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Soy Adhesives that Can Form Durable Bonds for Plywood, Laminated Wood Flooring, and Particleboard

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Abstract

Synthetic adhesives, including urea-formaldehyde (UF) and phenol-formaldehyde (PF), have generally replaced biobased adhesives over the past 70 years because of their durability, low cost, and ease of use. However, in the past few years, concern about formaldehyde emissions, cost, and interest in biobased materials have renewed interest in soy adhesives. The use of soy adhesives can be broken into four stages: soy flour selection, dispersing/denaturating conditions, cross-linking chemistry, and bonding conditions. Generally soy flour is used because of its low cost, but the adhesive properties of the soy depend upon flour type, as well as adhesive formulation and processing conditions. For the flour to be used as an adhesive, it must be dispersed in a solvent, usually water. In this paper, we emphasized protein properties, as they are critical for forming good durable bonds. The dispersed proteins are globular because proteins fold in water so that the outer surface contains mainly hydrophilic groups, whereas hydrophobic groups prefer to be on the inside. Globular structures are sensitive to conditions, such as pH, added denaturants, temperature, and salts. Typically, soy proteins provide good adhesion to wood and other materials; however, these adhesives have poor water resistance without chemical cross-linking. Denaturants not only open or swell the protein globules to increase adhesion to the wood surfaces but also expose more sites for cross-linking these proteins. Soy adhesives, like most adhesives, need to be tailored to the application. Thus, an adhesive for plywood is very different from that for particleboard, and a core adhesive is different from a face adhesive for wood composites.

Understanding these differences has led to soy-based products that provide commercially acceptable plywood, engineered wood flooring, particleboard, and oriented strandboard.

Keywords **S**oy, adhesive, structure, cross-linking, denaturation, viscosity

Introduction

Until the 20th century, wood adhesives had been obtained from natural materials, such as hooves, hides, milk, and soybeans (Lambuth 2003). Although casein (milk protein) adhesives are more water-resistant, adhesives from defatted soybeans create more cost-effective interior non-structural wood products, such as plywood. However, in the early 20th century, researchers found that urea–formaldehyde adhesives made superior interior products compared to bio-based ones, and phenolics made excellent exterior products. Durability and favorable economics, driven by the expansion of the petrochemical industry, led to the expansion of reconstituted wood products using synthetic adhesives into a wide variety of building construction materials and interior wood products to replace solid wood (Wescott et al. 2010).

Although there has been a desire to make greener adhesives, commercialization of soy adhesives was limited until suitable technology and market drivers existed. The innovative work of Li led to the use of the cross-linker polyamidoamine–epichlorohydrin (PAE) resin for providing acceptable water resistance for interior wood products bonded with soy adhesives (Li et al. 2004, Li 2007). The market driver is lower formaldehyde emission standards set by the California Air Resources Board (ATCM 2009). The technology for soy adhesives using PAE cross-linking has provided adhesives that are used in plywood and engineered wood flooring (Allen et al. 2010), as well as particleboard and medium density fiberboard (Wescott et al. 2010). Knowing more about the protein structures and soy flour composition will lead to understanding this technology and where opportunities lie for even better products.

Protein Structure and Soy Flour

Although soy flour contains carbohydrates as well as proteins, proteins are most likely the key adhesive components. Proteins are very different from most other adhesives in structure–property relationships.

For most adhesives, knowledge of the backbone structure sequence leads to good predictability of adhesive properties, but this is currently not true for proteins. While most adhesives are made from one or a few monomers, proteins are made from 20 different amino acids with different ratios and sequence for each protein. Although the main backbone structure is always a 2-aminoacetic acid, the side chains offer a wide variety of functional groups being aliphatic or polar (hydroxyl, thiol, carboxylic, and a number of nitrogen-containing compounds). In Figure 1, the hierarchical levels of protein structure are shown schematically. Proteins are linear polymers with each type of protein having a defined sequence that is encoded in the DNA of the organism. Thus, this primary sequence is critical to the folding of the protein and its ultimate function within the organism. The sequence effect can also be true in some synthetic polymers. For example, a random styrene–butadiene polymer behaves much differently than one containing blocks of the two components.

The next level involves the formation of crystallites, which are mainly intrachain in proteins but are usually interchain in synthetic polymers. Although most synthetic adhesives have little crystallinity, it can certainly be important for protein adhesives. Proteins not only have the sheet type of crystallinity common in synthetic polymers, but proteins also have helices. Specific amino sequences are needed to form these β-sheets and α-helices, as opposed to random coils.

