



Department of Wood  
Science  
University of BC  
2424 Main Mall  
Vancouver BC  
Canada

**Development of Thick MPB Strand Based Wood  
Composites  
(Year one of a three year project)**

by

Frank Lam  
Professor

J.F. Kadla  
Associate  
Professor

G.D. Smith  
Assistant  
Professor

Azzeddine Oudjehane  
Research Associate

J.B. Wang  
Ph.D. Student

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## **EXECUTIVE SUMMARY**

The largest Mountain Pine Beetle epidemic in Canada's history coupled with limitation of annual allowable cut will result in the utilization of a large volume of older dead Lodgepole Pine in the near future. The morphology and chemistry of this wood are altered and these properties are related to the time-since-tree-death. As well, this timber contains blue stain in the sapwood and extensive checking which lowers its commercial value for use in lumber and pulp. Furthermore, increased amount of fines and residues is expected from processing of this resource.

This proposal describes the first year of a three year study to generate fundamental knowledge needed to develop new MPB strand based thick wood composites. Year one is an exploratory study to understand the basic background issues relating to the properties, performance and manufacturing of such products.

During the first year of this project, the following activities have been successfully completed:

- A detailed literature review on adhesive interaction and mechanics has been initiated.
- Preliminary conceptual design of a small humidity chamber (20 mm x 20 mm x 20 mm) that can be coupled with the UBC DMA equipment to allow the study of the behaviour of adhesives considering the temperature and humidity interaction has been initiated
- Literature review on creep and non-linear visco-elasticity of strand based composites has been initiated.
- Development of a user friendly interface of the existing UBC 3-dimensional heat and mass transfer and consolidation model is underway
- Modification of existing UBC 3-dimensional heat and mass transfer and consolidation model to consider variable step closure of the press and preheating scheme
- Preliminary planning of experiments to be conducted with regards to the validation of the press-consolidation model and the non-linear creep study.
- A literature review on in-plane and out of plane permeability of wood composites.
- A tentative designs of the jig for measuring the in plane permeability while the mat is subjected to and out of plane stress.
- Feasibility and design of the jig test.

Herein, a summary of the respective findings is included.

## PROJECT RATIONALE

New MPB thick strand based structural composites products can help absorb a large volume of MPB wood. Such members can be used as beams, headers, columns in low-rise commercial, multi-family residential and single family residential market. Given the improved product consistency as an engineered wood product, it is especially suitable for low rise commercial and multi-family residential applications where load demands are higher compared to single family applications.

Currently some OSB mills are manufacturing OSB panels with a mixture of MPB and aspen wood. These mills are addressing many technical issues associated with OSB manufacturing with MPB wood including processing of the dry logs, excessive fine content, etc. Coupled with these issues is the fact that there is a shortage of MDI resin commonly used in OSB manufacturing. As an engineered wood product, one of the key properties is its structural response under long term loading (creep). Understanding the influence of this behaviour by the fiber, adhesive and pressing method is critical to the development of new engineered wood products

To manufacture a thick oriented strand product with MPB wood, additional fundamental knowledge is needed to consider the influence of strand properties, pressing parameters, heat and mass transfer phenomena in the mat during pressing, adhesive interaction, and the nonlinear viscoelastic behaviour of the material on the performance of the product. This proposed project builds on a past NSERC Strategic Grant (Steiner, Avramidis and Lam) and NSERC Collaborative Grant (Lam) where data base and models were developed on Aspen and PF based OSB panel product. The work will be extended to include study of modeling the pressing of thick products, alternative resin behaviour, and the creep behaviour of MPB strand based products using non-linear viscoelasticity modeling. An end goal is the development and set up a user friendly tool to predict the structural performance of thick MPB Oriented Strand product

# PROJECT FINDINGS AND ACHIEVEMENTS FOR YEAR 1

## Adhesive

### ➤ State of the Art

Glues have been around for a long time; the ancient Egyptian used them in veneering the treasure of Tutankamun and the ancient Greek word for glue is κολλα, from which we got colloid. In all centuries up to and including the 19<sup>th</sup> glues originated from plants and animals; during the 20<sup>th</sup> century synthetic chemical have largely taken over and the more respectable name of adhesives has been introduced. Adhesion is essential for printing inks, sealants, and paints and other surface coating, and at interface in composite materials such as steel or textile fibers in rubber tyres and glass- or carbon-fibers in plastics. Mother natures uses adhesion rather than mechanical fasteners (nuts and bolt, nails staples, etc.) in constructing plants and animals, and some animals are masters at exploitation of adhesion.<sup>1</sup>

Adhesives are not the only materials that must stick or adhere. A definition for adhesive is a material which when applied to the surface of materials can join them together and resist separation. The term adherent and substrate are used for a body or material to be bonded by an adhesive. Adhesive must wet the surface, spread and make a contact angle approaching zero. Intimate contact is required between the molecules of adhesive and atoms or molecules of the substrate in the surface. The adhesive must then harden to a cohesively strong solid. This can be by chemical reaction, loss of solvent or water or by cooling in the case of hot melt adhesives. Nowadays adhesives are used in all types of manufacture, in the construction of aircraft or plywood and in many cases have displaced other means of joining.<sup>1</sup> The use of adhesives is a daily occurrence in many wood-processing industries as well, such as in the particleboard, plywood, and finger-joints field. Adhesion is an important physicochemical phenomenon that has attracted considerable attention from many researchers in many fields of science.<sup>2</sup>

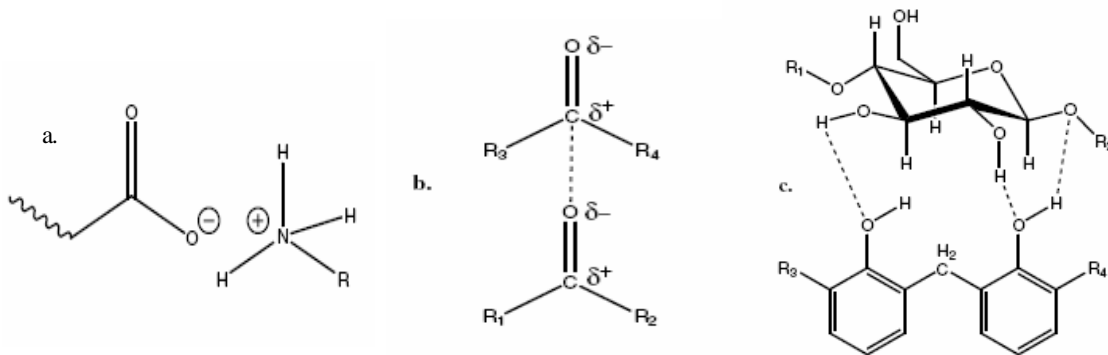
A disadvantage of adhesives as a mean of joining is that they are generally weakened by water and its vapor. Also, their service temperature ranges are less than for metal fasteners, being limited by their glass transition temperature and chemical degradation. Advantages include their ability to join dissimilar materials and thin sheet materials, the spreading of load over a wider area, the aesthetic and aerodynamic exteriors of joints and application by machine.<sup>1,2</sup>

Any materials that are bound to each other by an adhesive form a system that includes adhesion and cohesion.<sup>5</sup> Adhesion is established between two surfaces due to intermolecular forces whereas cohesion is the bonding of molecules together in the bulk. Adhesion refers to the interaction of the adhesive surface with the substrate surface. It must not be confused with bond strength. Certainly if there is little interaction of the adhesive with the adherent, these surfaces will detach when force is applied. However, bond strength is more complicated because factors such as stress concentration, energy dissipation, and weakness in surface layers often play a more important role than adhesion. Consequentially, the aspects of adhesion are a dominating factor in the bond formation process, but may not be the weak link in the bond breaking process. It is important to realize that, although some theories of adhesion emphasize mechanical aspects and others put more emphasis on chemical aspects, chemical structure and

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interactions determine the mechanical properties and the mechanical properties determine the force that is concentrated on individual chemical bonds. Thus, the chemical and mechanical aspects are linked and cannot be treated as completely distinct entities. In addition, some of the theories emphasize macroscopic effects while others are on the molecular level.<sup>3,4</sup> Thus, the mechanism of adhesion can be different for various materials.<sup>5,6</sup> In general the principal theories describing the phenomenon of the adhesion are the followings:

- Mechanical Entanglement/ Interlocking theory
- Diffusion theory
- Electronic theory
- Adsorption/Specific Adhesion theory
- Covalent Chemical Bonding theory



**Figure 1: Examples of various types of bonds, including (a) Ionic bond between an ammonium group and a carboxylate group (b) Dipole bond between two carbonyl groups, such as in a polyester, (c) Hydrogen bonds between a cellulosic segment and phenol-formaldehyde polymeric structure**

Adhesives may be defined as any substance capable of attaching materials together by means of surface attachment. This property is not necessarily an intrinsic characteristic of the substance itself since the adhesive may be much weaker than the materials joined together, but it is developed as the adhesive interacts with the adherends under certain conditions of temperature and pressure.

