Conventional Technology and Nanotechnology in Wood Preservation: A Review

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Wood products are usually treated with wood preservatives to protect them from deterioration. Pressure or non-pressure preservative treatments can be utilized to incorporate biocide into the wood, depending on the applications of the end-products. Thermal and chemical modifications of wood represent alternative treatments that enhance the dimensional properties of wood and provide biological resistance. However, there is also a current trend to apply nanotechnology for wood preservation. Nanomaterials with unique properties can enhance the performance of wood preservatives, thereby increasing the service lifetime of the wood products. Nanotechnology can be applied for this purpose through impregnation of wood with a suspension of metallic nanoparticles, or through encapsulation of biocide with nanocarriers. Additionally, various nanomaterials also can be used in wood modification, especially coating treatment to provide superior service ability. Nevertheless, more studies are required to provide guidelines regarding the safety upon application of nanomaterials. This review will give an overview of current wood preservation techniques. Additionally, this paper examines current research on how nanotechnology is being applied for wood preservation.

Keywords: Nanotechnology; Preservative treatments; Wood modification; Wood preservation; Wood preservatives

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INTRODUCTION

Wood is one of mankind's most valuable and useful natural resources. It plays an important role in various human activities, such as the construction and furnishing of building interiors. However, wood is prone to degradation caused by numerous organisms and abiotic factors. When wood is exposed to moisture and weathering conditions, fungi and insects can consume the lignocellulose component in wood (Upreti and Pandey 2005; Wong *et al*. 2014). A 10% weight loss due to fungal attack resulted in losing more than 50% strength of wood (Ross 2010). Consequently, various wood preservation methods have been developed to increase the service lifetime of wood, thereby reducing replacement costs and allowing for more efficient use of wood in many applications.

The most common wood preservation method involves the application of preservative chemicals (Okorski *et al*. 2015). Factors such as the treatment methods, type of preservatives used, degree of penetration, and retention of the preservatives on wood all contribute to the level of protection achieved by each particular treatment (Coggins 2008).

Apart from the application of preservative chemicals, heat or chemical reagents have been employed to modify the lignocellulose component in the wood cell walls (Hill 2006; Sandberg *et al*. 2017). Figure 1 shows a bibliometric analysis that indicates a dramatic increase in the number of publications on the modification of woods to improve both their dimensional properties and biological resistance. Moreover, several wood modification systems have already been established and commercialized. For instance, *Accoya*® (Accsys Group 2017), *Kebony*® (Kebony 2017), *Tricoya*® (Tricoya Technologies Ltd 2017), and *Westwood* (Westwood Timber Group 2017) are the well-known commercial brands for modified wood.