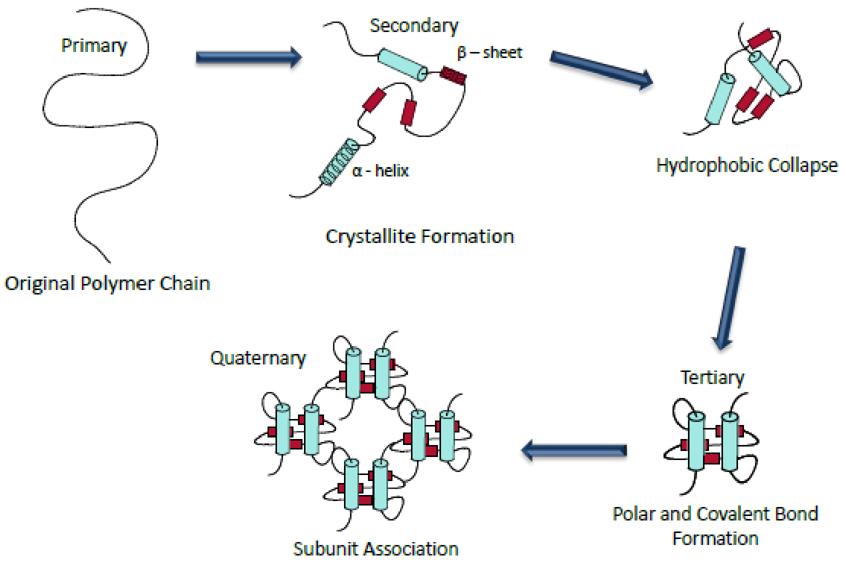

FIGURE 1 – Protein structure formation involves the primary structure of the amino acid sequence, secondary structure including α-helices and β-sheets, tertiary structure created by hydrophobic collapse and internal bonds, and quaternary structure involving association of protein molecules.

Whereas most synthetic polymers are usually in a highly entangled form allowing for high interchain interactions, an individual protein chain folds up on itself in aqueous environments to provide a tertiary structure. The first aspect is hydrophobic collapse caused by the non-polar side chains to minimize their interaction with the aqueous environment. The other aspect is interchain interactions, such as disulfide groups from thiols, acid-base interactions, salt bridges with multivalent cations, and hydrogen bonds. The small energy differences between native and denatured (non-native) structures means that changes of the aqueous environment by adding salts, other organics, or heating can cause significant changes in the tertiary structure of proteins. While the first three structural levels are intrachain processes to form globules, often these globules interact with one another to form the quaternary structure. These interactions are usually due to hydrophobic, hydrogen bond, salt, and disulfide formation. The high degree of internal chain structure makes proteins behave very differently from other polymers. Certainly not all the potentially reactive groups are on the surface. Often bonds in the interior tie up thiol groups as disulfide bridges, other polar groups such as hydrogen bonds and acids, and bases as salt bridges.

The denaturation, illustrated in Figure 2, is often the reverse of the folding process, but not necessarily the same exact steps. The challenge involves breaking apart the quaternary aggregates and opening the tertiary structure to provide more reactive sites without destroying too much of the secondary structure that contributes to adhesive strength. With pure proteins, methods are available to determine changes in structure, but with a mixture of materials, such as those found in soy flour, it is harder to determine much about the structural changes other than loss of secondary structure.