Most observations of adhesive interaction with wood are concentrated on scales of millimeter or larger. However, the wood-adhesive interaction needs to be evaluated in three spatial scales (millimeter, micrometer, and nanometer). The millimeter or larger involves observations by eye or light microscopy. The use of scanning electron microscopy allows observations on the micrometer or cellular level. On the other hand, the size of the cellulose fibrils, hemicellulose domains, and lignin regions are on the nanometer scale. The nanometer level is also the spatial scale in which the adhesive molecules need to interact with the wood for a bond to form. Tools, such as atomic force microscopy, developed for making observations on the nanoscale can be difficult to use with wood because its surface is rough on the micrometer scale.

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For the most part, adhesives used to bond wood together may be separated into two distinct groups, those adhesives such as animal, vegetable, casein, and blood glues which are formulated from materials of natural origin, and those adhesives which are based on synthetic resins derived from petroleum, natural gas, and coal, i.e., products of the petrochemical and related industries. The properties of various adhesives are discussed below separated according to these two categories, natural and synthetic adhesives.<sup>34</sup>

### ***Natural adhesives***

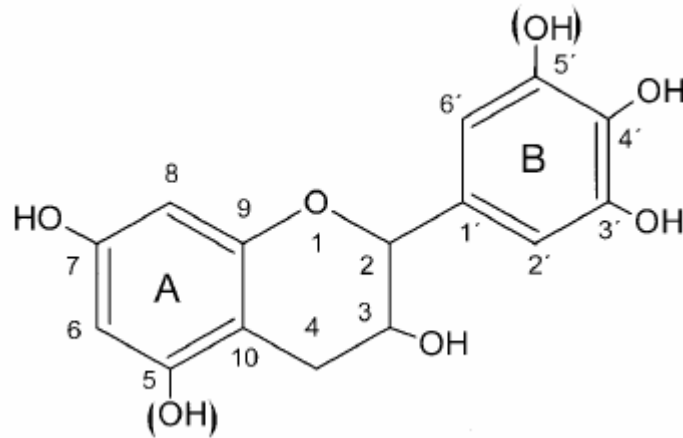
Natural adhesives have been replaced in many uses by synthetic polymers; but animal glues, starches, gums, cellulose and natural rubber cements continue to be used in large volumes. Organic adhesives derived from animal proteins made from collagen, a constituent of the connective tissues and bones of mammals and fish; blood albumen glue, used in the plywood industry; and glue made from casein, a protein constituent of milk, are employed in wood bonding and in paint. Vegetable adhesives include starch and dextrin derived from corn, wheat, potatoes, and rice used for bonding paper, wood, and textiles. Gums such as agar and algae when moistened provide adhesion for such products as stamps and envelopes.<sup>35,36</sup> The five groups of natural adhesives considered are:

- *Animal Glues:* usually made from the hides and bones of such animals as cattle, horses, and sheep and was usually classified as either hide or bone glue. Additional tissues such as sinews may also be used and substantial amounts of adhesive are made from fish. Animal glues may be purchased as a powder, as beads, or as a jelly. Animal glues have several shortcomings which have led to their replacement by other adhesives in a large part of the woodworking industry. Specifically, they have low moisture resistance, can be attacked by molds and fungi, inconvenient to use and must be applied at the proper temperature, and are relatively expensive. More importantly, there is strong competition for animal remains today, and as a result animal glues are in short supply.<sup>36</sup>
- *Vegetable based adhesive-Starch* Although starch glues still are used in the United States, they are used little in the wood industry. Of particular importance, they have been replaced by urea-resin adhesives in gluing interior-type hardwood plywood and furniture. As the name implies, the principal component of these adhesives is starch which may be obtained from a variety of plants including corn, potatoes, rice and cassava. Vegetable glues are relatively inexpensive and have a relatively long pot life. They set through loss of water, which may be quite slow, so that glued assemblies of vegetable adhesive must often remain clamped overnight. Vegetable glues were widely used in such applications as veneering. Lack of moisture resistance and dissipation by micro-organisms of vegetable glues cause their replacement with synthetic adhesives.<sup>37</sup>
- *Protein based adhesives-Soybean and Casein:* The principal protein-based glue is manufactured from either soybean meal or the vegetable protein isolated from it. Soybean glue has properties and characteristics which are similar to casein glue, but lacks its water resistance. Soybean glue may be hot-pressed, and in the past was widely used for interior grade Douglas-fir plywood. Now it is displaced largely by phenolic resin in the softwood plywood industry. Casein glue is prepared from casein curd which is precipitated from skim milk. About eight

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gallons of skim milk are required to make one pound of dry casein. To prepare the glue, the curds are dissolved in an aqueous alkaline solvent such as lime water to which other chemicals such as formaldehyde or copper chloride are added. Until the introduction of synthetic resins, casein glues were important because of their superior moisture resistance. They tend to stain those species of wood which are rich in tannic acid such as the oaks, so that casein glue is largely excluded from the furniture industry. They also dull woodworking tools rapidly and are attacked by molds and fungi. In recent years, casein glues have become expensive because of the increasing needs for milk to feed the hungry of the world.<sup>38</sup>

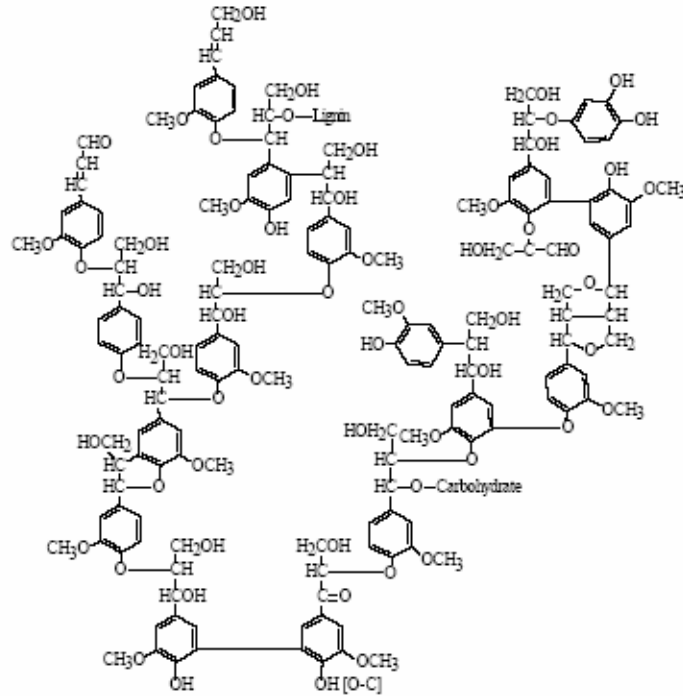
- *Flavonoids wood based adhesive-Tannin:* Poly-flavonoid tannin extracts have been produced and used industrially in many applications since the end of the nineteenth century.<sup>2</sup> Among their many uses, the major applications have been as tanning agents for the manufacture of leather and wood adhesives.<sup>1,2</sup> Polyflavonoid tannins are natural poly-phenolic materials, composed mostly of flavan-3ol repeating units (Fig. 2) and smaller fractions of polysaccharides and sugars. These polyphenolic materials can be hardened by reaction with formaldehyde or hexamethylenetetramine (HEXA) crosslinking agents.<sup>39</sup> There are some fundamental differences between different polyflavonoid tannins that can be used for thermosetting wood adhesives. Flavonoid units in such tannins present phloroglucinol or resorcinol A-ring and catechol or pyrogallol B-rings, the repeating units are linked to each other C4-C6 or C4-C8, the former predominating in tannins composed primarily of fisetinidin (resorcinol A-ring, Catecho, B-ring) and robinetinidin (resorcinol A-ring, Catecho, B-ring) repeating unit. The C4-C8 interflavonoid linkage predominates in tannins composed of catechin (phloroglucinol A-ring, catechol B-ring) and gallocatechin (phloroglucinol A-ring, pyrogallol B-ring) repeating units. When the polymeric tannins are composed of fisetinidin and robinetinidin units, the polymers are called profisetinidin and prorobinetinidin, respectively; when they are composed of catechin and gallocatechin, the polymers are called procyanidin and prodelphinidin, respectively. The free C6 and/or C8 sites on the A-ring are the sites reactive with formaldehyde, due to their strong nucleophilicity to form adhesive under the usual conditions under which these materials are used.<sup>2</sup> Tannin – formaldehyde adhesives are obtained by hardening of polymeric flavonoids of natural origin, or condensed tannins, by poly condensation with formaldehyde. As tannins are phenolic in nature one would imagine that the standard technology used to prepare and use synthetic PF adhesives would apply. The much higher reactivity of tannins toward formaldehyde due to their A-ring or phloroglucinolic nuclei ensures rates of reactions under parity of condition, which are between 10 to 15 times faster than the reaction of phenol with formaldehyde. Their faster reactivity also leads to much faster gel times and hence faster pressing times than for synthetic PF adhesives, and truly exceptional exterior-grade performance.<sup>39</sup> The most widely used industrial tannins are obtained from the wood of the quebracho tree (*Schinopsis balansae*, Argentine) and from the bark of mimosa (*Acacia mearnsii*, Brazil and South Africa). Most scientific studies and technological reports on the application of tannin extracts are related to their use in the manufacture of adhesives for particleboards and plywood, as well as for laminated woods.<sup>39,40</sup>