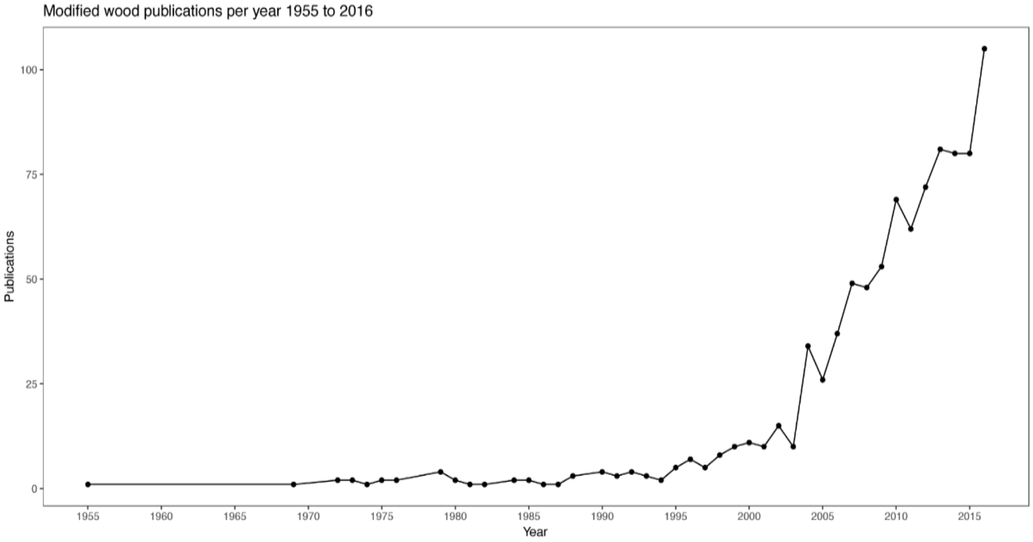

Fig. 1. Wood modification related publications per year, from 1955 to 2016. Reproduced with permission from Burnard *et al*. (2017).

One of the emerging fields that has seen great potential for the industry is the use of nanotechnology in wood preservation (Evans *et al*. 2008). With this method, nanosized metallic wood preservatives, such as silver, copper, and zinc oxide, can be directly applied to wood through a vacuum pressure treatment in which pieces of wood are impregnated under the vacuum and pressure in a closed cylinder. The treatment will result an adequate amount of preservative retained in wood and subsequently protect the wood from certain targeted fungi or insects (Taghiyari *et al*. 2014; Harandi *et al*. 2016). These preservatives may have a deeper level of penetration and higher homogeneous uptake of the particles into the wood, compared to the penetration and uptake rates when using conventional formulations (Matsunaga *et al.* 2009). Another advantage is the fact that the encapsulation of hydrophobic biocide with nanocarriers enables better water dispensability and protects it from degradation (Iavicoli *et al.* 2017). Additionally, various nanomaterials can be used for surface modification of wood such as coating treatment to enhance its hydrophobicity and resistance to weathering (Fufa and Hovde 2010; Hubbe *et al.* 2015). However, the assessment for the potential risk related with introduction of nanoparticles in wood preservation remains crucial. This review paper attempts to give an overview of the current wood preservation methods including wood modifications, wood preservatives, and preservation treatments. The first part of the review deals with so-called conventional technologies. The second main section evaluates current studies into the efficacy and introduction of nanotechnology into the field.

CONVENTIONAL WOOD PRESERVATION METHODS

The treatment methods and the utilization of particular chemicals depend on the following factors: the species of wood, the length of service lifetime, the cost of treatment, the decay risk, and the end-of-life disposal (Tascioglu *et al*. 2013; Brischke and Thelandersson 2014). The degree of wood preservation treatment depends on the level of protection required. There are currently two methods of wood protection, namely wood modification and wood preservative systems.

Wood Modification

The properties of wood can be improved by modifying the cell wall polymers with different types of modification methods. The modification methods usually produce non-toxic materials and can be easily disposed at the end of products life time without any environmental hazard. Modification methods that have been developed can be classed as thermal, chemical, surface, and impregnation modification methods.

Thermal modification

Thermal modification, or heat treatment, has the advantage of enabling treated wood to remain an environmentally friendly product without the use of chemical products. This treatment consists of heating the wood at predetermined temperatures (between 150 °C and 280 ºC) and times in hot media (water, steam, or oil) or under an inert atmosphere (Cao *et al.* 2011; Candelier *et al.* 2013; Wang *et al.* 2016b; Okon *et al.* 2017). These systems may be referred to as thermal (dry) or hygrothermal (high water vapour), depending on the treatment atmosphere.

The chemical reactions involved during thermal treatment include decomposition of hemicellulose, structural changes in crystalline cellulose, and lignin repolymerization (Yildiz and Gümüşkaya 2007; González-Peña *et al*. 2009). According to the study of Rekola *et al*. (2014), heat treatment is also most likely to increase the hydrophobicity of wood and thus reduce the absorption of liquids into the cell walls.

The industry has currently established several commercialized heat treatment technologies (Militz 2002), which are briefly outlined below. Each technology has its own strengths and suitability for different species of wood. The main difference between each of the industrial thermal treatment processes is the medium in which the wood is treated.