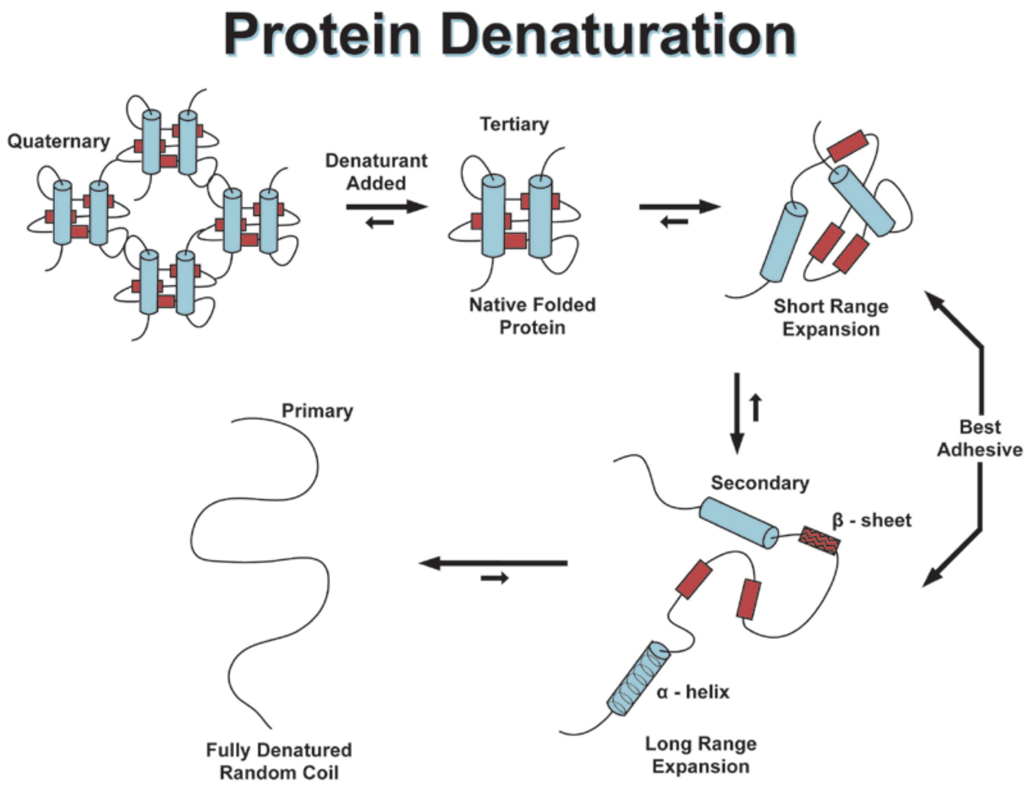

FIGURE 2 – Protein denaturation can involve alteration of the quaternary, tertiary, and secondary structure, but it is best to preserve some of these structures to have good strength.

Soy flour is a mixture of polymers that make it difficult to understand what changes are taking place. The flour is about half protein, a quarter insoluble carbohydrates, and a quarter soluble carbohydrates. Insoluble carbohydrates may play a minor role in strengthening the adhesive product. In contrast, soluble carbohydrates are more of a detriment in that they can increase dispersion viscosity, consume some of the cross-linker, and increase water absorption, leading to a softening of the adhesive under high moisture conditions.

Even the protein fraction is a mixture of different proteins, making it difficult to understand observed performance of the product. Although some enzymes are among the proteins, most proteins are for energy storage. The two main proteins are glycinin (in the 11S fraction) and conglycinin (in the 7S fraction) (Sun 2005, Utsumi et al. 1997). These two proteins are very different in their structures. In contrast, glycinin is made up of six acidic and six basic protein subunits that alternate in two structural rings and are partially joined by disulfide bridges. Conglycinin consists of more neutral aminoacids with few thiol groups for disulfide bridges. These protein subunits are held together by hydrophilic forces. Because of these complexities, most studies have utilized protein isolates, and 7S or 11S fractions (Sun 2005). Although these studies can be very helpful, they also suffer the limitation that the isolation process can denature proteins and eliminate the interaction of components that may alter the protein's properties (Sun 2005, Hunt et al. 2010).

Materials and Methods

Materials

Soy flours were obtained from Cargill (Cedar Rapids, IA) or Archer Daniels Midland (Decatur, IL), the polyamidoamine-epichlorohydrin (PAE) was obtained from Ashland Hercules Water Technologies (Wilmington, DE).

Methods

The Automated Bonding Evaluation System (ABES) Model 311c manufactured by Adhesive Evaluation Systems Incorporated (Corvallis, OR) was used to make small bonded samples to test the adhesives. Maple veneers were cut into 114.3- (grain direction) by 19.1-mm strips, with all wood conditioned at 120° C for 2 minutes. The bonded specimens were assembled by spreading adhesive to one side of a 5-mm bonding overlap. This overlap was maintained by clipping two wood pieces together with a binder clip while placing them in the ABES unit. Each specimen was pressed by the unit at 2.0 bar and at 120° C for 2 minutes. Each specimen was removed and allowed to cool and recondition overnight at 22° C and 50% relative humidity. Of the 14 specimens using each adhesive, half were pulled until bond fracture at ambient conditions and half were tested after a 4-hour water soak.