**Figure 2: Flavan-3-ol repeating unit in polyflavonoid tannin.**

- Wood based adhesive- Lignin:* The occurrence of lignin as a waste product in pulp mills has made it an attractive raw material for adhesive ever since the beginning of the sulfite pulping of wood. A lignin powder produced as a by-product of pulping or hydrolysis operations from straw is an acceptable adhesive. The first patent dealing with application of spent sulfite liquor (SSL) as an adhesive for paper, wood, and other lignocellulosic material, date back to the end of the nineteenth century. On the other hand, the technical utilization of lignin on a large scale is still at very low level for the amount produced worldwide. Lignin produced by hydrolysis would not contain sulfur and therefore could be rendered more reactive and produce a much more competitive adhesive. However, at the present time, there is a surplus of lignin from wool, thus any rice straw lignin product would face intense competition. As a major wood component, native lignin is neither hygrscopic nor soluble in water. However during technical sulfite pulping, lignin becomes soluble in water, due to partial degradation and introduction of sulfonic acid group (-SO<sub>3</sub>H). To apply SSL as an adhesive, it must be converted to an insoluble state during the curing period. Cross linking in lignin can be achieved either by condensation or by radical coupling reactions. However, either high temperature and long heating times or adding mineral acids are required for these condensation reactions, which cause structural changes or charring in the wood particles. Recently, cross-linking of the lignosulfonate molecules by radical combinations, which avoids mineral acids and high temperatures, has been developed, but this presents disadvantages as well, as the use of peroxide is not favored in wood-processing plants. Lignin is composed of phenylpropane (C<sub>9</sub>) units that are linked together by carbon to carbon as well as carbon to oxygen (ether) bond (fig.3).<sup>41,42</sup> According to its structure as a polyphenol, lignin as an adhesive should be similar to PF resins. This is true for native lignin in wood, while technical lignin (lignosulfonate or black liquor) has to be additionally cross linked to change it into insoluble resin. However, condensation reactions in lignin by heat or mineral acids cannot be as effective as in synthetic PF resins, due to the lower number of free positions in the aromatic nuclei of lignin and their considerably lower reactivity than in PF resins.

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**Figure 3: Lignin Structure Model**

First, there is only 0.5 free *o*-position (ortho to the phenolic groups) per C9 units; and the ortho positions are less reactive. Second, there is less than one benzyl alcohol or ether group per C9 unit in lignin while in synthetic PF resins up to three methylol groups can be introduced on each phenolic ring. Finally the aromatic nuclei in lignin are considerably less reactive than phenol toward hydroxybenzyl alcohol groups, due to the presence of methoxy equivalent groups rather than hydroxyl groups on the lignin aromatic rings. For these reasons, lignin in technical spent liquors cannot be as effectively cross-linked as synthetic PF resins.<sup>42,43</sup> In most cases additional cross-linking agents for lignin are necessary, such as epoxides, polyisocyanate, polyol and etc. therefore few numbers of patents have been pending during the past three decades dealing with lignin as adhesive for particleboard, plywood and fiberboard.<sup>42</sup>

