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A Review of Bio-Based Adhesives from Primary and Secondary Biomass for Wood Composite Applications

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Abstract **– Today there is a great demand in the market of wood-based panels like medium density fibreboard (MDF), plywood and oriented strand board (OSB). These boards provide functionality in various industrial fields from building to furniture production. All are produced from timber and some type of binding resin, the most often used in Europe are phenol formaldehyde (FF), isocyanate (MDI) and melamine urea formaldehyde (MUF). These resins guarantee sturdiness of the material but are toxic to humans and makes recycling of the wood-based panels very difficult. There are attempts of wood-based panels industry to transition away from fossil-based adhesives. Various resins have been developed using lignin and tannin or protein. Soy based adhesive SOYAD™ has already reached the market, other soy protein-based adhesives are integrated into ultra-low formaldehyde emission particle boards like Nu green 2® and Transform™. This paper gives an overview on bio-based adhesives that are used or have the potential to be used for wood-based panel production.**

Keywords **– Bioresources; plant-based; polyaddition; polycondensation; polymerization; urea-formaldehyde; wood composites**

1. INTRODUCTION

Adhesives are required for many fields of engineering, engineered wood products are no exception. Fibres in timber have great load bearing properties, but the strength of these fibres decreases dramatically when applied in any other way than perpendicular to the longitudinal axis [1]. Engineered wood products are created to even out the load bearing properties of timber by disrupting the natural timber structure and binding the biomass back together with adhesives. Wood-based panels (WP) can be used for construction and carpentry applications. The application of WP depends on their characteristics, such as hardness, surface properties, strength and composition. An important property of WP is moisture resistance; greater resistance can be acquired by the right adhesive or coating. One of the most popular WP are: particle boards (PB), fibreboards, oriented strand boards (OSB). Density of the boards ranges from 650 kg m^3 to 1000 kg m^3 [2]. Fibreboards are classified by density – low density fibreboards (LDF) with 400–600 kg/m^3, medium density fibreboard (MDF) 600–750 kg/m^3 and high-density fibreboard (HDF) with up to 1000 kg/m^3 density [3]. For easier processing and for economic reasons, wood panels are mostly made of softwood. Fibreboards usually use synthetic adhesive such as urea-formaldehyde, melamine-formaldehyde or phenol-formaldehyde [4]. Hardening adhesives are easy to handle with woodworking tools, hence in

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woodworking industry they are preferred over elastic adhesives. Downside of these popular formaldehyde adhesives is their formaldehyde emissions which are toxic to living organisms. The constant demand of wood-base panels in European Union market 2013 – 57 700 thousand m^3; 2014 – 68 800 thousand m^3; 2015 – 64 400 thousand m^3; 2016 – 65 000 thousand m^3 is forcing the development of new products [5].

To make the WP industry more sustainable and use of its products safer, greener adhesive options need to be considered. Despite the upper hand of green adhesives regarding sustainability and health benefits, greener adhesives fail to meet industry's requirements of high strength of internal bonds, dimensional stability, temperature resistance, fast curing, and good viscosity regulation [6].

The focus of this article is on working principles of bio-adhesives like lignin, plant protein, fatty acids and carbohydrates. This paper provides an overview of the working principles and accessibility of raw material for these adhesives. The current situation and possible technologies for the development of a bio-binder and the basis of previous research are reviewed in this article.

2. WHAT MAKES CHEMICALS STICKY

Adhesives have the properties to stick materials together – whether it is surface to surface or internally in the material [7]. Adhesion principles vary; hence it is important to be acquainted to them before choosing the right adhesive for a specific application.

The smallest unit of a substance – a molecule is held together by covalent bonds between its atoms. At the macro level the substance is held together by hydrogen, van der Waals or electrostatic forces. Such interactions in a substance are called cohesion and are influenced by cohesion forces. The effect between the surfaces of substances, on the other hand, is called adhesion – the forces of adhesion determine the specific work required to separate these surfaces [8]. The sum of these two forces – cohesion and adhesion, determines whether surface wetting will occur when a solid and a liquid interact.

Drops of the same volume of different substances on the same surface will form different shapes – with different contact area and droplet geometry. On hygroscopic surface the water drop will be more compact, while the olive oil drop will be rounder. A contact angle will form between the air layer, the surface, and the droplet. There is a direct relationship between this angle and adhesion – the narrower the angle, the greater the adhesion. Efficient adhesive has a strong cohesive, and strong adhesion [9]. Materials and their surface properties differ, hence different adhesives are created to adhere to specific surfaces.

