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Exploring tannin extracts: Introduction to new bio-based materials

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ABSTRACT In a world seeking environmentally sustainable products, bio-resources are investigated as suitable replacements to oil-derived products. Tannin extracts represent one of the most abundant phenolic resources of the earth, with more than 200,000 T/year. These extractives are constituted mainly of polyphenolic compounds and they are used industrially for various purposes, including leather tanning, wine-making, and water clarification. However, condensed tannin extractives can also be easily polymerized, and the resulting bio-macromolecule can be exploited for other applications such as adhesives and coatings. In recent years, tannin-based polymers have also been used for the synthesis of fire-resistant insulation foams and outdoor wood preservatives. This bio-resource imparts also outstanding water resistance to wood plastic composites (WPCs) and gives great antioxidant activity to nanofibrillated cellulose films, which become an interesting material for "active packaging". This literature review covers these four innovative solutions made from tannin extractives from mimosa or black wattle (*Acacia mearnsii*) industrial powder and provides some basic information about the purification of the industrial tannin extract that can be suitable for more chemically specific usages.
Keywords: Green materials; timber protection; natural foams; sustainable packaging; nanomaterials; flavonoid fractions.

Introduction

Environmental sustainability is currently a major focus in material science, and bio-based resources are fundamental players for producing new materials for the future. Natural components with comparable performances to synthetic ones represent a major advantage in environmentally-friendly engineering.

In this context, the proper exploration of various bio-resources will be a winning point for the future bio-economy that will rely on biorefineries. In these processes, bio-based feedstock's such as agriculture and forest derived products will be transformed into bio-materials and bio-fuels (REDDY et al., 2010).

Wood usage is currently living a new golden age due to the unmatched properties of timber - not only because it is still one of the most reliable materials for building construction purposes, but also because it is CO_2 neutral and this renders this bio-resource even more attractive.

Accordingly, the wood components are also becoming more valuable. The major components of wood, namely cellulose, hemicelluloses and lignin, are already used for a wide range of purposes (CARVALHEIRO et al., 2008; HUBBE et al., 2008; MOON et al., 2011; RAGAUSKAS et al., 2014; LIU et al., 2016; HUBBE et al., 2017; TABARSA et al., 2017). Nowadays, also the extractives representing between 1 and 10% of the dry wood (UMEZAWA, 2000) are becoming an interesting feedstock because of their relatively easy supply (SCHOFIELD et al., 2001; BELGACEM; GANDINI, 2008; ARBENZ; AVEROUS, 2015).

Extractives are composed of various molecules, such as low molecular mass sugars, terpenes and polyphenolics, but the types and the relative proportions of the various extractives are strongly dependent on the wood species. For

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wood species like oak, chestnut, pine, quebracho, and mimosa, the most abundant chemicals are polyphenols, and therefore the extracts are commonly called “tannins”. This name, indeed, refers to substances that are able to tan leather (HASLAM, 1989). Therefore, the tannins are only the polyphenolic substances, whereas the hydrocolloids, sugars, and organic acids of the tannin extract are the “non-tannins”. The two most common families of polyphenols in plant extracts are the hydrolysable and the condensed tannins. The former are esters of simple sugars with gallic or ellagic acid (ARBENZ; AVEROUS, 2015), while the latter are oligomers or polymers of oligomeric flavonoids (proanthocyanidins), as shown in Figure 1 (SCHOFIELD et al., 2001; HAGERMAN, 2002).

Tannins are the second most abundant phenolic resource in nature, just behind lignin. Condensed tannins (proanthocyanidins) constitute more than 90% of the total world production of commercial tannins (200.000 tons/year) and hence the condensed tannins are the most abundant extracted natural substances on Earth (JORGE et al., 2001; PIZZI, 2008).

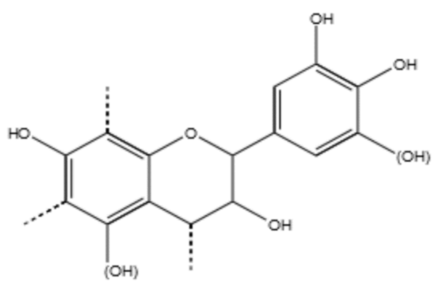

Figure 1. Chemical structure of a proanthocyanidin repeating unit of condensed tannin.