ThermoWood® process was developed and licensed to the International ThermoWood Association (Oksanen and Mayes 2003). ThermoWood® is the best established and most widely used technology in the industry. It has the highest production rates, about 179,507 m^3, in Europe (ThermoWood 2016). ThermoWood® offers two standard treatment classes, namely Thermo-S (stability) and Thermo-D (durability); each designed for different end-use applications.

In the Netherlands, the Plato process was developed by Royal Dutch Shell (The Hague, Netherlands) and patented in 1989 (Militz and Tjeerdsma 2001). This process utilizes the presence of the abundant moisture in wood cell wall to provoke reactivity in the cell wall components under comparably low temperatures. Because relatively mild conditions limit unwanted reactions, Plato process is therefore able to reduce strength loss due to heat treatment.

Furthermore, there are many other useful heat-based processes utilized in the industry, such as Westwood (Westwood Timber Group 2010), Retification (Vernois 2001), Termovuoto (Jebrane *et al.* 2018), Les Bois Perdure (Jebrane *et al.* 2018), hot oil (Unsal *et* *al*. 2011; Lacić *et al*. 2014), and hot-pressing methods (Candan *et al*. 2013; Silva *et al*. 2013). Although heat-treated wood possesses improved dimensional properties and fungal resistance, the disadvantage is that thermally modified wood is generally not resistant to termite attacks. Fortunately, this drawback can be overcome by combining the heat treatment with minimal chemical modifications. For instance, a study conducted by Salman *et al.* (2017) shows that the impregnation of vinylic monomers before thermal modification improves the termite resistance of wood. Another downside of thermal modification is heat treatment gives wood a brownish color, which turns into a greyish color when expose to sunlight (Baysal *et al.* 2014). Additionally, strength of wood will decrease depending on the treatment conditions (Mburu *et al.* 2007; Candelier *et al.* 2017). Therefore, thermal treated wood is not recommended for applications where wood strength is a necessity.

Chemical modification

Chemical modification of wood involves forming a stable bond between a reagent and wood cell wall components (lignocellulose). It can be classified as either cell wall modification, filling of wood cell cavities, or a combination (Ormondroyd *et al*. 2015). The main objective is to reduce the overall hydroxyl group content of lignocellulose in the cell walls of woods through these modifications, thereby increasing resistance of the cells to fungi or water. The main advantage of chemical over thermal modification is that mechanical strength of wood is less affected, and the resulting material is harder and denser. Chemical modification of wood, especially acetylation and furfurylation, has been well-studied, and it has consequently been scaled-up for industrial uses (Mantanis 2017).

Acetylation is a process in which the hydroxyl groups (OH) of hemicelluloses and lignin react with an acetyl group (CH_3CO) of acetic anhydride through esterification to form an ester (Kozarić *et al*. 2016). The reaction of hydroxyl groups within the wood cell wall with acetic anhydride is illustrated in Fig. 2 (Homan and Jorissen 2004). The acetylation of wood can be carried out with or without a catalyst, within a temperature range of 100 °C and 130 °C, followed by a vacuum process to remove unreacted volatile reagents or by-products (Obataya and Minato 2008). However, acetylation without the use of a catalyst is still preferable due to cost saving and environmental pollution issues (Ashori *et al*. 2014).