Fluids that can act as an adhesive are also characterized by viscoelasticity. This means that these fluids are viscous – slow flowing and at the same time flexible – they try to return to their original state [10], [11]. The natural viscoelastic substance is honey – it is viscous and sticky. Honey appears sticky when taken between the fingers, it wets the skin and has strong cohesion and adhesion to skin.

The principle of adhesion includes both mechanical binding and specific binding. Mechanical binding occurs when the adhesive enters the texture of the surface on which it is poured. Specific adhesion includes electrostatic forces – van der Waals forces, hydrogen bonds [12], or even covalent bonds if chemical reactions also take place between the surface and the adhesive. It is often observed that the adhesion between two glued surfaces becomes stronger over time, this is because due to its viscosity it slowly flows into the finer grooves of the surface, increasing the surface between the glued materials and the adhesive, thus increasing the total adhesion.

There are cases when better mechanical adhesion can be achieved by diluting the adhesive with a more flowing solvent – thus reducing its viscosity. This approach allows the adhesive to fill the surface relief of the substrate faster, in this case the challenge is to get rid of the solvent, because only when the adhesive regains its original viscosity or even hardens, adhesion is ensured [13]. Some solvents, like acetone, evaporate rapidly, but solvents like water take longer time.

2.1. Types of fossil-based adhesives used in industry (e.g., polyvinyl acetate, urea-formaldehyde, and others)

High throughput production plants are characteristic to wood-based industry. Particleboard production plants can have as high throughputs as 500 thousand m^3/year [14]. Although adhesive comprises only 2 to 5 % of the final wood-based board, it makes up for up to 25 % of the product costs, therefore making a great impact on production profitability. This promotes the research and development of new and improved adhesives, nevertheless popular synthetic adhesives are industry standards – providing durability and resistance to water [3].

For gluing wood substrate, the popular polyvinyl acetate (PVA) water emulsion is produced in factory reactors from vinyl acetate monomers. The polymerization reaction is catalysed by the free radicals formed from decomposition of hydrogen peroxide, inorganic persulphates or organic hydroperoxides. Polymerization in this case is by-product free, as nothing else is created in the chemical reaction. As PVA glue is an aqueous solution of different concentrations of PVA, the glue only starts to hold the surfaces together when the water has evaporated [15], therefore it takes time for the process to take place passively, if faster curing is required, forced convection can be applied.

An alternative to increasing the adhesion strength of adhesive solutions is chemical hot-melt adhesives such as the well-known polyamide [16], polyolefins, polyester and polyurethane. The basic principle of these adhesives is similar to the already mentioned PVA adhesive – they are synthetic polymers that do not chemically react with the substrate. Depending on the adhesive, they have a specific melting temperature at which it passes into the liquid phase, but as the temperature decreases, it returns to the solid phase [11]. Choosing the wrong temperature to melt them can release volatile compounds from the polymer, creating an unpleasant 'burnt plastic' odour [17]. Hot melt adhesives have been available for many decades and are widely used, however, they have performance limitations such as poor heat resistance, low resistance to UV radiation, and water or solvent permeability [16]. However, the wide application of such adhesives is determined by their diversity – each type has copolymer variants, providing a range of performance properties according to the desired application. Depending on the application, many forms of hot melt adhesives (granules, blocks, films, tubes) [11] and application patterns (dots, spiral spraying or continuous lines, etc.) are available on the market [18]. Hot melt films are ideal for laminating a wide range of fabrics and making joints without seams, for example in the manufacture of fabric raincoats.

The variation of the glue application method allows to control costs – consuming only as much material and energy as needed for the specific case. As such, adhesives are generally difficult to spray due to the need for uniform temperature, contact with cooler air allows them to cool faster. Therefore, in practice it is customary to apply the adhesive in the form of a powder [11] – it provides less difficulty in storing the adhesive compared to the film, to ensure a thin layer of application on the substrate. Most hot melt adhesives are available in powder form in a variety of particle size ranges. Polyester and melamine urea formaldehyde in the form of fibres are used in the production of thermal insulation wool – for bonding glass, and stone, as well as various alternative ecological substrates [19].

Chemical adhesives are essentially like the adhesive solutions mentioned above, the difference being that the polymers are formed directly between the substrates and are not previously formed and dissolved to make a low viscosity solution. There are three polymer formation reactions:

1. Polymerization (unbound polyester adhesives, cyanoacrylates, anaerobic adhesives) [20];
2. Polycondensation (polyamides, phenolic adhesives and silicones) [19];
3. Polyaddition (epoxides, polyurethane, and silicone adhesives) [21].