Rheological tests were run using a controlled-stress rheometer (Paar Physica UDS 200, Physica Messtechnik, Stuttgart, Germany) with a 25-mm serrated parallel plate.

For the plywood and engineered wood flooring research see the details given in Allen et al. 2010, for particleboard see Wescott et al. 2010, and for oriented strandboard see Wescott and Frihart 2008 and Wescott et al. 2006a and 2006b.

Results and Discussion

The original soy flour adhesives of the early 20th century were made by dispersing soy flour in caustic and adding chemicals to develop some cross-linking (Lambuth 2003). Caustic does denature proteins and if the temperature is not above 100° C, chain scission is minimized. We have generally seen much better performance if the protein chain is not fractured (unpublished results). A large increase in viscosity is observed and most likely is due to opening up the proteins to create greater interactions between chains. This high viscosity stage is only temporary and decreases over time as the proteins refold or hydrolyze. However, this open protein structure gives good products when reacted with phenol and formaldehyde (Wescott et al. 2006a). Like most phenolics, these products had greater viscosity stability than soy-caustic products, but were not completely stable. However, acidification of these alkaline dispersions led to much more stable soy dispersions (Wescott and Frihart 2008, Wescott et al. 2006b). Thus, more neutral soy-phenolics not only reduced further polymerization of the phenolic, but also generated a stable protein structure. This adhesive was also light in color and did not give caustic burns in the wood. The literature indicates that many other compounds can denature soy (Sun 2005), but they fail to give us adhesive bonds with good water resistance. Compounds, such as urea, guanidinium hydrochloride, and various surfactants, have been reported to denature the protein as a way to improve adhesion (Sun 2005), but we have not seen acceptable water-resistance (see Figure 3) on our small-scale veneer adhesion testing (Frihart et al. 2009).

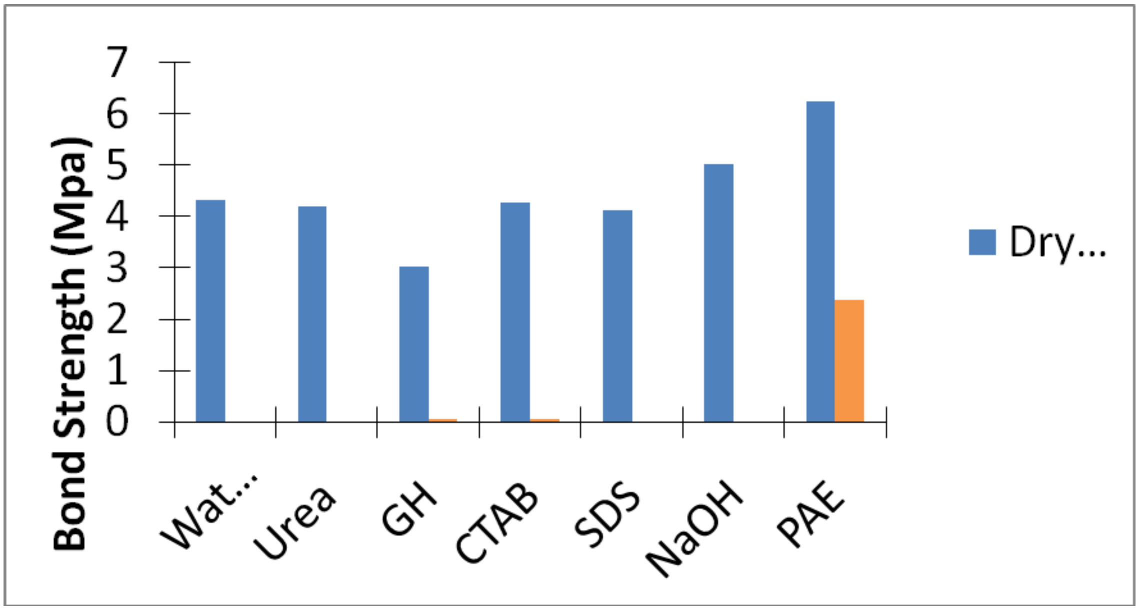

FIGURE 3 – *Adhesive strength versus denaturants and a cross-linker with soy flour where GH represents guanidinium hydrochloride, CTAB is cethyltrimethylammonium bromide, SDS is sodium dodecyl sulfate, and PAE is polyamidoamine-epichlorohydrin resin.*

Although none of the dispersion methods on their own gave good wet strength (Fig. 3), the polyamidoamine-epichlorohydrin (PAE) resin is an effective cross-linker for soy proteins. This innovative technology (Li 2007, Li et al. 2003) has been the main method used to develop good water resistance for soy–wood bonding applications (Zhong et al. 2007, Allen et al. 2010, Wescott et al. 2010). Use of a PAE resin with soy also results in an improvement in the dry adhesive properties. The PAE provides products that meet the no-added formaldehyde (NAF) regulations (Williams 2010) and green building standards (Cribb 2010).