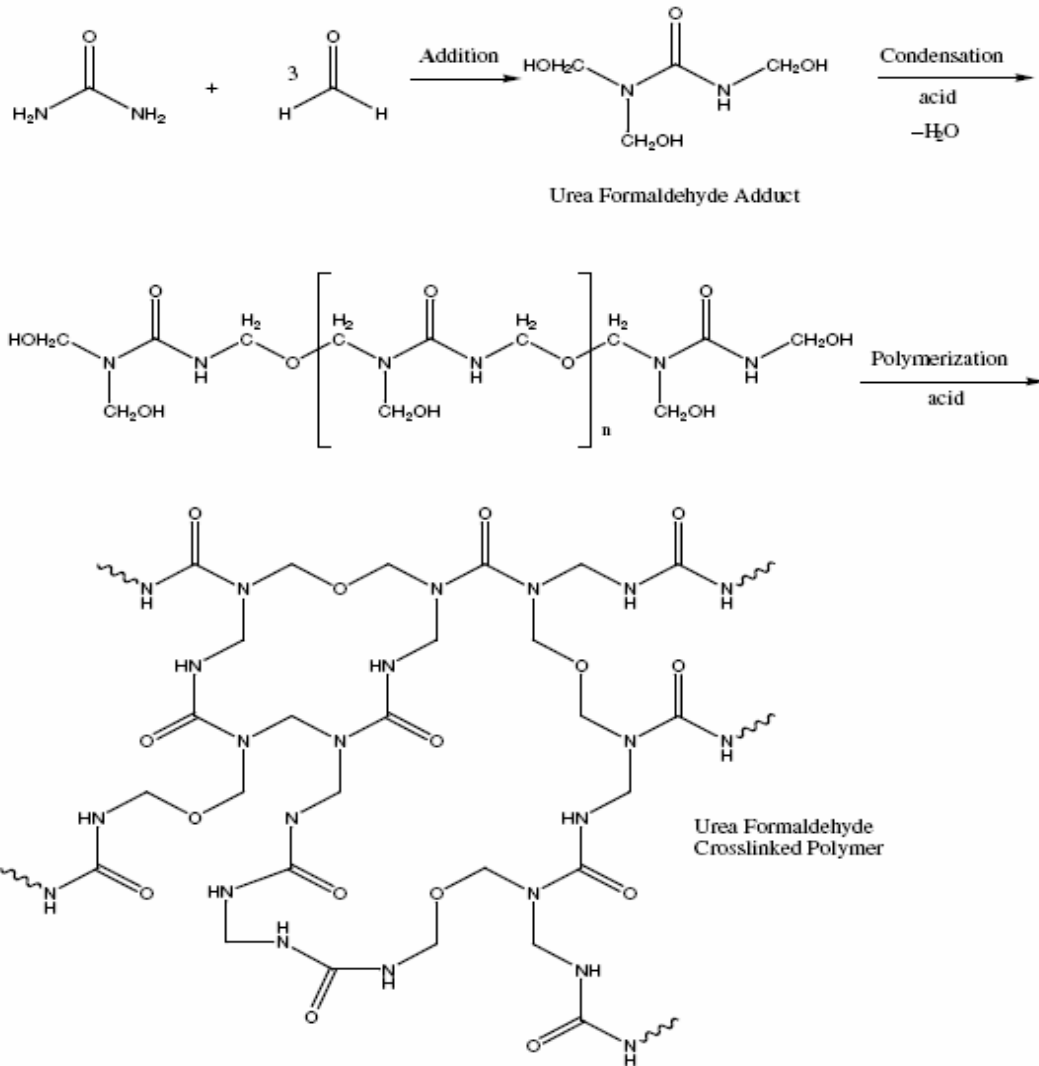
### Synthetic Resin Adhesives

Synthetic resins are man-made polymers which resemble natural resins in physical characteristics and can be tailored to meet specific woodworking requirements. These resins impart to glue lines and joints the highest water resistance attained to date. In contrast to the natural adhesives which at the best can resist only a moderate amount of moisture, properly formulated synthetic adhesives appear able to withstand repeated direct wetting. Synthetic resins were introduced as woodworking adhesives during the early 1930's. Synthetic resin adhesives are separated into two distinct categories thermosetting adhesives and thermoplastic adhesives. Thermosetting adhesives during cross-linking reaction, undergoes an irreversible chemical and physical change which render them insoluble. Thermoplastic resins are pre-polymerized and set by loss of dispersing solvent. They do not undergo a chemical cross-linking reaction while curing, therefore, remain in a reversible state and can readily be softened by heating.

### *Thermoset Adhesive*

- *Urea-Formaldehyde (UF)*: Urea-formaldehyde resins are the most important and probably the most widely used thermosetting resin for wood. UF resins are polymeric condensation products of the reaction of formaldehyde with urea. Urea is reacted with formaldehyde, which results in the formation of addition products such as methylol compound. Further reaction and the concurrent elimination of water leads to the formation of low molecular weight condensate UF that is still soluble in water. Higher molecular weight polymer, which are insoluble are obtained by further condensing the low molecular weight of UF.<sup>44</sup> The reaction between urea and formaldehyde is complex and combination of these two chemical compounds results in three dimensional network in cured resin. This is due to functionality of four in urea and functionality of 2 in formaldehyde. The most important factors determining the properties of the reaction products are the relative molar portion of urea and formaldehyde, temperature and PH values. The rapid initial addition reaction of urea and formaldehyde is followed by a slower condensation, which results in the formation of polymer. The rate of this condensation polymerization of urea is PH dependent and decreases exponentially from a PH of 2 to 3 and to neutral PH value. No condensation occurs at alkaline PH values. It is very important in the commercial production of UF resins to be able to control the size of the molecules by condensation reaction, since their viscosity increases continuously as they grow larger. The most common method for the preparation of commercial UF resin adhesive is the addition of a second amount of urea during the preparation reaction. This consists of reacting urea and formaldehyde in more than equivalent proportions. Methylolation can in be carried out in much less time by using temperature of up to 90 to 95 °C. The reaction is completed under reflux by increasing the PH as soon as right viscosity is reached.<sup>45</sup> The advantage of UF adhesives are their 1) initial water solubility, 2) hardness, 3) no flammability, 4) good thermal properties, 5) absence of color in cured polymers, 6) easy adaptability to a variety of curing condition. They are widely used for the manufacture of interior grade plywood and also for the manufacture of particleboard. In particular, they are extensively used in producing hardwood plywood for furniture and interior paneling and for furniture assembly. Urea-formaldehyde resins may also be fortified with melamine resins to improve both their moisture and temperature resistance.<sup>46</sup>

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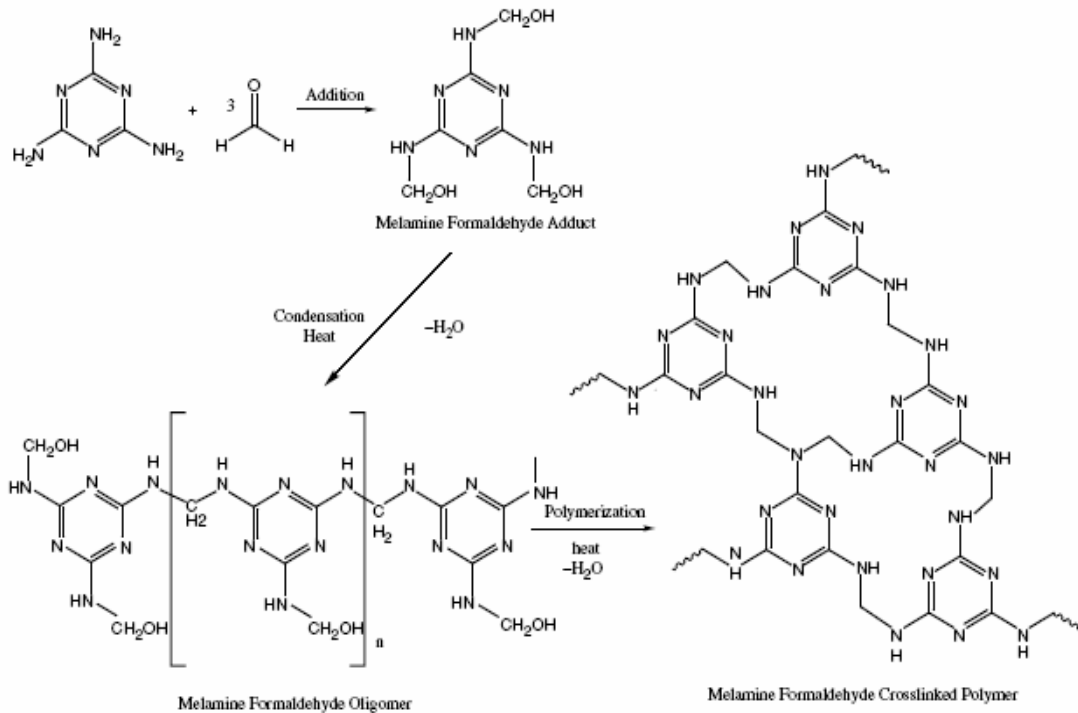


**Figure 4: Urea-formaldehyde polymerization goes through an addition reaction and then condensation to give an oligomer that is applied to the wood. After application, the polymerization is completed to give a crosslinked network.**

- Melamine-Formaldehyde (MF):** The condensation reaction of melamine with formaldehyde is similar to but different from the reaction of formaldehyde with urea. As with urea, formaldehyde first attacks the amino groups of melamine, forming the methylol compounds. However, formaldehyde addition to melamine occurs more easily and completely than addition to urea. The amino group in melamine easily accepts up to two molecules of formaldehyde. Thus complete methylolation of melamine is possible, which is not the case of urea. Up to six molecules of formaldehyde are attached to a molecule of melamine. Because melamine is less soluble than urea in water, the hydrophilic stage proceeds more rapidly in MF resin formation. Therefore, hydrophobic intermediates of the MF condensation appear early in the reaction. Another important difference is that MF condensation to give resins and their curing, can occur not only under acid conditions, but also under neutral or alkaline conditions. The mechanism of the reaction of methylol melamine to form hydrophobic intermediates is the same as

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from UF resins, with splitting off of water and formaldehyde. Methylene and ether bridges are formed and the molecular size of the resin increases rapidly and the final curing process transforms the intermediate MF to insoluble resin.<sup>47</sup>

