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Biocomposites: Use of Lignin as a Core Material in Synthesis of Various Industrially Important Biomaterials

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ABSTRACT: In the last century, the major achievement of the material science is the generation of number of synthetic polymers and composites which has revolutionized the world. But these materials are a potent source of pollution also, as these are non-biodegradable under natural environmental conditions. Lignin is one of the major constituents of natural and biological fibers, along with cellulose, hemicellulose, pectin, xylan, inulin etc. It is universal and the most essential part of plant cell walls of all types, making it one of the most common natural polymers in the world. It is second only to cellulose in its abundance. The application of lignin and lignin materials as a natural fiber to utilize in synthesis of polymer composites has been topic of interest in recent past. The research focused on lignin materials synthesis is due to its easy availability, rigid structure, resistance to different chemicals, ecofriendly nature, cheap cost, easy extraction from plant polymers and its sustainability. The aim of this study is to provide a systematic review of use of lignin in synthesis of various lignin fiber reinforced polymer composites (LFPCs) and their applications. The comprehensive use of lignin in aerogels, lignin reinforced thermoplastic composites, thermoset composites, bioplastic composites, composite carbon nanofibers, and rubber composites will discussed in the text to come.

KEYWORDS: Biocomposites, Lignin-composites, Lignin-reinforced polymers, Nanofibers

I. INTRODUCTION

At the present about 80% pulp manufactures kraft process is the main method of wood cooking in the world. During kraft process about half of the wood is dissolved and as the result of this process the black liquor contains inorganic chemicals and large quantities of organic materials, mainly lignin, hemicelluloses, poly-, oligo- and monosaccharides, and lignocellulosic fraction [1]. In order the maintain of profitability pulp and paper mills, producers have begun to research new opportunities to increase their income through the production not only pulp and paper as well as other biomaterials. At the present also increased interest in renewable resources because all around the world expected shortages of access to oil and gas [2], and concerns about the accumulation of greenhouse gases in the atmosphere [1]. Lignin is a recalcitrant biopolymer composed of varying proportions of cross-linked phenylpropane units (Fig. 1). The lignin also has a number of applications as a "drop-in" material for industrial composites and admixtures. His is particularly valuable as the loading of lignin in strictly synthetic polymers can yield composites with equivalent or improved physical properties [3] with the additional benefit of improving environmental and cost performance issues.

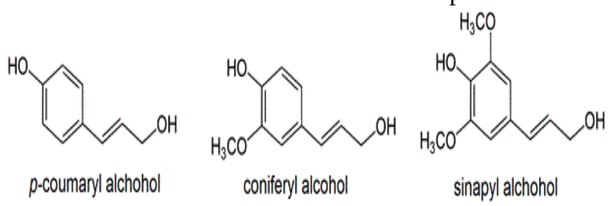
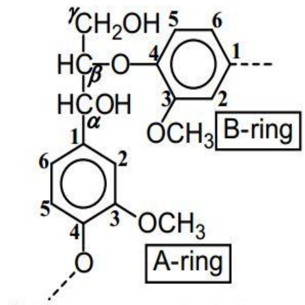


Fig 1: The monomeric constituents of lignin

The concept of integrated forest products biorefinery now is interesting for many researchers who have decided to try to extract, lignin from wood biomass and by-products from the pulp and paper industry [1]. The lignin is valuable product of the biorefining process of black liquor. The basic structural elements of lignin are phenylpropane derivatives. The main precursors of lignin are three monomers phenylpropanoids, namely p-coumaryl, coniferyl and sinapyl alcohols. The most common linkages between these monomers are the β-O-4 or α-O- 4 ether linkages (Fig 2).



β-O-4 substructure in lignin

Fig. 2: β-O-4 sublinkage in lignin molecule

Lignin is a polymeric compounds with molecular weigh 1000-4000. Ability to lignin dissolve depends on the ability to form hydrogen bonds and depends on their molecular weight, structure. Reactivity and chemical properties of lignin depend on functional groups, ie.: -OCH3 and OH and carbonyl groups [2,4]. Extracted polymer fractions from black liquor can be used in various industries. Lignin modification allows to obtain innovative polymeric materials with special properties (for example for medical applications or in plant protection products), lignin could be a substitute phenol in the production of plastic materials. Change structure of lignin can result for example in increase of content functional groups - OH - alcoholic or carbonyl and reduction of phenolic groups. The result is a polymer with completely new properties with a focus on thermoplastics or improvement of biodegradability in the case of a merger with other plastics [3, 4]. Accordingly, the materials made on the basis of the lignin-polymers complex could find wide application in medical, hygiene products and in the packaging industry.