Although the soy flour, water, and PAE resin combination can provide useful adhesives for wood bonding, these formulations are not the most effective for commercial production. Denaturants and viscosity modifiers have been shown to be very effective for making soy adhesives. Although the original caustic denaturant was useful for older soy adhesives, it does not work well with PAE due to poor pot life. Thus, other dispersants were needed to denature the soy by opening the structure for greater interaction with the wood and the cross-linker. Urea is an effective denaturant for soy proteins (Wescott and Birkeland 2008).

Studying the use of denaturants and optimization of soy flour PAE systems have been carried out on an empirical basis for the following reasons:

- Isolating proteins often disrupts the native structure, especially when using soy protein isolates. Heat denaturation can cause interaction of 7S and 11S protein subunits. Thus the denaturant effect with soy flour may be different from that observed with protein fractions.

- The non-protein components of soy flour can influence the curing reaction with the urea denaturant (Hunt et al. 2010).

- Organic solvents and inorganic salts are known to alter the structure of proteins (Boye et al. 1997). The mechanism of the very common denaturant urea with pure proteins is still being debated (Canchi et al. 2010).

In making soy adhesives, several aspects of soy protein need to be considered. There are several types of soy flour due to different treatments after defatting (removal of soy oil). The most native in structure are those characterized by a high protein dispersibility index (PDI). Usually the high end is with 90% (90 PDI) of the protein staying in the supernatant after vigorous mixing and centrifugation. Greater heat exposure reduces the PDI by denaturing the protein, making it less dispersible but also improving its use for some food (human and animal) applications. Both low (20) and high (90) PDI give good adhesive bonds with wood using PAE curing (unpublished results). However, this does not mean that low and high PDIs behave the same in formulating adhesives. Different PDIs can respond differently to denaturant and viscosity modifiers. However, all soy protein dispersions are shear thinning. Shear thinning is a very important difference between soy-based and phenolic and amino resin adhesives that are normally Newtonian in rheological measurements. Viscosity of adhesives is measured at low shear rates, but commercial adhesive application involves high shear rates. Thus, a soy adhesive with a high apparent shear rate can behave as well as a synthetic adhesive where shear rate does not affect viscosity.

This shear thinning behavior is an important aspect of soy adhesives. Shear thinning indicates that soy particles have weak physical and chemical bonds, such as by hydrophobic and dipolar bonds, for each other that can be broken by agitation. After a short time of no agitation, reassociation of these interactions takes place. These associations are an important property of soy proteins, whether the dispersion uses low, medium, or high PDI flour, concentrate, or isolate. The parallel lines for shear thinning in Figure 4 show that changes in formulations can affect viscosity, but does not necessarily alter the shear thinning behavior. Many researchers have mistakenly assumed that soy formulations need to be of similar apparent Brookfield viscosity as synthetic adhesives to fit with current processing technology in wood composites manufacturing. To do this, researchers have chemically or enzymatically hydrolyzed soy proteins, but this can destroy the secondary structure and makes it much harder to make a cohesively strong adhesive. Specially designed polypeptides can provide good bonds (Mo et al. 2008), but this is not true of fragmented proteins. Research has shown that protein dispersions with high apparent Brookfield viscosities can work in adhesive application equipment used for manufacture of plywood, engineered wood flooring, particleboard, fiberboard, or oriented strandboard (Allen et al. 2010, Wescott et al. 2006a, Wescott and Frihart 2008, Wescott et al. 2010). However, lower viscosity is valuable in certain applications; for example, spraying adhesives.