Such adhesives consist of fillers, monomers, catalysts, and other additives such as reaction accelerators or inhibitors that promote a slower and more uniform formation of polymers. The properties of such adhesives have several advantages – resistance to high temperatures > 200 °C, silicone-based adhesives can withstand above 200 °C temperatures. These properties are suitable for aerospace applications where strength may not be compromised by high temperatures [22]. Most types of adhesives lose their strength at around 60 °C [23]. The high heat resistance can be used for improving plywood resistance to fire, by replacing the polyurethane adhesive in the outer most layers of veneer with silicone based elastomer with fire retardant additives [22].

Synthetic polymers can have high resistance to abrasion [24], and bending [25], withstanding up to 40 MPa mechanical forces. Therefore, such adhesives are used in cases where they must withstand heavy loads, such as in the production of particle board. Polyurethane adhesives have excellent flexibility withstanding repeated elongation – up to 60 % before breakage [26].

It is also possible to glue metals using adhesion forces, for this purpose resins are usually used, they are a type of anaerobic glue. Once they have hardened – polymerization has taken place, they are difficult to deform. They are used in the assembly of joints (by gluing metal screws) and are known for their ‘locking’ ability. The specificity of the resin is that the polymerization takes place only when oxygen has been removed from the system [27]. However, this is not difficult to achieve, as the compounds for which the resin is used are usually sealed.

2.2. Bio-based adhesives

The first adhesives were exclusively of organic origin, even back in the 20th century [28]. At the beginning of World War I, bio-based polymer solution in water was often used as adhesive. First analogue of PVA glue was a starch solution in water. This solution has the same working principle – when the water evaporates, long polymer molecules, in this case – starch, are left behind on the surface [28]. Starch is a polysaccharide made up of glucose molecules linked by α-1-4 or α-1-6 glycosidic bonds [29]. Therefore, similar to PVA, starch solution penetrates the pores of the substrate and, when the starch molecules intersect, they are mechanically bonded together when water has completely evaporated. Starch is formed in plants as energy reserves, so it is easily available, it is obtained from grains, legumes, potatoes and corn [29]. Bone glue was another bio-based polymer-water solution. Bone glue was obtained from animal connective tissues by boiling them. In this case, the polymer that acts as an adhesive is collagen, a protein that *in vivo* provides tissue elasticity. Collagen consists of long protein molecules that are usually linked together by sticky amino acid residues. When these bonds break under the influence of elevated temperatures, the sticky ends are released and as the temperature decreases, they again adhere chaotically to each other and to the substrate by electrostatic forces [10]. Additional benefit of such adhesive is the sustainability of raw material – as secondary bioresource, bones are residues and do not compete with food resources [30].

Over time, these protein adhesives were improved, and denaturants and crosslinkers were added to soybean protein adhesives to improve their water resistance, shelf life, and consistency of durability. The added substances were urea, sulphur compounds and inorganic salt complexes. Each of these groups of compounds reacts with the components of soybean meal, starch and protein [13]. Formaldehyde used to be additive to soy protein, as formaldehyde itself cures very well, but it hardens too quickly and is therefore difficult to control when used alone.

However, there are health risks associated with formaldehyde, so solutions are being sought to reduce the amount of formaldehyde in wood products used indoors. One such way, without significantly reducing the mechanical properties of the material is to replace part of the formaldehyde with lignin. This method is not new, however, with increasing public health concerns, a few MPa reductions in material strength are not as significant as the reduced health risks. Lignin is a natural aromatic polymer that contains cellulose fibres, forming lignocellulose found in the walls of plant cells and ensuring their firmness. Cellulose is mechanically bound to lignin - cellulose is structurally similar to starch – made from glucose molecules, which are bound together by β-1,4-glycosidic bonds, while lignin forms a complex lattice by tying filamentous cellulose fibres. The production of cellulose pulp produces significant residues of lignin, as it is found in wood in similar amounts as cellulose [31]. Depending on the pulp production method, several types of lignin are formed. The chemical extraction of pulp is either sulphite or alkaline. Sulphite pulp is pulverized with sulphite under acidic or alkaline conditions [32]. This process produces lignosulphonate, which is quite high in molasses, but the sulphonic acid in the solution keeps this lignin in solution. Sulphonic acid groups ensure the surfactant properties and hygroscopicity of lignosulphonate. These properties of lignin make it a suitable emulsifier and adhesive. However, in the context of adhesives, these properties, and the poor ability of the lignosulphonate to bind to, for example, phenol-formaldehyde adhesive mean that it is not suitable for application in formaldehyde resins. In turn, lignin obtained by alkaline pulping has several phenolic hydroxy groups, which allows it to bind to phenylpropanoid groups [33].