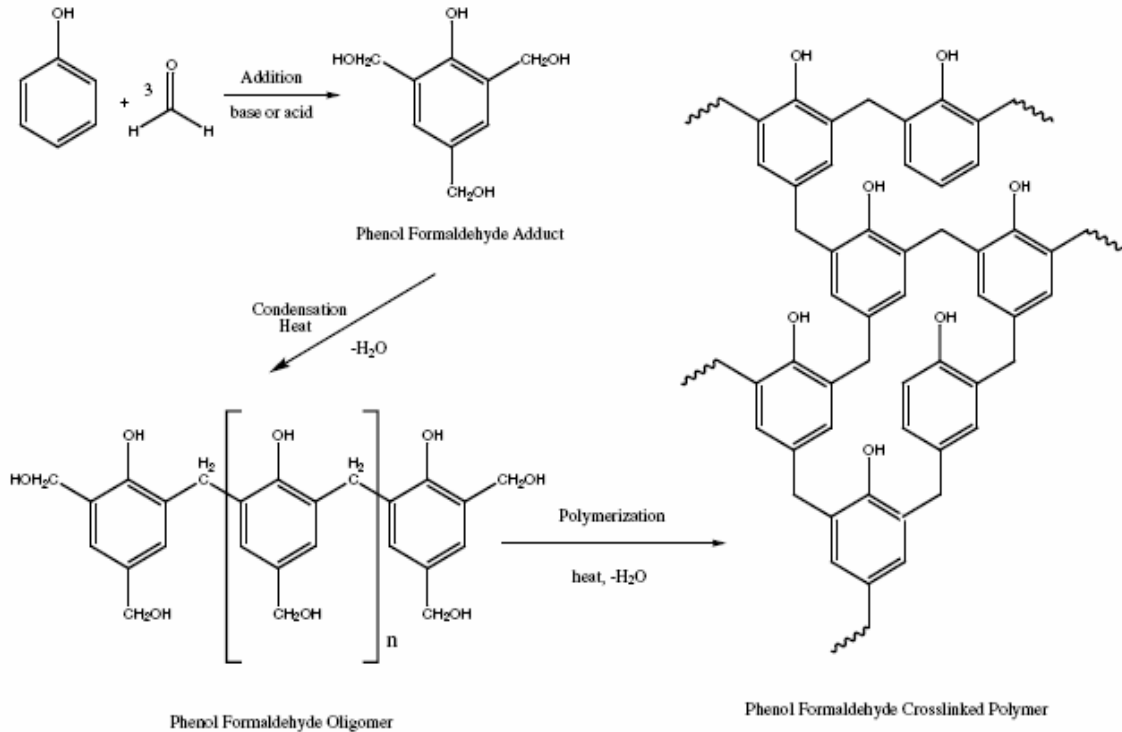


**Figure 5: Melamine-formaldehyde chemistry goes through similar steps as the urea-formaldehyde in Figure 3.**

Melamine resins are used primarily to improve the moisture resistance of urea-resin adhesives. In this respect, they are substantially more resistant than urea-resins but not as resistant as phenol and resorcinol resins. Melamine resins must be cured at temperatures of at least 240° F for most applications. They are also quite expensive relative to the urea resins. These two factors have limited the use of straight melamine-resins to a few special applications such as marine plywood where the need for a light-colored water-resistant adhesive justifies their cost.<sup>2</sup>

- Phenol-Formaldehyde (PF):** Phenolic resins are poly-condensation products of phenol and aldehyde particularly formaldehyde. Phenolic resins are the first true synthetic polymers to be developed commercially. In initial attack, poly-functional phenols may react with formaldehyde in both the ortho and para positions of the hydroxyl group. The second stage of the reaction involves the reaction of methylol groups with other available phenol or methylol phenol, (fig.6) leading first to the formation of linear polymers and then to the formation of hard-cured highly branched structures.<sup>48</sup>

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**Figure 6: Phenol-formaldehyde chemistry involves first formation of the hydroxymethyl group, followed by partial polymerization to the oligomer that makes up the adhesive. After applying adhesive to the substrate the polymerization is completed to form a crosslinked polymer network.**

- PF-resins are classified into two groups: resols and novolaks. Resols are made by base catalysis (caustic, amine) with an excess of formaldehyde cutting off the reaction at a certain condensation degree where PF resins are still liquid or soluble. The curing is done by heating and/or addition of catalysts. In the case of novolaks the poly-condensation is brought to completion. The molecular growth is limited by the low molar ratio F/P (<1). Novolaks can be cross-linked by adding of curing agents such as formaldehyde and hexamethylenetetramine. The difference between acid and base catalyzed process is in the rate of aldehyde attack on the phenol, in the subsequent condensation of the phenolic alcohols and to some extent in the nature of the condensation reaction. With acid catalysis, phenolic alcohol formation is relatively slow. Therefore, this is the step that determination the rate of the total reaction. The condensation of phenolic alcohols and phenols forming compounds of the dihydroxydiphenylmethane type is, instead, rapid. The latter are therefore predominant intermediate in novolak resins. The application field of PF-resins is wide spread. Phenol-formaldehyde resins are widely used to produce softwood plywood for severe service conditions. These resins are dark reddish in color and are available as liquids and powders or in film form. Their use is almost mandatory in plywood to be used in severe service conditions. Most types used in the United States are alkaline-catalyzed. Acid catalyzed systems are also available, primarily for use at curing temperatures of 70° to 140° F, but are used little in the United States. Principal limitation is the possible damage to wood by the acid catalyst.<sup>48,49</sup>

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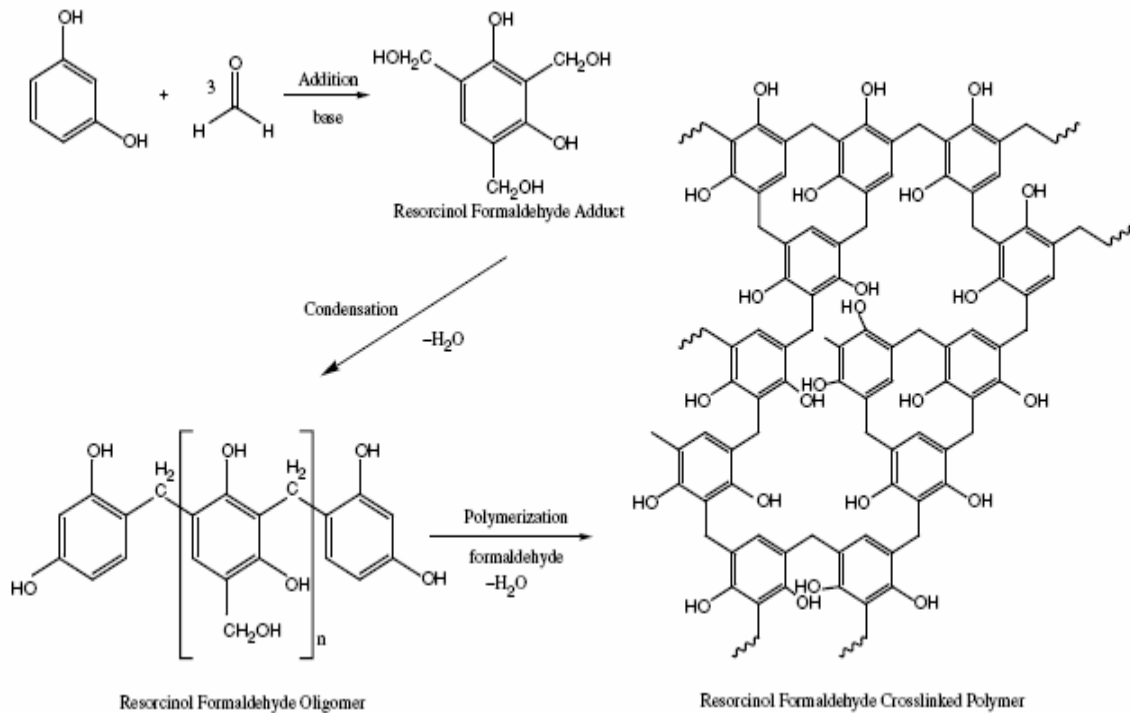
- *Resorcinol Formaldehyde (RF) and Phenol-Resorcinol Formaldehyde (PRF):* Resorcinol is chemical species that gives cold-setting behavior characteristic to resorcinol adhesives. It gives accelerated and improved cross linking, at ambient temperature and on addition of hardener, not only to the resorcinol-formaldehyde resin but to the PF resins onto which resorcinol been grafted by chemical reaction during resin manufacture. They produce bonds not only of high strength but also of outstanding water and weather resistance when exposed to many climatic conditions. PRF resins are prepared primarily by grafting resorcinol onto the active methylol groups of the low-condensation resols obtained by the reaction of phenol with formaldehyde.