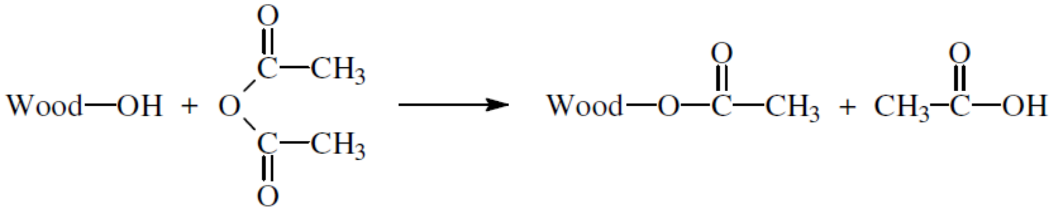

Fig. 2. Reaction of hydroxyl group in wood cell walls with acetic anhydride

Other acetylation reagents such as ketene, vinyl acetate, isopropenyl acetate, and commercial vinegar are often considered as an alternative option to acetic anhydride in acetylation (Jebrane *et al.* 2011b; Azeh *et al.* 2012, 2013; Nagarajappa and Pandey 2016). However, different performances were observed when an assortment of acetylation reagents were used as by-products, whose impact on wood properties were produced after the modification process. For example, non-toxic and volatile acetaldehyde is produced as a by-product after wood is treated with vinyl acetate *via* a transesterification reaction (Cetin and Ozmen 2011). This method gives a comparatively stiffer product, compared to acetylation by means of acetic anhydride (Jebrane *et al.* 2011a).

Because the acetyl functional group is less reactive towards water molecules, acetylation results in a decrease in hygroscopicity of the treated wood. Several studies have demonstrated that acetylation improves the dimensional stability, fungal resistance, photostability, and weathering of treated wood (Özmen *et al.* 2013; Popescu *et al.* 2014; Giridhar *et al.* 2017). Acetylation does not drastically change the wood's initial color and creates a pale color that is stable towards weathering (Gobakken and Lebow 2009). However, the acetylated wood is vulnerable to staining fungi and sensitive to alkali (Gobakken *et al.* 2010)

Furfurylation is another well-known and environmentally friendly modification method. It improves the physical properties of wood by causing cell wall bulking (Sandberg *et al*. 2017). Furfuryl alcohol ($C_5H_6O_2$) is a low molecular organic chemical that has a strong polarity and can be obtained from hydrolysate of agricultural waste (Tathod and Dhepe 2015). Figure 3 demonstrates the polymerization of furfuryl alcohol and its reaction with wood cell wall polymers (Gérardin 2016). Wood is first impregnated with a mixture of furfuryl alcohol *via* vacuum pressure treatment. It is then heated to form a wood polymer composite through *in-situ* polymerization (Li *et al.* 2016).

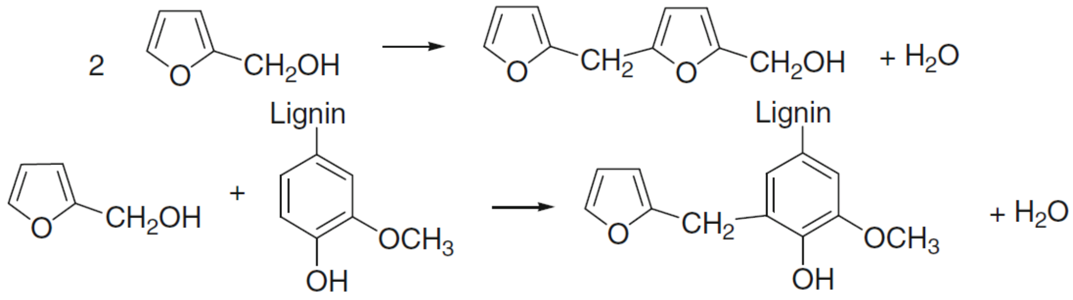

Fig. 3. Polymerization of furfuryl alcohol and its reaction with wood cell wall polymers

A catalyst is essential for controlling the polymerization of furfuryl alcohol (Li *et al*. 2015). Sejati *et al.* (2017) investigated the effects of different catalysts (maleic anhydride, maleic acid, citric acid, itaconic acid, and tartaric acid) on the furfurylation of beech wood (*Fagus sylvatica* L.). The authors suggested that tartaric acid is a promising new catalyst for wood furfurylation. In another study conducted by Yao *et al*. (2017), lignin-derived acids, such as lignosulfonic acid and sulfomethylated lignin acid combined with borate, could also be a promising catalyst system.