Among the sources of condensed tannins, the industrial tannin extracts from mimosa (*Acacia mearnsii* De Wild, also known as black wattle) are the most sustainable because i) its bark contains 30 to 45% of the tannins; ii) the species grows fast, with a plantation cycle of 7 years. Statistical data show that the surface of planted mimosa increased by 26% during the 2010 to 2015 period, reaching 160.000 hectares in 2015 (IBA, 2016). Regarding the economic use of the raw material, the value of the acacia bark is around $90/T, while the wood (with a density of 650 kg/m^3) is sold at around $30/T (AGEFLOR, 2015; DELUCIS et al., 2016). Acacia mimosa wood is used mainly for charcoal and pulp production. Mimosa tannin extracts are obtained through hot water extraction of bark chips in a counter-current series of autoclaves, using different parameters of temperature, pressure and time according to the required final properties of the extract (ARBENZ; AVEROUS, 2015; MISSIO et al., 2017b; MISSIO et al., 2018).

Condensed tannins have been used for centuries for leather tanning (PIZZI, 2008), and for decades for the production of adhesives (CARVALHO et al., 2014), wines (RINALDI et al., 2016), and water filters (BELTRÁN HEREDIA; SÁNCHEZ MARTÍN, 2009). More recently, the use of these phenolic substances was also examined for their antioxidant and antifungal activities in pharmaceutical products (WEI et al., 2015; AIRES et al., 2016; MISSIO et al., 2017b), for coatings (PAN et al., 2015), and for the synthesis of advanced ultra-lightweight materials (AMARAL-LABAT et al., 2013). In particular, polymers synthesized from mimosa extractives resulted in plastics that were less brittle than those made from other condensed tannins, and the polymerization of mimosa extractives was easier to control (PIZZI, 1994).

This literature review examines the application of tannin extracts from Acacia mimosa for the synthesis of innovative materials, such as wood preservatives, insulation foams, wood plastic composites, and nanocellulose films. Finally, a short overview on the purification method for this phenolic bio-resource is also provided.

New Bio-Based Materials

Wood Preservation

Wood can be affected by extrinsic factors that limits its service life (CADEMARTORI et al., 2015b; MISSIO et al., 2016). Wood quality decreases with outside exposure, and the rate of deterioration is dependent on the timber species (LAZAROTTO et al., 2016), the preservative treatment applied and the environmental conditions (e.g., weather, soil, etc.). Among these, the preservative treatment applied is the factor that can be more easily controlled to enhance the service life of wood (MAGALHÃES et al., 2012). Over the years, many processes have been developed in order to protect wood against xylophagous agents (CADEMARTORI et al., 2015a) and the severe environmental restrictions related to the use of toxic preservatives, like creosote, copper-chrome-arsenic (CCA) and, more recently, copper-chrome-boron (CCB) have renewed the interest in finding more environmentally sustainable preservatives.

According to LEBOW (2010), wood preservatives must meet two broad criteria: i) they must provide the desired wood protection for the intended end use and ii) they must do so without presenting unreasonable risks to people or to the environment. Therefore, the idea of protecting wood with wood-derived preservatives has been studied over the past decades by several research groups.

In this context, tannin-based preservatives are a very attractive bio-mimetic solution. Increasing the concentration of these substances, already synthesized by the trees to protect themselves, helps to protect the wood against biologic and UV-light attacks during its service life as a building material (HAGERMAN et al., 1998; TONDI et al., 2013b).

Extractives obtained from mimosa, quebracho and pine have shown moderate resistances to biologic attack by fungi and termites (TASCIOGLU et al., 2013); this resistance could be enhanced with the addition of copper and/or boron salts (SCALBERT et al., 1998; YAMAGUCHI; YOSHINO, 2001; YAMAGUCHI et al., 2002). However, every study in which leaching processes were proposed, serious problems in relation to treated wood and water were observed (TONDI et al., 2012a; TONDI et al., 2013a). Logically, tannins which are obtained by water extraction are also highly soluble after application. A novel approach has been proposed to overcome the leaching problems by using the *in-situ* polymerization of condensed tannins. Formulations containing hexamine as hardener have been proposed and such preservatives exhibited outstanding biologic resistance against *Pycnoporus sanguineus* (THEVENON et al., 2008). These initial findings obtained with a water-based tannin formulation containing hexamine (6%) and boron (<1%) resulted in several interesting findings: i) limited tannin and boron leaching due to the polymerization; ii) wide-spectrum biological resistances (against fungi and insects); iii) improved mechanical and fire properties (TONDI et al., 2012a; TONDI et al., 2012b; TONDI et al., 2013a; TONDI et al., 2013b).