Lignin fibre composites are materials which combine various proportions of lignin together with other natural fibres and additives, and sometimes petroleum-based compounds, to create specific desired characteristics [5]. Many of these composites are designed specifically as ‘green’ alternatives to displace petroleum-based plastics. There are ‘pure green’ versions, which mix lignin with fine natural fibres made of wood, hemp or flax and natural additives such as wax. In Germany, the Fraunhofer Institute has developed a technology of this type and, through a commercial firm – Tecnaro – produces and markets a proprietary thermo-plastic granulate used for injection moulded wood applications. The material is being used for making car interior parts and various other applications, potentially including commercial scale mouldings and doors. Reportedly, one of the advantages of ‘Arboform’ over conventional plastics is that it shrinks very little on moulding. For the lignin-based application being considered for Alberta in this report – plastic and composite windows – this would be an important attribute.

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Fig 3: Different forms of sheets, base material made of lignin composites (Source: www.technaro.de)

II. LIGNIN FIBER COMPOSITES

'Hybrids' (The lignin and petroleum product mix materials)

Petroleum-based plastics are well-developed in the market place, versatile and generally inexpensive (i.e. in terms of direct costs: see later discussion regarding the societal costs of slow rates of waste decomposition, plastics pollution and low levels of recycling). Clearly, petroleum-based plastics are the established 'players' that command a dominant market share in a vast array of end-use applications. In this sense, virtually all natural polymer and natural fibre composites target the overall plastics market for their potential growth, and most (because they are limited to specific attributes and performance properties) are designed to displace specific types of petroleum-based plastics [6]. Not surprisingly, especially with the growing importance of being 'green', petroleum-based plastics manufacturers are willing to consider 'hybrids' which combine hydrocarbon with carbohydrate materials. Several innovative bio-plastic firms have emerged to meet this need. Cereplast Inc., based in California, manufactures proprietary 'pure' and 'hybrid' bio-plastics from starches (corn; what, tapioca and potatoes) which are used as substitutes for petroleum-based plastics in the major converting processes such as injection molding, thermoforming, blow-moulding and extrusions - at a pricing structure that is reported to be competitive with petroleum-based plastics. As one of the firms on the cutting-edge of bio-based plastic material development, Cereplast offers resins to meet a variety of customer demands [1,7]. Cereplast Compostables Resins® have 100% renewable content and are 100% de-compostable. They are suited for single use applications where high bio-based content and compostability are advantageous, notably in the food service industry. Cereplast Hybrid Resins have approximately 50% renewable content. They combine high bio-based content with the durability and endurance of traditional plastics, making them ideal for applications in industries such as automotive, consumer electronics and packaging.

Fig.4: Food serving cups, plates and containers made up of lignin composites (Adapted from: www.cereplast.com)

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Technical and Strategic Reasons for Lignin Composites

Pure lignin in liquid form is highly viscous. It can be used to produce a wide range of bio-plastics through injection moulding technologies (e.g. building panels). By combining lignin with other natural polymers and natural fibres, however, its range of extruded products potentially is very wide. These other compounds also provide more scope for pure lignin (or ‘’liquid wood’) to be designed and engineered to develop desired characteristics. This is important because there are two primary development strategies available to bio-plastics producers [7]. Firstly, they can work to carve-out new product niches in emerging ‘green markets’ and eventually hope to develop these to large volume markets. Secondly, they can ‘target’ various oil-based plastic market segments (e.g. polypropylenes) and try to displace them. In the latter case, collaboration with already well-established and well-capitalized oil-based plastics producers could yield potentially mutual benefits.