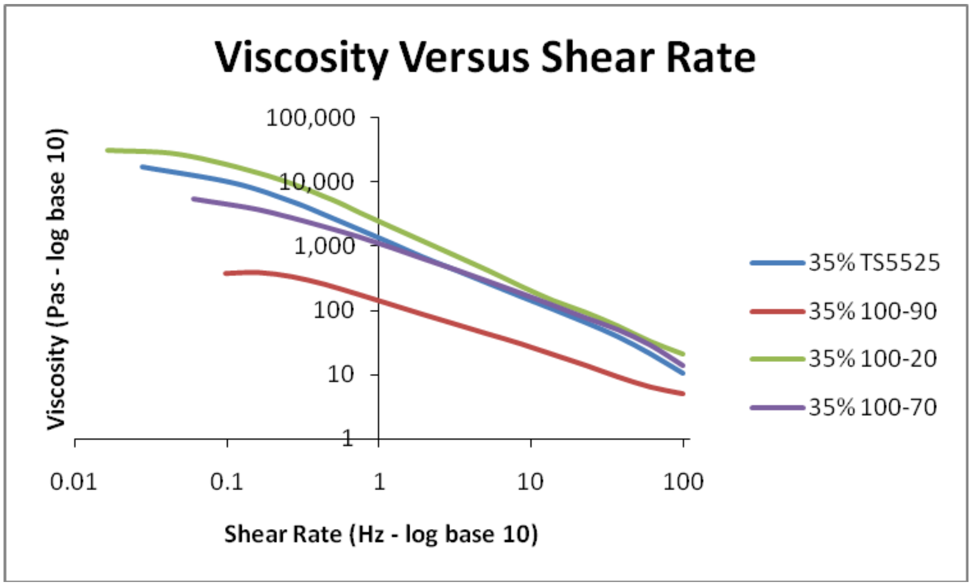

FIGURE 4 – *Viscosity as a function of shear rate for aqueous soy flour mixtures (viscosity measured @ 25) demonstrates similar shear thinning characteristics for different soy flours mixed with water at 35% solids (ADM TS5525, Cargill Prolea 100-90, -70, and -20).*

Performance of soy adhesives can be altered by using a variety of additives and conditions. Although it seems reasonable that the most dispersable soy flour should make superior adhesives, a wide range of soy flours can be used with proper modification of the adhesive formula (unpublished results). The most common additives are denaturants, but they can also include co-solvents, like glycerin, or viscosity modifiers. The viscosity modifier is important in that specific modifiers can reduce apparent viscosity, but not hurt adhesive performance. This has been shown to be very useful in developing higher solids soy formulations (Allen 2010).

Soy flours can be useful for in-plant production, whereas most synthetic adhesives need to be made and shipped from adhesive manufacturers. The hazardous nature of reactants and the requirement for specialized equipment for making synthetic adhesive resins make them unsuitable for in-plant production. On the other hand, soy flour is already polymerized; the flour can be dispersed and mixed with cross-linking chemicals in many application plants. This allows the ability of the soy adhesive to be customized to the production process.
One main advantage of soy adhesives is their environmental acceptability. Given the desire in many countries to reduce formaldehyde emissions from wood products, the no added formaldehyde (NAF) nature of soy adhesives means they are more environmentally suitable. NAF means that any formaldehyde in production of the wood product and in the product itself comes from wood and not from the adhesive (Birkeland et al. 2010). In fact, products can be produced with lower formaldehyde emissions than from wood itself (Allen et al. 2010, Birkeland et al. 2010, Wescott et al. 2010). Lower formaldehyde emissions occur not only

under standard testing conditions, but also under elevated heat and humidity, which occur under common exposure conditions that the wood products may see (Frihart et al. 2010). Soy flour and PAE cross-linker involve less plant hazards than those adhesives containing formaldehyde or isocyanate. Renewable content of wood products comes much closer to being 100% derived from biomass sources with soy adhesives.