With the retention of polyfurfuryl alcohol, the properties of wood such as hardness, resistance to microbial decay and insect attack, modulus of rupture, modulus of elasticity and dimensional stability can be improved (Lande *et al*. 2004). The degree of improvement depends on the loading of polyfurfuryl alcohol. Generally, furfurylation is more suitable for wood species with more open pits that have loose and ordered structures (Dong *et al*. 2016). As furfurylated wood is more rigid than acetylated wood; therefore, it is better suited with applications such as decking and countertops. However, furfurylated wood has a lower dimensional stability than acetylated wood and thus, it is not suitable to be used in joinery products such as windows and doors (Brelid 2013). Furthermore, high hardness is achieved at the cost of increased brittleness of furfurylated wood. In addition, furfurylation also has a greater effect on the color of the wood by darkening it and graying on weathering (Mantanis and Lykidis 2015).

Other than acetylation and furfurylation, impregnation modification is another technique to modify the wood where a monomer is impregnated into wood, and then polymerize, co-polymerize, or cross-link it *in-situ* with a carrier monomer, using either a catalyst or radiation techniques (Kartal *et al.* 2004b; Sun *et al.* 2016). Additionally, the surface of the wood also can be modified to improve its hydrophobicity. For instance, Filgueira *et al.* (2017) demonstrated that the hydrophobicity of the surface of beech wood (*Fagus sylvatica* L.) is improved using water insoluble condensed tannins and hydroxypropyl condensed tannins from *P. radiata* bark, assisted by laccase.

While modifying treatments can usually improve the resistance against fungal attack and mechanical properties of wood, most of them do not provide sufficient long-term protection against wood damaging insects. Therefore, chemical protection from preservatives is still currently regarded as indispensable for wood preservation.

Preservative System

A preservative system refers to any wood preservation treatment that applies wood preservatives (chemical substances) to protect wood against fungi and insects. These preservatives can be distinguished by their respective solvents such as oil-borne, water-borne, and organic solvent-borne preservatives. Each of the preservatives has different characteristic and chemical properties. Thus, when choosing the suitable wood preservative, several factors should be considered such as long-term chemical stability, insignificant effects on wood strength, and safety of the treated wood products to consumers (Ozdemir *et al*. 2015).

Wood preservatives

In general, oil-borne preservatives such as creosote and pentachlorophenol can provide long-term protection (Kitchens and Amburgey 2015). These preservatives have excellent thermal and chemical stability and are highly resistant to leaching as they are insoluble in water. However, these oil-borne preservatives give the treated wood a blackish color and exude a pungent smell (Kang *et al.* 2005). Generally, they are also harmful to humans and the environment (Chen *et al.* 2006). Therefore, oil-borne preservatives are usually only applied on poles, train railways, and other outdoor applications that present no risk of human contact and minimal effects on the environment (Roman 2015).

Water-borne preservatives, which mainly include metallic preservatives that can be solubilized in water, are effective against a wide range of wood-destroying organisms. Chromium copper arsenate (CCA), copper azole, and ammoniacal copper zinc arsenate are the common water-borne preservatives used in market (Rawat *et al.* 2015; Sivrikaya *et al.* 2016). The preservatives are "fixed" onto wood by reacting with or precipitating in treated wood (Sabiha *et al*. 2015). Although these preservatives are odorless and do not affect wood's appearance, metallic preservative additives may cause corrosion of the coating or metal fasteners in wood products (Zelinka 2014; Narciso and Eng 2017). Metallic preservative additives are also easily leached through rainfall, and they represent an environmental hazard because they contain heavy metals (Mercer and Frostick 2012, 2014). Therefore, CCA has been banned in many countries due to arsenic's toxicity (Coles *et al*. 2014).