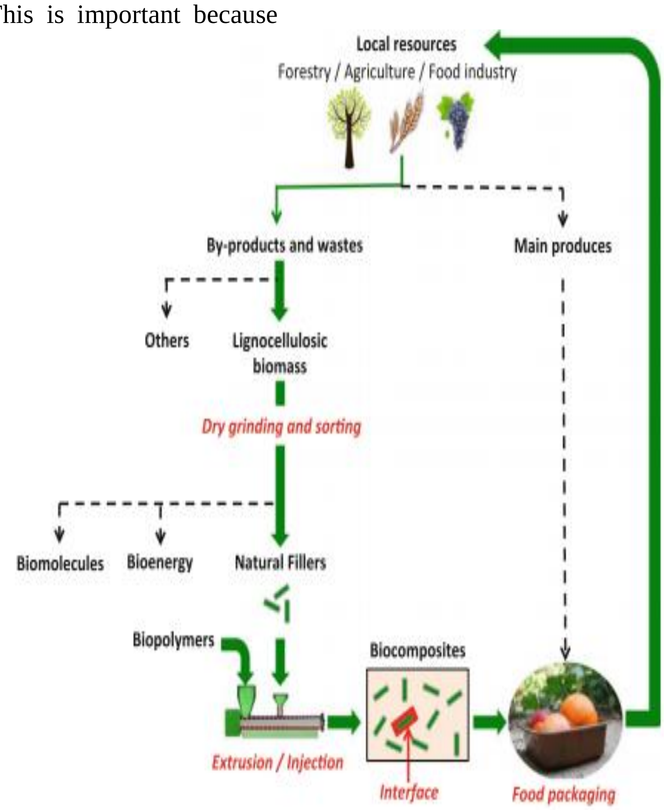

Fig 5: Conversion of lignocellulosic biomass for various composite formation

III. SURFACE MODIFICATION OF LIGNOCELLULOSIC FIBER

The use of natural fibers as reinforcement in polymer composites is attracting much interest due to its potential, however, the hydrophilic character of the fibers reduces the compatibility with the hydrophobic matrices resulting in composites with poor mechanical properties. Most of the research related to lignocellulosic fibers and polyolefin matrices showed that the adhesion between the lignocellulosic fiber and polymers such as polypropylene (PP) or polyethylene (PE) was poor [2,3,8]. However, these polymers could form hydrogen bonds with the hydroxyl groups of lignocellulosic fibers under specific reaction conditions. The adhesion between lignocellulosic fiber and polymer matrix can be improved (i)modifying the surface of fibers, (ii)modifying the polymer or (iii) modifying both of the at the same time. Surface modification is essential to reduce the hydrophilic character of the lignocellulosic fibers, improve the wet ability between the fiber and matrix and to improve fiber/polymer matrix adhesion.

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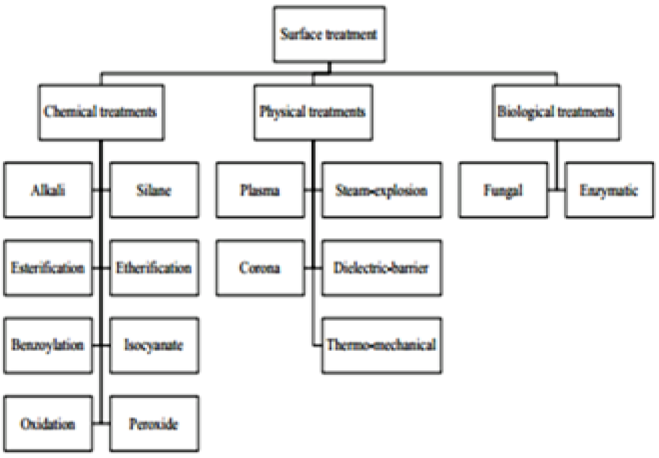

Fig 6:. Main methods for surface treatment of lignocellulosic materials.

IV. LIGNOCELLULOSIC FIBERS RE-INFORCED THERMOSET COMPOSITES

The Composites manufactured by natural constituents must meet the following requirements [2,4,5,6,7, 10]:

(a) Use of renewable and/or biodegradable,

(b) Elimination of environmental and toxicological effects (emissions of degradation components, additives or fillers) at may possibly take place at any stage of the product life cycle

(c) Implementation of an option for waste managing (recycling, composting and incineration) to guarantee the return of the energetic cost of the product.