Unfortunately, only moderate weathering resistance was observed. Indeed, the rigidity of the tannin polymers and the sensitivity against radical degradation strongly affect the outdoor application of this formulation (TONDI et al., 2013a). However, several studies have been performed and are still ongoing in order to increase the elasticity of the hardened polymers so that this drawback can be solved (HU et al., 2017; TONDI et al., 2017).

Tannin Foams

Other interesting tannin-based materials are the tannin foams (Figure 2). These porous materials are obtained by copolymerization of the tannin extract with furfuryl alcohol in an acidic environment. The obtained copolymer cures simultaneously with the evaporation of a low-boiling point solvent resulting in a lightweight porous material (TONDI; PIZZI, 2009). These tannin foams are black porous solids that have a skeletal structure and are completely bio-derived: condensed tannin extract represents the largest component (60-80%) while furfuryl alcohol, a derivate molecule of lignocellulosic biomass and hemicelluloses (AGUILAR et al., 2002; CLIMENT et al., 2014; CANHACI et al., 2017), is the remaining part (20-40%). In the tannin foams, different molecules can be added for improving some specific properties. Formaldehyde and isocyanates can be used to increase the mechanical properties (TONDI et al., 2009; LI; RAGAUSKAS, 2012); polyurethanes increase the elasticity (BASSO et al., 2014) while polyaniline can be used in order to obtain a semi-conductive material (TONDI et al., 2015).

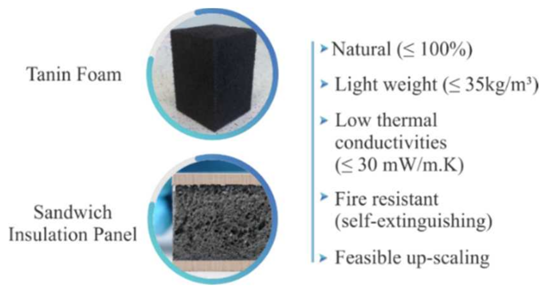

Figure 2. Tannin foam and sandwich insulation panels.

The wide range of formulations developed in the last decade allows tannin foams to be considered for a large set of applications (LINK et al., 2011; KOLBITSCH et al., 2012; TONDI et al., 2014 ; TONDI et al., 2015). In particular, the light tannin foams have shown low thermal conductivities and good fire resistances, which make them an ideal insulation material. Hence, these foams have already been produced in a semi-industrial scale (TONDI et al., 2016b). Interesting properties are observed in different processing technologies introduced in recent years. The tannin-furfuryl alcohol copolymer can be hardened at room temperatures and with external heat sources like conventional ovens (through convection), hot-presses (conduction) and microwave and IR radiations (LINK et al., 2011; KOLBITSCH et al., 2012; TONDI et al., 2014). These processing modifications further extend the applicability of the material because they contribute in shortening the production time. Selection of the proper production method can reduce the amount of catalyst and hardener required, finally leading to the production of a sustainable tannin foam tailored for specific applications.

Wood-Plastic Composites

Wood plastic composites (WPC) are materials containing wood-derived resources (e.g., fibers, sawdust, and wood flour) combined in a matrix of a thermoplastic polymer (e.g., polyethylene (PE) or polypropylene (PP)) (Figure 3). WPCs are used in various building construction materials, such as flooring, exterior cladding and decking (ASHORI et al., 2013). These construction materials combine the mechanical resistance of wood with the formability and hydrophobicity of the polyolefin.

The advantages of WPCs are that they are more environmentally sustainable than plastic alone, and they offer higher durability, lower maintenance and higher abrasion resistance than wood alone (EL-HAGGAR; KAMEL, 2011).