As the above-mentioned synthetic adhesives have been proven to be the most effective in achieving the performance of wood fibre panels, only reduction in the concentration of these adhesives in search for various natural fillers such as lignin can be seen in production [34].

2.3. Sources of bio-based adhesives

Bio-based adhesives and their corresponding sources are depicted in Table 1.

TABLE 1. BIOBASED ADHESIVES AND THEIR USES FROM SCIENTIFIC LITERATURE

Biological source	**Compound**	**Polymer formation reaction**	**Primary raw material***	**Uses**	**References**
Penicillium oxalicum	Anhydrous citric acid	Polycondensation	No	Wood composites	[35], [36]
Shrimp and other crustaceans	Chitosan (Carbohydrate)	Polycondensation	No	Medicine, wood composites	[37], [38], [46]
Vibrio parahaemolyticus	Exopolysaccharides	Polyaddition	No	Research, low technology readiness	[39]
Flowering plants	Latex (Isopropene)	Polymerization	Yes	Wood composites	[28], [40]–[42]

Biological source	**Compound**	**Polymer formation reaction**	**Primary raw material***	**Uses**	**References**
Wood	Lignin (Aromatic polymer)	Polycondensation	No	Wood composites, foams	[43], [44]
Oleaginous plants	Polyols	Polyaddition	Yes	Wood composite, foam	[8], [45], [46]
			No	Wood composites	[45], [47]
Wheat	Protein	Polycondensation	Yes	Paper	[48], [49]
Fish	Protein	Polycondensation	No	Wood composites	[50]
Rapeseed cake	Protein, carbohydrates, and other residues after oil press	Polycondensation	No	Wood composites	[51]
Potatoes	Starch (Carbohydrate)	Polycondensation	Yes	Packaging	[29], [52], [53]
Tree bark, cork	Suberin	Polycondensation	No	Wood composites	[54], [44]
Potato tubers		Polycondensation	No	Research, low technology readiness	[35]
Flowering plants	Tannin (Polyphenol)	Polyaddition	Yes	Wood composites	[6], [55]
Wood	Hemicellulose (Carbohydrate)	Polycondensation	No	Wood composites	[44], [56]
	Vanillin (Phenol)	Polycondensation	No	High temperature environment	[57]
Vanilla planifola	Vanillin (Phenol)	Polycondensation	Yes	High temperature environment	[57], [58]

* Biological source marked as “No” is classified as secondary or tertiary raw material.

Additives such as citric acid and 1,2,3,4-butanetetracarboxylic acids may be added to increase the performance (strength and resistance to moisture) of natural binders and to facilitate their use [7]. The use of organic acids in adhesives or their production is a common approach. For example, citric acid as a crosslinker and a hydrolytic agent can be used as a plasticizer in starch matrices due to its structural properties. Another organic substance which can be used as an alternative to formaldehyde resins are tannins because they have many phenolic rings in their structure. Citric acid promotes the reaction of tannin and sucrose at lower temperatures, thus potentially reducing energy consumption. An alternative to citric acid can be ricinoleic acid, which can be obtained from renewable sources – castor oil [6]. Ricinoleic acid is a C18 fatty acid that is also used in the production of lubricants, its properties are made so different by the dual nature of fatty acids – their acid functional group makes them polar, while the long tail of the molecule has non-polar properties [59]. Tannins, in addition to greater mechanical strength, also help protect the material from water. To make the adhesive easier to work with, it is desirable to obtain a relatively flowing consistency to avoid unnecessary consumption and ensure the homogeneity of the material [6], [60]. As already mentioned, a more fluid adhesive that flows into the gaps in the surface of the substrate increases the contact surface between the surfaces of the substrate, thus also increasing the tensile strength and modulus of elasticity. Proportions vary, but experiments show that acid concentration in solution should be around 25 % in order to achieve the desired viscosity [6], [7].