Recently, organic solvent-borne preservatives such as triazoles and pyrethroids have been developed to replace the toxic metallic preservatives (Volkmer *et al.* 2010;

Tapin-Lingua *et al.* 2016). Preservatives in this class are non-toxic, non-volatile, odorless, and hypoallergenic (Griggs *et al*. 2017). As a result, they are used for indoor applications. Usually, these additives are delivered by using a light organic carrier solvent such as white spirit or petroleum-based hydrocarbon. Additionally, they can be emulsified in solvent-emulsifier mixtures before being dispersed in water (Cui and Preston 2010). Organic solvent-borne preservatives can also work in combination with antioxidants and metal chelators to enhance the biocides' effectiveness and dispensability in water (Schultz and Nicholas 2002). However, the relatively high cost of organic solvents and emulsifiers limits their use, so most of the relevant existing industrial facilities still support the water-based formulations. Additionally, certain organic solvent-borne preservatives are unable to provide long-term protection, as they are easily degraded by sunlight or microorganisms (Zhu *et al*. 2017). Therefore, they are not intended for use where the treated commodity comes into contact with the soil (Cookson 2011).

Environmentally friendly wood preservatives that are naturally formed, such as wood extractives, plant extracts, or biomass, have also been reported (Morard *et al.* 2007; Oramahi *et al*. 2014; González-Laredo *et al*. 2015; Salem *et al*. 2016). For instance, Nakayama *et al.* (2001) reported that resin materials extracted from the guayule plant (*Parthenium argentatum*) have both insect and microbial resistant properties. Another study conducted by Kartal *et al.* (2004a) demonstrated fungicidal and termiticidal properties of filtrates from biomass slurry fuel produced from Sugi (*Cryptomeria japonica*) and Acacia (*Acacia mangium*) wood. The authors stated that phenolic compounds in filtrates could provide resistance against fungi. These natural wood preservatives are biodegradable *via* microorganisms and therefore, it is preferable when it comes to their disposal. However, their availability and economic feasibility have not promoted their extensive use and hence, more efforts are required to commercialize this type of preservative in future.

In the following section, some of the common treatments used to incorporate wood preservatives into wood are outlined.

Wood preservative treatments

There are many methods to treat wood with wood preservatives. The treatment process can be achieved using chemicals and/or specific equipment, with each method having its own strengths and weaknesses.

A pressure treatment uses a combination of vacuum and pressure to force chemical preservatives into the cellular structure of the wood (Salamah and Dahlan 2008; Tripathi and Poonia 2015). By using this treatment, uniform preservative retention and deep penetration are achieved, along with long-term performance and a substantial increase in service lifetime of the wood product. Moreover, preservative retention levels can be controlled by regulating the concentration of the treating solution (Rabbi *et al*. 2015).

Generally four types of pressure processes are used: full cell, modified full cell, empty cell, and double vacuum process (Selamat and Said 1989; Nasheri *et al*. 1999; Islam *et al*. 2008; Winfield *et al*. 2009).

Table 1 compares the different pressure processes commonly in use. Generally, the moisture in the wood is first reduced and the wood is then transferred to a horizontal cylindrical pressure treatment tank. A vacuum or initial pressure is applied before the cylinder is flooded with respective preservative solutions.

Further pressure is then applied to force the solution to diffuse deep into the porous structures of the wood until the required level of preservative retention is achieved. A vacuum is drawn in the final process to clean off any excess chemicals left on the wood's surface. In addition to this conventional pressure treatment, some other modified pressure processes, such as the axial impregnation method and the double-vacuum processes, have also been developed (Mihajlovski and Bahchecandziev 2013; Damay *et al*. 2015).