Polymeric composites can be made using a matrix of high density polyethylene, HDPE (ZADOROZHNYY et al., 2016), polyethylene glycol, PEG (TSUBOI et al., 2016), polyethylene terephthalate, PET (MERIJS MERI et al., 2014), or polyvinyl chloride, PVC (YAZDANI et al., 2016). However, polypropylene, PP (MATTOS et al., 2014; CADEMARTORI et al., 2015c; CADEMARTORI et al., 2017) is used more widely for applications like automotive components, electrical devices, food packaging and household equipment (IZZATI ZULKIFLI et al., 2015). Its leading position in the WPC market is due to the combination of economic

(THAKUR et al., 2014a; THAKUR et al., 2014b) and technological performance factors (AYRILMIS et al., 2015).

Various lignocellulosic resources, like sugar cane fibers, ramie, jute, flax, pineapple, sisal, coconut fiber, castor seed cake, cotton, pupunha cover and wood residues, have been used to derive fibers and flours used in composite plastics (SATYANARAYANA et al., 2009; MAGALHÃES et al., 2013; MATTOS et al., 2014; CADEMARTORI et al., 2015c). Nevertheless, the market is largely dominated by WPCs where the lignocellulosic resource is wood (WOOD-PLASTIC, 2017).

The main technological drawback for producing WPCs is the limited adhesion affinity between the wood and the plastic matrix components. Substances that "bridge" these two materials are called compatibilizers and their use for this material represents the principal scientific frontier. Until now, one of the best known WPC compatibilizer is maleic anhydride (TUFAN et al., 2015) which contributes in increasing the mechanical strength of the composite. Recently, renewable substances, like lignin, have been used successfully as a WPC compatibilizer. They increase the thermal stability and the storage modulus of the resulting composites (LEE et al., 2015). These positive results encouraged the investigation of other bio-resources as compatibilizers, like tannins, which have smaller molecular structures when compared to lignin but retain similar functional groups and structure. Furthermore, this bio-resource presents less variability than lignin resulting in a more consistent and reliable compatibilizer.

These studies have highlighted that the mimosa tannins plays an active role in filling the gap at the interfaces between PP and wood and this compatibilization was confirmed by the enhanced storage modulus and hydrophobicity of the WPCs surface (MISSIO et al., 2017a).

Figure 3. Examples of raw material components in WPC products. * PEAD = high density polyethylene; PET = Polyethylene terephthalate; PP = Polypropylene.

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Nanocellulose-Tannin Films

Thin films and bags are extensively used in packaging for the protection of delivered goods from dirt, germs and liquid or gas contaminations (MCKEEN, 2013). These films are usually prepared from polypropylene (PP), low-density polyethylene (LDPE) and linear low-density polyethylene (LLDPE) since these plastics are readily available, have low cost and light weight, and are inert. In Europe, 49 million tons/year of oil-derived plastics are produced, of which almost 40% are used for packaging purposes (PLASTIC, 2017). This trend is no longer sustainable and the need for more natural alternative materials with similar properties is growing exponentially (DE LÉIS et al., 2017; WANG; WANG, 2017).

Simultaneously, the packaging industry is beginning a "new age" in which the packaging simply being inert and impermeable is not sufficient. Delivered goods may require also some chemical support (such as antioxidant and microbiological properties) during the storage phase and therefore "active packaging" was created (EU, 2009). These materials interact with the goods to be protected by releasing antioxidant or antimicrobial compounds into the items to enhance preservation (AL-NAAMANI et al., 2016; DE VIETRO et al., 2017), resulting in prolonged shelf-life.

Future trends indicate that a new frontier of research in this field is the synthesis of active packaging using environmentally sustainable resources. Interestingly, trees provide the basic chemical components to produce active package for the future: cellulose and tannin (Figure 4) (MISSIO et al., 2018).

Cellulose is the most abundant biopolymer on earth; its production is estimated at about 10^{11} tons per year. Cellulose can be found in nature in two forms. The first form is called pure cellulose and is present in cotton, some algae cellulose, and bacterial cellulose. The second form is called complex cellulose, which is present in most plants found in nature as a fundamental component of the cell wall (PECORARO et al., 2008).

The recent addition to the environmental scenario and sustainability in new product development, cellulose, more specifically, fibrillated or nanofibrillated cellulose (or cellulose nanofibers – CNFs) can play an extraordinary role (VALLE-DELGADO et al., 2016; HUBBE et al., 2017). CNFs have dimensions of approximately 5-60 nm in diameter and lengths of few micrometres; these nanofibers are produced through a mechanical fibrillation (friction), and in some cases with the aid of chemical or enzymatic pre-treatments, of cellulosic pulps (IWAMOTO et al., 2008; ISOGAI et al., 2011; KLEMM et al., 2011). One of the main attributes of CNFs – individually or in a nanocellulose matrix, which adds to their value - is the high mechanical strength, such as the high modulus of elasticity that ranges between 10 to 150 GPa (IWAMOTO et al., 2009; LEE et al., 2012).