The lignocellulosic fibres may be modified by chemical or physical methods. Physical methods, such as stretching, calendering, heat treatment, laser, gamma rays, UV, plasma, used to change the surface and structural properties of the fiber [11]. Chemical modifications include treatment with sodium hydroxide, the silane (silicon alkoxide functionalized), acetic acid, or based molecules of benzoyl, iso-cyanate, triazine or imidazolidinone, etc. as discussed in the figure above.

Table 1 : The physical properties of various lignin fibres

	Density (g/cm^3)	Tensile stress (MPa)	Young's modulus (GPa)	Specific Young's modulus (GPa)	Deformation (%)
Flax	1.45	500–900	50–70	48	1.5–4
Hemp	1.48	350–800	30–60	41	1.6–4
Kenaf	1.3	400–700	25–50	38	1.7–2.1
Jute	1.3	300–700	20–50	38	1.2–3
Bamboo	1.4	500–740	30–60	36	2
Sisal	1.5	300–500	10–30	20	2–5
Coir	1.2	150–180	4–6	5	20–40
Glass fiber	2.5	1200–1800	72	29	2.5
Carbon fiber	1.4	4000	235	168	2

For thermoset composites, the lignocellulosic fibres are combined with phenolic matrices, epoxy, and polyester resins to shape composites. Thermosetting polymers are three-dimensional structures containing reactive groups, which help the development of the interface [3,12].

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These thermosetting matrices, owing to their structure, acquire during cross linking a form which cannot subsequently be changed by rising the temperature. The most developed applications for automotive Body parts.

Fig. 7: The various kraft lignin biomaterials and their applications

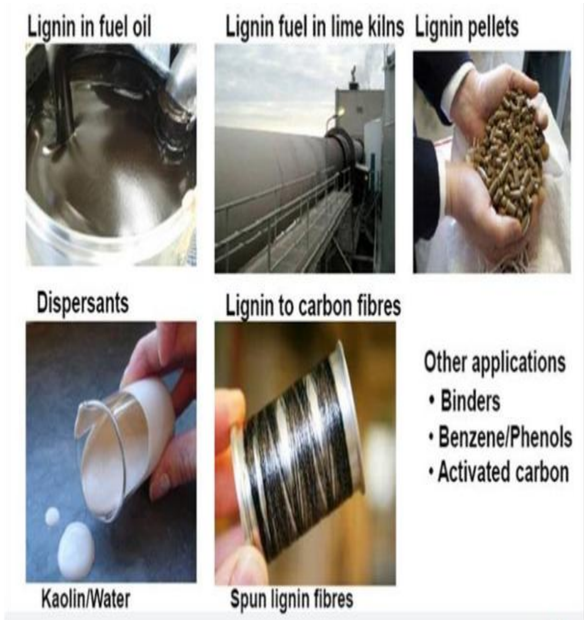

Fig. 8: Miscellaneous applications of lignin

V. LIGNIN IN THERMOPLASTIC COMPOSITES

These are the high density polymeric compounds which are non-biodegradable . However, use of starch, lignin or cellulose core in making of these plastics, render these polymer easily biodegradable without compromising their wear and tear qualities.

Table 2a. :Biomaterials used in thermoset and thermoplastics

Synthetic polymer/thermoset	**Bioploymer used**	**Elastomer formed**
Polymer: Polypropylene, polyethylene, HDPE, High impact polystyrene, PVC **Thermoset:** Polyester, poly	Biodegradable polyester, Starches, Soya proteins, hydroxyl butyrate	Natural rubber

urethane, vinyl-ester		

VI. LIGNOCELLULOSIC FIBRES-BASED BIOCOMPOSITE MATERIALS FOR FOOD PACKAGING

Among innovative technologies through out the food supply chain, food packaging is a particular key player to improve food preservation, quality, safety and retailing/consuming conditions, and thus to reduce food losses in all countries.