The same chemical mechanisms and driving forces presented for PF resins apply to resorcinol formaldehyde resins. Resorcinol reacts readily with formaldehyde to produce resins, which harden at ambient temperatures if formaldehyde is added. The initial condensation reaction, in which liquid resins are formed, leads to the formation of only linear condensates when the resorcinol/ formaldehyde molar ratio is approximately 1:1. This reflects the reactivity of the two reactive sites (4- and 6- position) of resorcinol. The difference in reactivity of the two types of sites (4- or 6- relative to 2- position) is 10:1. Therefore, linear components always appear to form in preference to branched components. The reaction rate of resorcinol with formaldehyde is dependent on the molar ratio of the two components, the concentration of solution, the PH, the temperature, the presence of various catalysts and the amount of certain types of alcohol present. Methanol and ethanol slow the rate of reaction. The retarding effect on the reaction is due to temporary formation of hemiformals between the alcohols and the formaldehyde. This reduces the reaction rate because lower concentration of available formaldehyde.<sup>50</sup>

PRF adhesives are generally prepared by reaction of phenol with formaldehyde to form a polymer that has been proved to be in the form of linear structure. Then in the reaction, the resorcinol chemical is added in excess, in a suitable manner, to PF to react with the  $-CH_2OH$  groups to form RPF polymer with high molecular weight network structure.<sup>50</sup>

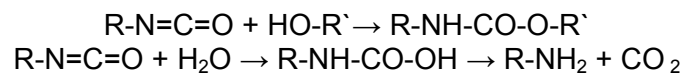
Like the phenol-formaldehyde resins, these adhesives form very durable bonds. They are resistant to both bond failure and to degradation. The main drawback to resorcinol adhesives has been the cost of the resorcinol. To lower the cost, but to maintain the room temperature curing properties, phenol-resorcinol-formaldehyde (PRF) adhesives were developed. Because of their cost, resorcinol based adhesives are not widely used for plywood manufacture; rather, they are used as assembly glues in solid wood products which must resist exposure to the weather and to water such as glued laminated wood beams. They have been of particular value in a few unique exacting applications such as the manufacture of wood aircraft.<sup>51</sup>

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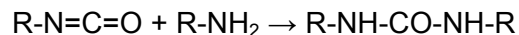


**Figure 7: Resorcinol-formaldehyde chemistry is similar to the phenol-formaldehyde but the reaction rates are fast enough that heat does not need to be applied.**

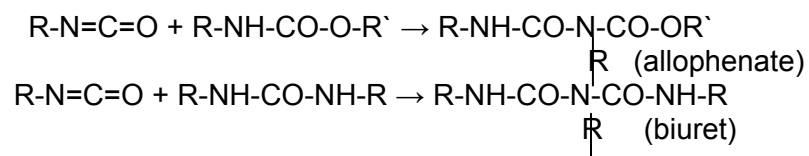
- *Isocyanates*: The isocyanate group reacts with any hydroxyl group to form a urethane bridge. The reaction between isocyanate and hydroxyls extends to water, which the isocyanate group reacts readily with the liberation of carbon dioxide and simultaneous formation of substituted urea groups.<sup>52</sup>



As primary and secondary amines are the other favorite group with which isocyanate react, the amine formed by the reaction above reacts immediately with additional isocyanate to form a substituted urea as follows:

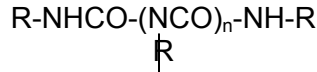


The secondary amine groups present in the urethane bridge and in the substituted urea formed react further with available isocyanate groups to continue cross-linking and hardening of the material by the formation of allophenate and biuret bridges:



## Development of Thick MPB Strand Based Wood Composites

As to reach R belongs at least two –NCO groups, a hard, cross-linked network is formed rapidly.

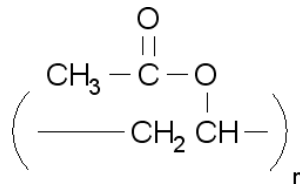


Isocyanate based adhesives were first used in the 1940's, but their high cost, along with technical difficulties and associated health hazards, largely prevented their commercial application. Technical improvements (copolymerization), along with the demand for board products which are totally free of formaldehyde emissions, subsequently lead to the use of isocyanate binders for particleboard manufacture. Many numbers of co-polymer of polymeric diisocyanates (MDIs) with a variety of other resins exist to yield thermosetting wood adhesive with excellent performance. These copolymers decrease cost, toxicity, ease of handling the resins and use for plywood. Some of these diisocyanate copolymerized resins are MDI-PF, MDI-MF and MDI-UF. They have excellent resistance to moisture and hence are well-suited for exterior applications.<sup>52,53</sup>

In addition to wood, isocyanates may also be used to bond agricultural cellulosic. The basic bonding mechanism consists of forming urethane bridges with the hydroxyl groups of the cellulose. This results in an extremely strong wood to adhesive bond, which is resistant to moisture. Isocyanates are also used to assemble glues, but the cost of the adhesive limits their use at the present time.

### ***Thermoplastic Adhesives***

- *Polyvinyl Acetates (PVAc)*: The synthesis and patenting of vinyl acetate monomer by Dr Fritz Klatter in 1913 (fig. 8), in Germany, provided the foundation for many valuable and now essential plastic products. He found that the catalyzed reaction of acetylene with acetic acid gave a polymerized low boiling liquid (vinyl acetate) to yield a potential range of dense solid materials. These are now often denoted as PVAc polymers. Klatter found that PVAc was compatible with other polymers and plasticizers which could give valuable adhesives and coatings for cellulose and textile products.<sup>54</sup>



**Fig 8: Polyvinyl acetate (PVAc)**

Polyvinyl acetate (PVAc) is an important type of industrial glue. Also in the wood working industry the direction is toward industrial use of glued materials and construction instead of nailing or screw joints. The most rapid development is taking place in the building product area, where a more efficient joining technique is needed. The use of glued-laminated timber (glulam), particle, and fiberboards (MDF, OSB) is becoming increasingly popular. Wood is also extensively used as curved laminated veneer in furniture. The properties and durability of adhesives exposed to different climates are becoming more and more important. However, polyvinyl acetate is nonresistant to moisture and if such adhesive joints are exploited in moist environment its strength substantially decreases. Sufficiently moisture resistant adhesive joints are obtained by modifying PVAc dispersion

## Development of Thick MPB Strand Based Wood Composites

with special compounds characterized by high reactivity. Such monomers have chemically active groups with the aid of which spatial structures of molecules are formed. Polyvinyl alcohol is known as having high reactivity with hydroxylic groups, including polyvinyl acetate, acting as netting agents. They are applied seeking to reduce the solubility of poly(vinyl alcohol), to change OH groups by hydro-phobic ones, to make induce the netting of molecules. Other example, as modifying the dispersion additive is suggested monoaldehyde-formaldehyde. However, the drawback of this method lies in free formaldehyde in isolation of a poisonous substance from adhesive joints. There is another method to modify PVAc dispersion by dialdehyde – glyoxal, which is an expensive product. Other substances to modify PVAc dispersion are known as well, such as isoprophylene alcohol, iron trichloride, potassium bichromate, butyl acrylate, methyl methacrylate, alkoxysilane, polyisocyanate.<sup>55,56</sup>

- *Hot Melt Adhesives*: Hot melt adhesives (HMA) are solvent-free chemical compounds that are used to join materials. They can be repeatedly softened by heat and hardened by cooling which is property of thermoplastic polymers. This allows parts to be removed or repositioned during assembly. Most HMA solidify at temperatures below 180°F and become low viscosity fluids at temperatures above 180°F. They form strong bonds, set rapidly upon cooling, and are relatively easy to handle. In general, [hot melt](#) adhesives are more resistant to water, moisture, and humidity. HMAs mostly contains acetals, acrylics, elastomers, natural or synthetic rubbers, epoxy resins, water-based resins, wax-based binders and styrene compounds or vinyl systems. Others contain polymer substances such as ethylene copolymers, styrene copolymers, polyamides, thermoplastic polyesters, vinyl esters, or polyethylene (PE), polypropylene (PP) and polyolefin. Filled or reinforced compounds resins, fillers, pigments, or chopped fibers are also used to modify thermal and mechanical properties of HMAs.<sup>57,58</sup>

One of HMA thermoplastic polymer is poly (ethylene- co-vinyl acetate) (EVA) which is formulated with four main components: polymer, tackifier, wax and antioxidant. The polymer contributes strength and toughness, while tackifier enhances wetting and tack. The wax lowers the melt viscosity and reduces cost. The antioxidant reduces thermal degradation during processing. EVA-based HMAs are widely used in packaging, paper laminating, nonwoven textiles, and book bindings.<sup>57</sup>

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### **Pressing model of thick composites**

#### ➤ State of the Art

Creep, the time-dependent increase of deformation of material under a constant stress, is an important material characteristic because it sometimes leads to structural failure as either excessive deformation or worse as collapse. As creep can lead to collapse, load duration is another important lumber characteristic. This failure or rupture under long-term loads is termed creep-rupture (Laufenberg, 1988), which is used to describe the combined phenomena of duration-of-load (DOL) and creep. There are three basic stages in the creep-rupture process: primary, secondary and tertiary creep as distinguished by the decreasing, constant, and increasing rates of deformation. These stages occur in many materials including wood-based composites (Nielsen, 1972).