The production of nanocellulose films is similar of nano-papers (URRUZOLA et al., 2014). Nanofibrils suspended in water generate a gel that, after filtration, produces a very dense film (SEHAQUI et al., 2010). These films show interesting combination of high modulus of elasticity, tensile strength and barrier properties (water and/or gas diffusion) when dry. This makes such films attractive for industrial applications like bio-based packaging, even if they still present high susceptibility to moisture and water (MOON et al., 2011; LAVOINE et al., 2015; LAVOINE et al., 2016; HUBBE et al., 2017).

When tannin was embedded within the nanofibrillated cellulose, the resulting film showed highly antioxidant activity when in contact with water, and this may also represent a certain protection against fungi (YAMAGUCHI et al., 2002; PIZZI et al., 2004; TONDI et al., 2013b; MISSIO et al., 2017b).

Figure 4. Films formed from nanocellulose and tannin.

It was observed that nanocellulose films impregnated with tannin result in a film surface that is more hydrophobic than pure nanocellulose films, which is due to an intimate interconnection between the flavonoid tannin and the cellulose (MISSIO et al., 2018). Such a new, naturally derived material represents one of the most promising "active packaging" materials with antioxidant properties (OLEJAR et al., 2014; ZHOU et al., 2016).

Tannin Purification and Fractionation

The four innovative materials mentioned in the previous sections have the great advantage of using an industrially-available material. However, the presence of non-tannins in the raw tannin powder limits the application of this bio-resource when more controllable applications are needed. Hence, purification of the industrial extract is required when the tannin has to be utilized for more advanced purposes (Figure 6) (MISSIO et al., 2017b).

The tannin industrial raw material containing "non-tannins" can be produced and used in large volumes for low value products (e.g. leather tannin, water treatments). Conversely, after purification, a lower yield of higher value added products is also possible for specific purposes (e.g. films/packages, foams, compatibilizers and antioxidants) rendering the purification process economically viable (LUONG et al., 2012). In addition, diversifying the use of raw materials and processing technologies, as well as reducing dependence on the production of only one product, can provide new combinations needed in different market areas (GHATAK, 2011). For instance, secondary metabolites, such as gums, terpene resins and tannins that are derived from forest resources, can be used for the production of high value-added chemicals, such as cosmetics, pharmaceuticals, animal feeds and food flavours (NAIK et al., 2010).

The separation of wood extractives has a long history. The main process of tannin fractionation is countercurrent chromatography (PUTMAN; BUTLER, 1985). The chromatography process usually uses a Sephadex LH-20 column (TIBE et al., 2013), or newer techniques, such as supercritical fluid and ultrasound assisted extraction (PANSERA et al., 2004; SOUSA et al., 2014). These methods required complex analytical techniques and expensive equipment; hence, easier fractionation methods have been developed. In fact, very few studies have been published on this topic (TENG et al., 2013; TENG et al., 2015) even though the Soxhlet method has been shown to efficiently fractionate other hydroxy-aromatic compounds such as lignin (YUAN et al., 2009; LI; MCDONALD, 2014). Hence, black liquor can be