Table 2b: Studies of lignin in thermoplastic composites

Polymer Matrix	Source of Lignin	Lignin modification	Remarkable properties
PP	Hardwood Kraft	Esterification by anhydride	Flexural strength and thermal stability
PP	Softwood Kraft	Esterification by anhydride	Stiffness and tensile strength
PP	Sugarcane bagasse	Alkylation and arylation modification in different solvents	High accurate modeling for mechanical properties by various volume fraction of lignin
PP	Wheat straw alkali	Chemical grafting with P, N and Cu elements	Fire-retardant
PE	Alkali	Reverse micelle formation	UV-blocking
PE	Softwood Kraft	Functionalized SEBS	Flexural strength, modulus and toughness
PE	Softwood Kraft	Free-radical grafting	Tensile strength
PE	Softwood Kraft	Methylation and fractionation of lignin	Melt Stability and strength
PS	Kraft	Graft copolymerization of styrene by ATRP	Toughness
PS	Softwood Kraft	Esterification by anhydride	Flexural and impact strength and thermal stability
PA	Soda straw	Used as a filler	Ductility
PVA	Industrial alkali	Used as a filler	Tensile strength and thermal stability

In this context, lignocellulosic based biocomposites appear as innovative and promising materials of future for the food packaging sector. In the objective of reducing the overall environmental impact of the food/packaging system, increasing attention is given to full-biocomposites, i.e. composite materials based on constituents all biosourced and biodegradable (Berthet et al.2015a).

Full-biocomposites are based on a biosourced and biodegradable polymer matrix, i.e. bio-polyesters (e.g. polyhydroxyalkanoates) and agro-polymers (e.g. proteins, polysaccharides, etc.), and lignocellulosic particles or fibres as fillers. The cheapest and most environmentally virtuous lignocellulosic fillers would be those derived from agricultural wastes, forestry by-products or food industry solid by-products. The drawback shampering currently implementation of full-biocomposites at industrial scale are mainly their limited processability, unstability in usage conditions, high production cost (e.g.bio-polyesters) or controversial environmental claims. Developing full-biocomposites for food packaging requires taking into account all the previously presented stakes: reducing the overall environmental impact of the food/packaging system by (i)designing materials with functional properties that allow ensuring food quality and reducing food waste and losses, and (ii) reducing the environmental impact to f the packaging material itself by an appropriate choice of resources and material end of life, while maintaining an economical competitiveness using conventional shaping processes at industrial scale, and ensuring food safety and health of the consumer.

VII. CARBON NANOFIBERS

Application of Lignocellulosic Fiber Composites

The lignocellulosic fibres attracted more interest as alternate industrial materials, especially with wood fiber, which their use has spread to virtually all areas (automotive, construction, furniture). Thermoset composites reinforced by wood fibres are applied in many applications in desks, docks, windows frames, and panel components [5,9,12]. Pipes, panels and protruded profiles fabricated with polyester resin are also very popular.

The use of lignin as a renewable resource to produce less-expensive carbon fibers has been taken into great consideration in recent years [13]. The level of interest in lignin-based carbon fibers can be seen in some recent government and industrial programs. The properties of carbon fiber enable their application in a broad variety of products. Carbon fiber has inherent superior specific mechanical, electrical and thermal properties. Among the precursors for the production of carbon fibers, polyacrylonitrile (PAN) is the predominant material, due to the excellent mechanical properties of PAN-based carbonfibers. However, the high cost of PAN precursors, which makes up 43% of the carbon fiber manufacturing cost, limits its utilization in automotive parts [6, 8, 14, 15, 16]. These nano-carbon materials are again of great value (Table 3).

VIII. LIGNIN – REINFORCED RUBBER

Natural rubber (NR) is an important industrial raw material used extensively in many applictions. NR displays remarkable properties and reinforcing fillers are often added in this matrix to improve the modulus, hardness, wear resistance and reduce the material cost [17,18]. The studies has shown that the role of filler networks for the excellent reinforcement of rubber in which lignin acted similarly to inorganic fillers. In another study, authors [1] developed 50% renewable thermoplastic elastomers called acrylonitrile-butadiene-lignin (ABL) as alternative to ABS by replacing styrene with nanoscale lignin (Figure 9).