Alternatively, it is possible to follow the path of the synthetic additive by adding vinyl acetate to the starch. In this case, the long starch molecules are crosslinked with smaller vinyl acetate molecules that could be linked to the hydroxyl group of glucose by ester bonds. This process is called grafting as the smaller monomers are added on the sides of starch polymer [61]. Such addition of synthetic excipients can increase not only the mechanical strength, but also the water repellence. Vinyl acetate prohibits water penetration, but starch on the other hand forms hydrogen bonds – it attracts water and swells very easily, which in turn reduces the mechanical strength of the material [61]. Samyn describes some biomimetic and gene engineering solutions for green adhesives, but at this point these approaches are at low technology readiness levels [62].

Starch structure – its branching intensity, also differ from plant to plant. Hence different results can be achieved from corn [63], cassava [64] and other starch sources [10], [65].

Chitin is a similar natural polymer to starch and cellulose. It forms the cell membranes of fungi as well as the exoskeletons of invertebrates. The chitin monomer is glucose, which, like cellulose, is linked by β-1,4-glycoside bonds, the difference being that the hydroxyl group at carbon 4 in the glucose monomer is replaced by an acetyl amine group. Chitin, unlike cellulose, also contains nitrogen. By treating chitin with alkali, it can be hydrolysed to smaller oligosaccharides. Chitosan is obtained in this way, but it must be deacylated by treatment with an organic acid, such as acetic acid, to make it sticky. The obtained glue can be used not only for gluing timber, but also for wound treatment, helping to stop bleeding. There is evidence that chitosan can be used as coagulant in wastewater treatment plants [66]. Chitosan is electrostatically attracted to negatively charged surfaces, which is possible because the deacylation of chitin leaves a free $-NH3^+$ group [67], [68]. The polarity of chitosan means that this adhesive also binds water well, so the properties of the wood deteriorate in the presence of water.

The positive aspect of chitosan is its production potential from the production residues of other products, so it can become a by-product, such as in production of shelled shrimp [67].

Another promising adhesive derived from natural raw materials is polyol adhesive. It can be obtained by transesterification of vegetable oil with glycosylated starch [8]. Higher hydroxyl content in polyol improve the bond strength, hence making these polyols more suitable for wood-based panel production [63], but are not compostable at the end of their life cycle [69]. Polyols are transesterified fatty acids, when they are joined by ester bonds, there are few microorganisms or enzymes in nature that could break them down. Polyurethane, on the other hand, is obtained by reacting isocyanate with fatty acids, the biggest negative aspect of which is toxic cyanide [55] when it is formed during combustion, so flame retardants are always added to polyurethanes, which makes it more difficult to process at the end of its life [70]. Due to cyanide safety issues, nonisocyanide polyurethanes (NIPUs) are developed by using tannins as isocyanide replacement [55].

With the push and support from policy makers to green chemistry, safer adhesives have been developed but at this point there are only few available on the market [71], [72], but at this time they do not reach the >95 % bio-based components requirements. Most of the adhesives' summarized in Table 1 working principle is based on condensation reactions as in urea-formaldehyde. Research in bio-based adhesives field could be divided into multiple groups – specific compounds (latex, vanillin), compound groups (e.g., lignin, hemicellulose, suberin), and non-specific substances with adhering properties (e.g., bark powder). Although all might result in good adhering properties, the specific compound development would be favourable in industry as can ensure the most persistent product quality for the user.

3. CONCLUSION

Finding a sustainable raw material, and persistent product quality are the main challenges in the field of bio-based adhesives. Nevertheless, research papers rarely investigate potential costs and possible production volumes. As discussed in this review, adhesive costs make up a considerable part of total wood composite production costs. In addition, large throughput of wood composite production plants requires adhesives that are available in required quantities and for a price that cannot be higher than synthetic fossil-based options already used in the industry.

Product integrity is still and will continue to be a topical issue, as bio-based adhesives are prone to damage from water. There is more success with bio-based adhesives in medicine and food production where the water attraction is beneficial. According to the bio-based adhesives state of the art, lignin and polyols are the most suitable for wood-based panel applications. Biotechnology and biomimicry approaches are rarely used in adhesives research, hence at this point, it is unclear whether these fields will have a significant impact on bio-based adhesives' further development.

Plant material has been the main raw material for research on bio-based adhesives. Adhesives are widely used and sold for relatively low prices as bulk chemicals, hence research on plant-based adhesives should be propelled by the availability of secondary plant biomass, possible production volumes of the bio-based adhesive and potential market price of the bio-based adhesive.

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