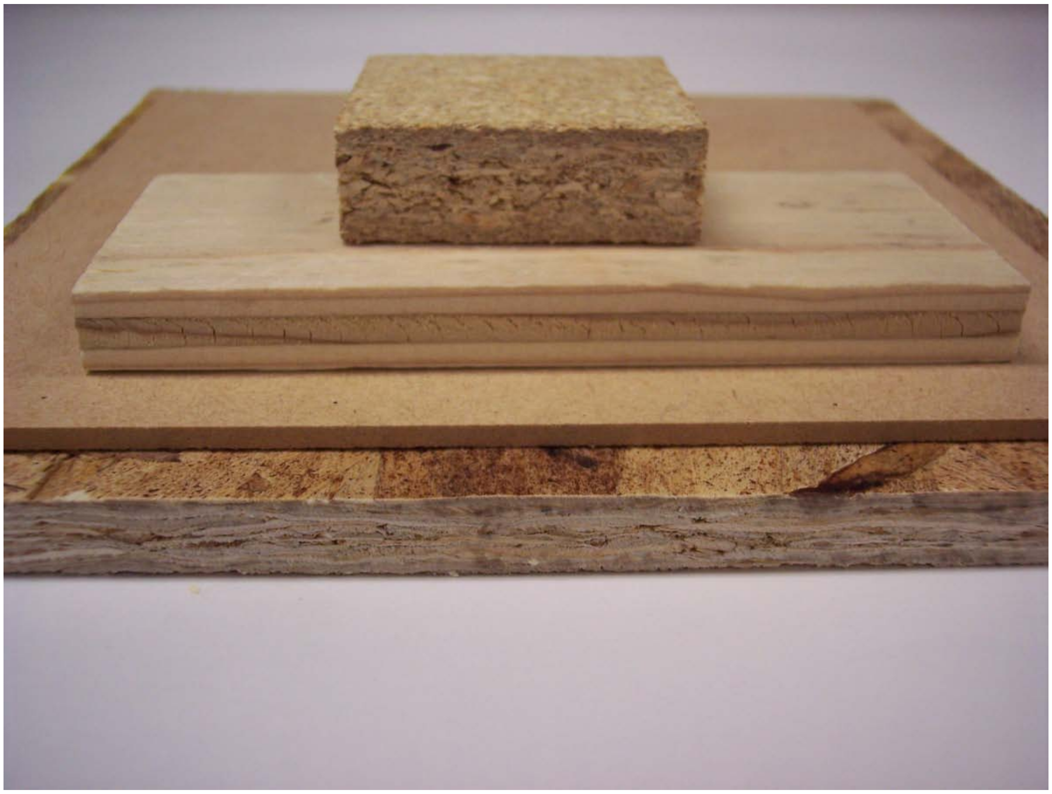

FIGURE 5 –*Soy adhesive can be used commercially to make a variety of wood products, from top to bottom particleboard, plywood, fiberboard, and strandboard.*

A wide variety of soy resins have been developed for different wood bonding applications (Fig. 5). To avoid overpenetration and fill gaps between wood surfaces, adhesives for veneer bonding need to be higher in viscosity than those used for spraying onto strands, particles, or fibers. Higher viscosity soy adhesives that work well commercially in interior or decorative plywood, and engineered wood flooring have been developed. In many ways, these plywood and flooring products are similar in their cross-ply construction. However, they are also different in that for decorative plywood, the face veneer is quite thin, requiring that the adhesive both provide a smooth, strong bond but not bleed through. In contrast, the engineered wood flooring has a thick face veneer with rougher surfaces. This has been accomplished by developing improved generations of soy-PAE adhesives as illustrated in Table 1 (Wescott et al. 2009). These adhesives are not limited to veneer bonding because the basic technology can be used to develop lower viscosity products as a binder resin for wood composites, such as particleboard and fiberboard. The requirements for these products are quite different than for veneer bonding (Wescott et al. 2010). However, soy can be modified or blended with other resins to meet not only the product performance criteria, but also production process requirements.

System Attributes	**Gen 1**	**Gen 2**	**Gen 3**
Capital Investment	Required	Minimal/None	None
Adhesive Formulator	Mill	Supplier	Supplier
Viscosity	15,000–100,000 cP	300–20,000 cP	300-5,000 cP
Pot Life/Shelf Life	Short	Extended	Extended
Total Solids	< 40	40–55%	40–60%
Sprayable	No	Yes	Yes
Components	2	2	1

Table 1 – *Development of the soy flour technology has led to successive generations of improved Soyad® resins.*

Conclusions

Although soy flour was commonly used in plywood manufacturing in the early 20th century, it was displaced by better performing and more cost-effective synthetic adhesives based upon fossil fuels. However, there is now a resurgence of interest in soy flour adhesives because they can be cost competitive and more environmentally acceptable, especially with the emphasis on reduced formaldehyde emissions. To produce these products from abundant soy flour, new technology was needed to both disperse and cure soy adhesives. To make effective soy adhesives, it is important to understand the large structural and performance difference between proteins and synthetic adhesives. Protein structure is much more dependent on intrachain interactions than are most other polymers. These intrachain properties influence the interchain properties and can hide many of the potentially cross-linkable functional groups. A better understanding of these properties has led to better performing soy adhesives and can open new opportunities in the future.

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