Table 3: Nano lignocellulosic materials and their potential applications

Material	Applications
Nanocellulose	• Biomedical applications → Artificial skin and cartilage, medical implants, tissue engineering, drug delivery, wound-healing, vessel substitutes, cardiovascular applications, etc. • Dielectric materials → in oil-filled high-power transformers and cables • In composite manufacturing for various applications • Advanced functional materials → Aerogels, composites for light emitting devices/electronic applications, oil/Water absorbing gel materials, water purification systems • As templates for the chemical synthesis of inorganic materials
Nano lignin	• In the development of composites/biobased polymers: as thermoplastic polymer composites, rubber-based lignin composites, lignin-reinforced biodegradable composites, foam-based composites, besides many others indicating the vast expansion of application for nanolignin • In drug delivery • As UV barrier and antibacterial agent → in finishing process in the textile industries for functional textiles by coating the fabric surface to overcome the problem of color change
Nano silica	• In composites • In encapsulation of living cells

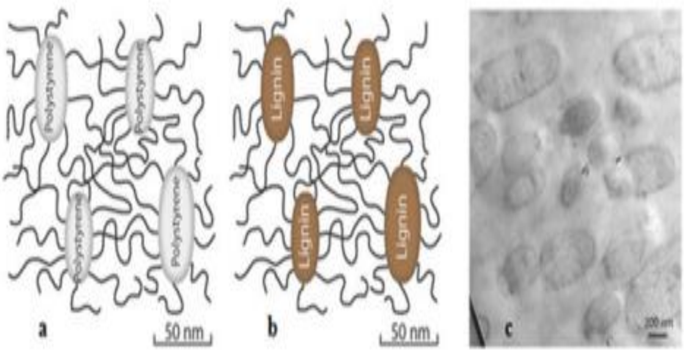

Fig 9: a) Schematic of styrene-butadiene-styrene Block copolymer morphology. b) Hypothetical lignin multiphase polymer in soft matrix. c) Transmission electron microscopy image of NBR-Kraft softwood lignin blend

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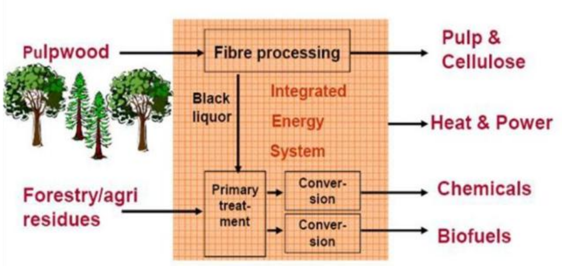

Fig 10: Future perspectives in composite & material research involving plant polymers

IX. CONCLUSION & FUTURE PROSPECT

Future lignin opportunities include the generation of adhesives, resins, and carbon fibers [1, 4, 19, 20]. Carbon fibers are essentially graphite fibers that are virtually 500% stronger than steel, yet at only 20-30% of the weight consequence. Moreover, carbon fibers also have a thermal expansion that is lower than most commonly used alloys implemented today and can be spun into a strand thinner a hair of a human being but with the tensile strength greater than titanium or shaped into rigid molds are be suitable for many applications in the automotive, aviation and power generation industries [5, 7, 11, 15, 21, 22]. For example, carbon fibers as a replacement for aluminum parts in the manufacturing of cars would reduce the weight of the vehicle without sacrificing strength [23, 24], thereby improving fuel efficiencies without sacrificing the performance properties of today's motors or electric/hybrid engine systems that are in use today [16, 25, 26].

The notable properties of lignin, including high availability, low cost, ecologically friendliness as well as antioxidant, antimicrobial, biodegradable properties and reinforcing ability succeeds in exploiting as an ideal candidate for an extensive variety of applications [18, 27]. Substantial efforts are now being made to successfully use lignin as one of the components in polymer matrices for high performance composite applications [5, 7, 8, 19, 20]. The progress of efficient biorefineries that integrate production of bio-based products can reduce costs [4-7] and allow biobased products to compete more effectively with petroleum-based products on price [18,19, 20]. In contrast to most synthetic polymers, lignin has complex structure [21,22,23], which makes its characterization and processing the main factors limiting for wide – scale use in biorefineries [24].

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