–PF researchers have run methylolation reactions for several days at room temperature rather than at higher temperatures and shorter times, as is typical with PF resins. Shorter reaction times would be desirable, but accelerated molecular weight growth and viscosity problems limit this approach. The kinetics of KL methylolation and resin synthesis are poorly understood and should be investigated.

KL–PF catalysts merit research attention since they may have the potential to accelerate resin cure, decrease press time, and lower press temperature. For example, recent work with acid-catalyzed KL–PF resins has produced strong, water-resistant panels.[23] Lignin solubility is a problem with this approach, but the development of acid-catalyzed, powdered KL–PF adhesives appears promising.

The effect of higher press temperatures on improved reactivity and wet strength needs further investigation. A study on the optimal condensation temperature of organosolv lignin adhesives[44] and a preliminary DSC analysis of KL resins in our laboratory[45] suggest that curing begins at approximately 170°C which is higher than the cure temperatures used by most investigators. The temperature required for resin cure may be closer to particleboard and waferboard pressing temperatures than those used for plywood.

Additional studies delineating the effect of lignin molecular weight on adhesive characteristics appear promising and may lead to commercial development of KL–PFs. Ultrafiltration appears to be a promising method for isolating homogeneous kraft lignin fractions that could have improved viscosity, solubility, reactivity, and permeability characteristics.

The ultimate goal in future research will be to produce a KL–PF adhesive that has competitive physical properties as well as a significant price advantage over PF resins. In order to obtain this goal, the economics involved in obtaining and processing lignin must remain a prime consideration.

CONCLUSION

Commercial KL–PF resins may be used as wood adhesives in the future, but many inherent problems with lignin must still be overcome. Future research efforts should attempt to understand the fundamental physical and chemical properties of lignin and its resins. Lignin molecular weight can affect many resin variables and may be the key to producing a commercial KL–PF resin. Ultrafiltration of black liquor may be capable of yielding a relatively inexpensive homogeneous kraft lignin that would produce a good adhesive. Even though there are substantial problems with lignin-based adhesives, the search for a cheap, renewable, lignin-based wood adhesive system should continue.

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