Creep-rupture is a continuous deformation phenomenon which may end in catastrophic failure of the material under long-term stress. However, past experience has treated this topic as an uncoupled problem in terms of modeling, and has provided two basic behaviors to model: deformation and time-to-failure (load duration).

The purpose of this project is to provide insight into the load-duration phenomenon of strand-based wood composite by incorporating creep behavior study into it in an attempt to develop new models to predict creep-rupture behavior in a coupled way. The new models should also be compared with the new ASTM Standard Specification for Evaluation of Duration of Load and Creep Effects of Wood and Wood-Based Products (ASTM D6815). Therefore, literature review in both fields of duration-of-load and creep is performed with the aim of establishing a unified approach to deal with both phenomena.

In terms of DOL modeling, the application of damage-accumulation models and linear viscoelastic fracture mechanics are well-established tools for the prediction of DOL. The damage accumulation we are concerned with stems from material behavior at the

## Development of Thick MPB Strand Based Wood Composites

microscopic level. Because knowledge of behavior at this level is generally incomplete, it is difficult to postulate corresponding models for damage accumulation, based on physical laws. As an alternative, damage accumulation models have been proposed which are phenomenological in nature, that is, they are based on our correct understanding of the phenomena at the macroscopic level and upon experimental data. Several types of damage accumulation models have been recorded, of which the most important are: 1) the model derived from the hyperbolic Madison Curve (Wood 1951), 2) Barrett and Foschi model I and II (Barrett and Foschi, 1978), 3) Foschi and Yao model (Foschi and Yao, 1986; Yao, 1987), and 4) Gerhards model (Gerhards, 1979; Gerhards and Link, 1987).

Another promising approach employs fracture mechanics to study the process of slow crack growth through timber (Johns and Madsen, 1982; Nielsen, 1986, 2000, 2005; Sorensen, J.D. and Svensson, S., 2005). Pitched at the microscopic level, this approach provides some complementary information on cause and effect in the failure process. Also note that the strain energy approach proposed by Fridley (1992) provides a new method to study the load-duration effects in structural lumber.

For many structural applications the most important mechanical property of wood is its resistance to deflection, including elastic and creep deflection. Creep includes three distinct types of behavior, which are difficult to separate because they can all operate simultaneously. These are time-dependent (viscoelastic) creep, mechano-sorptive (moisture-change) creep, and the pseudo-creep and recovery that have been ascribed to differential swelling and shrinkage (Hunt, 1999). Emphasis will be put on the former two types of creep.

Much work has been done for the mathematical modeling of viscoelastic creep under constant moisture content and temperature. Some of the models are purely empirical expressions, such as power law, and exponential functions etc. Alternatively, the creep can be interpreted with the aid of rheological models, such as Maxwell unit, Kelvin unit, and Burger model. Also some nonlinear viscoelastic models are suggested by introducing nonlinear functions into the linear viscoelastic equation.

In terms of creep modeling under changing moisture content, many attempts have been made since the late 1970s to develop a model for mechano-sorptive behavior, the deformation which is directly related to the interaction of change in moisture content and mechanical stressing. Especially the researchers of Finland, Sweden, France and UK have done a lot of work on this subject. A few of these models have been explanatory or descriptive in nature. They seek relationships at the molecule, ultrastructural or microscopical levels. But most of them have been either purely mathematical, with the aim of producing a generalized constitutive equation, or partly mathematical, where the derived equation is linked to some physical phenomenon, or change in structure of the timber under stress (Hanhijarvi, 1995).

### ➤ User interface

Based on previously developed models at UBC under NSERC Strategic Grant (Steiner, Avramidis and Lam) and NSERC Collaborative Grant (Lam), a user friendly software has been put together to enable simulation an prediction of the heat and mass transfer and densification of strand based wood composites during and after processing. Within this framework, the approach that was used combines a forming of structure model (A.

## Development of Thick MPB Strand Based Wood Composites

Oudjehane), a 3D model on heat and mass transfer (P. Garcia) modified in order to account for the manufacturing parameters, and a consolidation model (A. Oudjehane).

**Figure 9. User interface of the UBC wood strand composites processing simulator**

The screenshot shows the user interface for the 'Modeling simulator of strand based wood composites Manufacturing processes'. It is organized into several sections:

- End product properties:** Includes input fields for Length in MM, Width in MM, Thickness in MM, and Oven dry density in KG/M3.
- Strands properties:** Includes input fields for Length in MM, Width in MM, Thickness in MM, and Oven dry density in KG/M3.
- Forming process conditions:** A table with columns for Length MM, Width MM, Thickness MM, Density KG/M3, Volume fraction %, Forming angle, and Moisture Content %. Rows are provided for Top face layer, Core layer, and Bottom face layer.
- Hot pressing conditions:** Includes input fields for Total press time (seconds), Press closing time (seconds), Press opening (seconds), Press temperature (Celsius), Initial temperature (Celsius), and Resin content (%).
- Control Buttons:** On the right side, there are buttons for 'Form the wood composite mat', 'Hot press the wood composite mat', 'Consolidation process', 'Save into userin.tbl', 'Close', 'Hot pressing f(time)', and 'Density distribution'.

The user interface (Fig. 9) was developed to account for parameters used in most process control of OSB mills. While the user interface is flexible to accommodate for simulation of MPB strand based wood composites, we anticipate proceeding with further changes to the models as we incorporate more parameters retaining to findings from both the adhesives and permeability studies. Further validation to have these models applicable to MPB strand based wood composites will be undertaken in the second year of this project.

An extended summary of this original approach will be submitted for publication.

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### **Design small humidity chamber and permeability test jig**

#### ➤ State of the Art

The work on the permeability of strand based composites reported in the literature has focused on the transverse or through the thickness permeability. This is due to the ease with which these measurements can be made using existing equipment for solid wood. For measurement of permeability in the radial or tangential direction, a thin disk of solid wood is cut so that the plane of the disk corresponds to either the tangential-longitudinal or the radial-longitudinal plane, respectively. The pressure drop as gas flows through the sample thickness is measured and the permeability of the section computed as described by Siau (1984, 1995). The permeability in these directions is typically a few orders of magnitude lower than in the longitudinal directions, and thus thin samples, often only a few millimeter thick, are sufficient for measurement. For the longitudinal direction the permeability is much higher and a longer, thicker sample is required. For solid wood these samples consist of a small cylinder of wood with the axis of the cylinder corresponding with the longitudinal direction. These samples are typically 10 to 15 centimeters in length and 1 or 2 centimeters in diameter. Due to the vertical density profile (VDP) of oriented strand board, and to a lesser extent of oriented strand lumber, the permeability of the sample changes by orders of magnitude moving from the face to the core layer. Thus, in-plane permeability measurements on samples with a significant VDP do not produce meaningful results.

Measurement of the transverse permeability for oriented strand board has been undertaken by Hood (2005) for boards consisting of strands only and by Fakhri (2005) for boards consisting of mixtures of strands and fines in the core layer. To date, only Hood (2005) has measured the in-plane permeability of the oriented strand mats but those samples contained neither resin nor fines. He made these samples by compressing oriented strand mats between platens made of plywood to different densities without resin, and measured the pressure drop of those samples. From this he was able to compute permeability values. In this work, we will measure the

## Development of Thick MPB Strand Based Wood Composites

permeability of randomly oriented resinated strand mats containing different ratios of stands to fines and pressed to different densities. The mats will then be cured by slow heating of the press to fully cure the resin and lock in the furnish particles in place. The permeability of these mats will then be measured using the new equipment to be built in the near future. The design of that equipment is discussed in the following section.