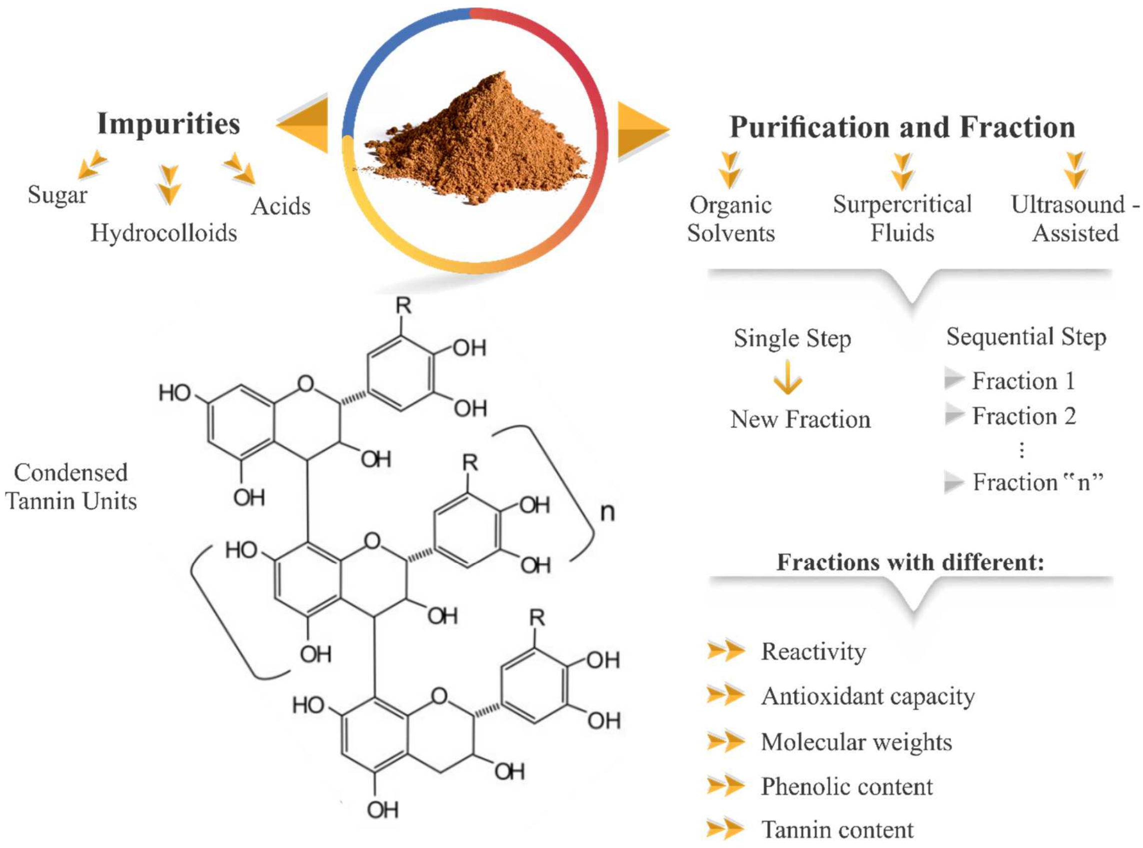

Figure 6. Various tannin fractionation methods and resulting purified fractions

decontaminated and fractionated to yield phenolic monomers of high commercial value (ERDOCIA et al., 2015) to be used as biofuels (GORDOBIL et al., 2016), rigid foams (LI; RAGAUSKAS, 2012; XUE et al., 2014; TONDI et al., 2016a) and composite additives (GORDOBIL et al., 2015; SPIRIDON et al., 2015).

In general, fractionation using organic solvents is characterized by successive extractions with different solvent polarities in order to purify the raw material into fractions containing specific molecular weights components with specific characteristics.

Accordingly, fractions with different molecular weights, antioxidant capacities, condensed tannins, ash levels and phenolic contents can be obtained, each of which can be used for targeted purposes (MISSIO et al., 2017b). Specific tannin fractions can also be isolated through sequential solvent extractions when particular chemical compositions are required.

Conclusions

Tannin is a very interesting bio-resource for many applications and recently several new bio-based materials containing considerable amounts of tannin were successfully produced.

i) Timber preservatives based on tannins have shown very high biological and water resistance, and are already found suitable for indoor wood preservation.

ii) Insulation foams derived from tannin and furfuryl alcohol present low densities, low thermal conductivities and high fire resistances, resulting in an alternative product to synthetic commercial foams, such as polystyrene and polyurethane.

iii) Wood and polypropylene can be compatibilized with tannin in WPCs, increasing the storage modulus and surface hydrophobicity.

iv) Films of nanofibrillated cellulose fortified with tannin have shown higher hydrophobicity and antioxidant activity, producing a new composite with strong potential as active packaging material for food and pharmaceutical goods.

The purification of industrial tannin extracts by targeted fractionation processes allows production of fractions that can be explored for improving the properties of the already produced tannin-based materials of for the synthesis of chemically controlled materials such as ordered aerogels, xerogels or slow chemically releasing structures.

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