Table 1. Comparison of Main Features of Pressure Treatments

Features of Different Pressure Treatments
a) Full-cell (Bethell) Process • Both the cell walls and the lumen of wood are filled with chemical. • High retention, but not necessarily deeper penetration, than that of other processes. • Generally used where a large amount of preservative is needed for certain applications, such as treatment of utility poles, farm fences, bridge timbers, and pier timbers.
b) Modified Full-cell Process (Low-weight Schedules) • Uses lower levels of preliminary vacuum and often uses an extended final vacuum. • Residual air in wood expands during the final vacuum to drive out part of the injected preservative solution. • Most common method for treating timber with high moisture content or refractory species with water borne preservatives • Suitable for timber that is difficult to treat when seasoned
c) Empty-cell Process • Deep penetration is obtained with a relatively low net retention of preservative and thus, save the cost of treatment • The preservative is only retained within the cell walls. • Uses initial air under pressure instead of vacuum. • Expansive force of compressed air drives out part of the preservative injected into lumen at the end of the treatment process.
d) Double Vacuum Process (Low-pressure Process) • Wood is first subjected to a short and relatively weak initial vacuum, after which the treatment vessel is flooded with a preservative solution and reduced to normal pressure. • Preservative intake is therefore greatly reduced, compared to other vacuum pressure processes. • Commonly used with light organic solvent preservatives • Used particularly for the impregnation of dry timbers that must retain dimensional accuracy, such as windows and door frames.

Non-pressure processes include brushing, spraying, dipping, and many variations of soaking (Maclean 1946; Killmann and Fink 1996; Hyvönen *et al*. 2005; Ma *et al*. 2013; Khairunnisha *et al*. 2017). Non-pressure processes are conducted under atmospheric pressure, without the use of artificial pressure. The differences between several non-pressure processes is shown in Table 2. Various non-pressure processes differ widely in their penetration and retention levels and in the degree of protection provided by each. Most of the non-pressure treatments, particularly those involving surface applications, generally do not produce a good level of protection in comparison with pressure treatments. Nevertheless, these various non-pressure processes do serve a useful purpose when more thorough treatments are impractical, or when little protection is required.

Table 2. Comparison of Main Features Among Non-Pressure Treatments

Non-pressure Treatments
a) Surface Application • Preservative is applied with a brush, or by dipping or spraying. • Used for preserving small individual items such as window sashes, frames, and other millwork that does not contact the ground and is exposed to moisture for only brief periods.
b) Gedrian's Bath (Hot and Cold Bath) • Wood is immersed in a bath of preservative, heated (60 to 110 °C) for a few hours, and then allowed to cool (30 to 40 °C) while the wood is still submerged in the liquid. • During the heating period the air in the cells expands and much of it is expelled as bubbles. • When the woods are changed to the cold bath (the preservative can also be changed), a partial vacuum is created within the lumen of the cells, causing the preservative to be drawn into the wood.
c) Sap Displacement Method • Only applies to round timbers in green condition. • Uses hydrostatic pressure due to gravity to force preservative from the butt end of the round timber. • A cap is fitted to the butt end of a freshly sawn pole or round timber. One end of a flexible tube is connected to the cap and the other end to a tank containing the preservative located at a higher place.
d) Diffusion Method • Diffusible water-borne preservatives are used (*e.g.* sodium arsenate, copper sulphate, and zinc chloride). • Preservatives diffuse into wood from the treating solution. • Deep penetration of the preservative is achieved without the need for expensive equipment, but this method requires a long storage time and the treated wood is susceptible to leaching. • The double-diffusion process can overcome this leaching issue. In this process, wood is sequentially soaked in two aqueous chemical solutions. Leaching is ultimately prevented by a precipitate that forms upon the reaction with the wood matrix that is highly resistant to leaching and toxic to fungi and termites (Pavia 2006).

Although many wood preservatives have been developed, only a few of them have been implemented in current commercial treatments. This is because most of the established wood treatment plants use water-based preservative treatments for practical reasons. Hydrophobic biocides, for example, are less preferable to industries due to their relatively high cost, which is caused by the use of an emulsifier or organic solvent. In contrast, hydrophilic biocide also faces the excessive leaching problem. For these reasons, researchers are currently proposing the utilization of nanotechnology to overcome the shortcomings in the present methods of wood preservation.

NANOTECHNOLOGY IN WOOD PRESERVATION

Nanotechnology has become a popular topic recently. Figure 4 shows the number of publications based on the Scopus search engine, where the keywords used were nano and wood. Based on the graph, it can be observed that there has been an increasing trend in the number of publications on wood-related nanotechnology. The number of publications increased more than 15-fold from year 2007 to 2017 (from 6 to 93). This shows that this field is getting more attention from researchers and industry.