### ➤ Drawing design of a permeability test jig

The small humidity chamber, which is built based on the theory from Siau (1984, 1995), is available in the Wood Drying Lab at UBC and will be used for this work. In order to identify and debug any issues with connecting the permeability jig to the existing permeability apparatus, wooden prototypes of the jig have been made. The design of the jig is shown in Figure 10; the composite mat is placed between the top and bottom platens. Two pieces of plywood with sealant layer will be put on two opposite sides of the mat to prevent gas leakage on these sides during permeability measurement. This assembly is loosely clamped between the top and bottom jig platens. This permeability jig and mat is then placed between the press platens and the sample compressed to the required thickness in displacement control. The screws on either side of the jig are then tightened up such that the distance between the jig platens remains constant when the press is opened. Permeability  $k_l$  will then be determined by measuring the pressure drop across the sample and the permeability computed. The sealant on the edges of the samples will then be trimmed off and new sealant and plywood edges applied to the edges oriented 90° to the first edges, i.e., sample 2 in Figure 11. This process is repeated for the 45° direction by trimming these edges off and applying sealer again, sample 3 in Figure 11. The smaller size of sample 3 requires a smaller permeability jig and necessitated the construction of a second, smaller wooden prototype. The design including an interface between the jigs and the humidity chamber is in the last stages of being finalized and additional proof testing on the design is underway.

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Figure 10. The clamping of a sample in the permeability jig showing the direction of gas flow.

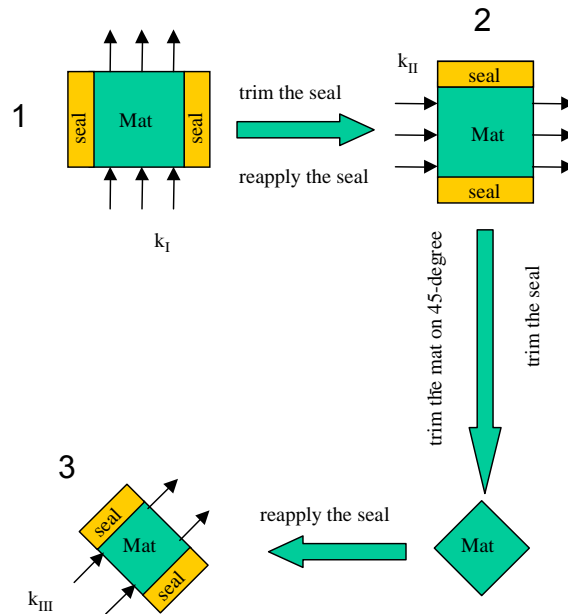
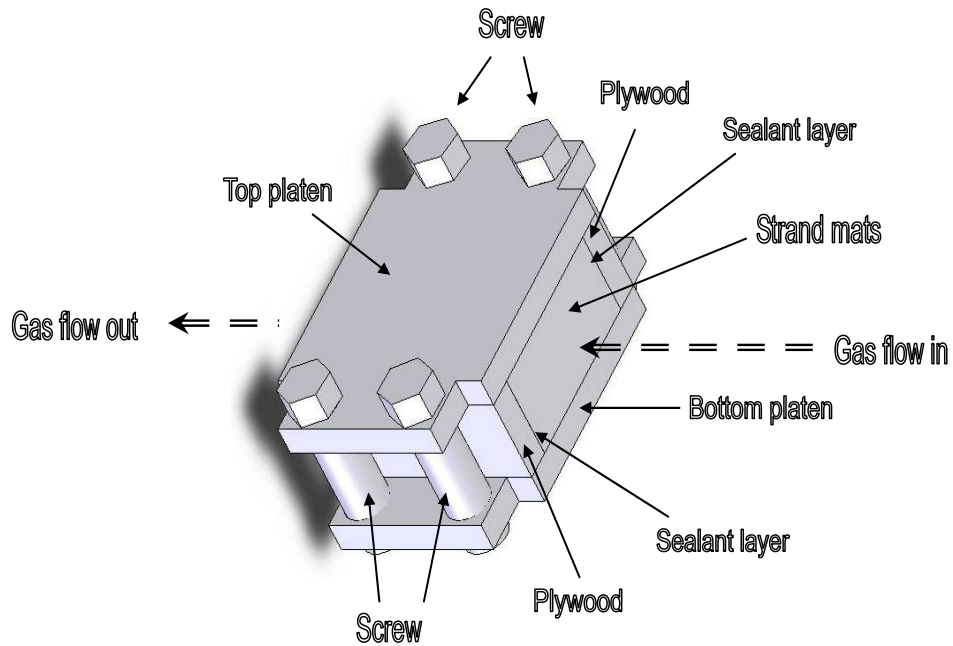


Figure 11. Successive sealing and cutting of the sample edges showing the direction of gas flow.



## FUTURE WORK

Year 2 of the proposed three year study will cover the followings:

### Fibre – Adhesive Interaction

The fibre-adhesive interaction of wood composites is one of the most important aspects of the successful development of new structural wood composites. Although there are many new adhesive formulations developed, their interaction with wood are perhaps least understood. This is particularly true with MPB wood where the chemistry of the lodgepole pine has been altered as a result of the beetle infestation. The development of bond strength between the wood and the adhesive is governed by the surface chemistry of the fibre. This is further influenced interactively by the temperature and moisture effects.

We will built a small humidity chamber unit for the UBC DMA equipment with feedback controls to provide both temperature (upto 90°C)and humidity control (upto 90% RH) during specimen testing. A series of experiments (small specimen lap shear and bending) will be initiated using the UBC DMA equipment considering the following variables: adhesive type, adhesive content, time-since-death of MPB wood, surface characteristics of the wood, temperature, relative humidity, temperature and relative humidity interaction. The specimens will be obtained from formed boards as well as pressed strand stacks). Experiments will be conducted to relate results of DMA to conventional IB and durability (boiling) tests. (One Ph.D. student and PDF 50% time)

### Pressing model of thick composites

The UBC 3-dimensional heat and mass transfer and consolidation model will be calibrated against test results. This UBC 3-dimensional heat and mass transfer and consolidation model has been advanced from year one of the study to consider variable step closure of the press and preheating scheme. A User Friendly interface has also been implemented in the software. A series of pressing studies with MPB furnish will be carried out to obtain information on internal gas pressure and temperature time history profile during pressing. Variables including time-since-tree death, press strategy, adhesive type, strand alignment, and fines contents will be considered. The database will be use to calibrate the key input variables in the models using a nonlinear error minimization procedure to match the time history response of the model against the test data. Experiments will also be conducted to quantify the performance of the pressed material (Research Associate AO 50% time, Research Engineer 50% time)

The performance of structural wood composites is not well understood and some consider this aspect as the Achilles heel of development of new structural wood composites. A new ASTM standard was recently developed to quantify the acceptance of new wood composites on the basis of a 3 month constant load test subjecting the specimens to a load level of 55% of the 5th percentile strength. This test checks both the strength and creep [creep rate and the relative creep (ratio of displacement after 3 months to displacement after 1 minute)] response of the material. The intention of this approach is to establish product performance equivalency compared to solid sawn wood products. It is a go/no-go process. This type of testing does not fully quantify the behaviour of structural wood composites especially strand based wood composites

## **Development of Thick MPB Strand Based Wood Composites**

where the strength to stiffness ratio tends to be high compared to lumber and other existing products. Further long term creep rupture response cannot be fully established from a 3 month test at relatively low load level.

A nonlinear visco-elastic model on the creep behaviour of oriented strand products will be initiated. A series of experiments will also be initiated to quantify the creep and creep-rupture behaviour of oriented strand products. The experiment under consideration will include the both constant load (reference) and low-cycle fatigue loading. These tests will offer data for model calibration and model verification. This database will be linked with the DMA based data in future. (Ph.D. Student JW, Research Engineer 50% time)

The gas flow of mat during pressing is one of the key parameters that control the development of gas pressure development during the pressing process. If excessive pressure is built up before adequate bond strength can be developed, blows can result. This is one of the critical issues facing the manufacturing of thick strand based material.

A test jig will be built to allow measurement of in plane permeability of gas flow of formed mat under out of plane pressure. Experiments will be conducted to measure in plane permeability of gas flow of formed mat under out of plane pressure. (M.Sc. Student)