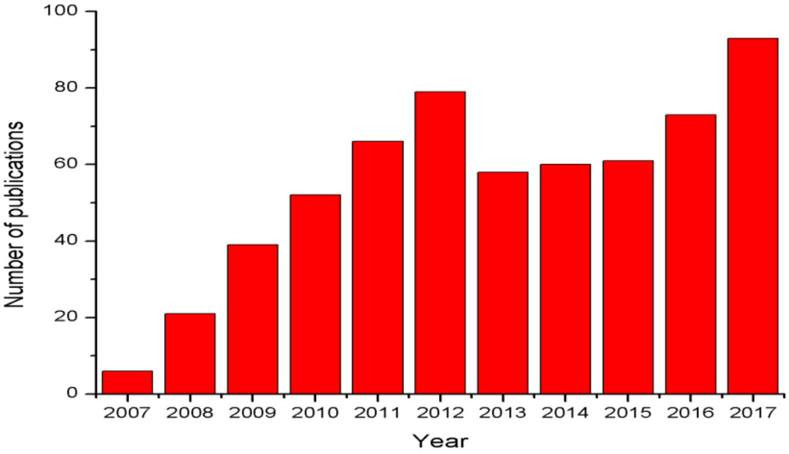

Fig 4. Number of wood-related nanotechnology publications based on Scopus from the year 2007 to 2017

Nanotechnology shows great potential to be introduced into wood preservation to overcome problems associated with the existing methods of wood preservation. Nanomaterials have several advantages, such as the capacity to treat a large effective surface area, high dispersion stability, and presence of a reservoir effect that is capable of providing long-term protection (Clausen 2012; Mattos *et al*. 2017). Currently, the application of nanotechnology in wood treatment is achieved *via* three approaches, namely the direct impregnation of nanosized biocide into wood, the controlled release of biocides embedded in a nanocarrier, and wood modification. Such applications can be achieved by using a variety of nanomaterials such as nanosized metal, polymeric nanocarriers, nanotubules, and other nanomaterials.

Nanosized Metals

In recent years, micronized copper systems have seen commercial success in the USA since launching commercially in 2006, with more than 75% of the residential lumber produced being treated with these systems (Civardi *et al*. 2016). Copper compounds, such as copper(II) carbonate, can be micronized through mechanical grinding using a commercial grinding mill (Zhang and Leach 2013). Suitable nanosized material can also be obtained through chemical means with a co-biocide to enhance its efficacy. Nanosized metal can be synthesized using different biological and chemical approaches as shown in Table 3 (Reddy *et al.* 2011; Golinska *et al.* 2014; Habibi and Karimi 2014; Malviya and Chattopadhyay 2015; Thandavan *et al.* 2015; Wang *et al.* 2015; Geetha Devi and Sakthi Velu 2016; Hong *et al.* 2016; Jeevanandam *et al.* 2016; Poletti Papi *et al.* 2017;

Rajeshkumar and Bharath 2017; Gupta and Srivastava 2018). As is well known, the synthesis methods of metal nanoparticles play a very significant role in determining the physicochemical characteristics of nanosized metals (*e.g.*, size, dispersity, morphology, surface energy, and crystal structure, *etc.*) (Stankic *et al*. 2016).

Table 3. Example of Preparation Methods for Metal Nanoparticles

Preparation Methods of Metal Nanoparticles		
Solution-based synthesis	Vapor-based synthesis	Biological synthesis
Sonochemical Co-precipitation Solvothermal Sol-gel Microwave-assisted solvothermal Microemulsion Laser ablation	Chemical vapor deposition Combustion Template/surface-mediated	Fungi Plants

There are currently two commercially available nano-micronized copper systems, namely micronized copper quaternary (MCQ), where dimethyldidecylammonium carbonate/bicarbonate is used as a co-biocide, and micronized copper azole (MCA), where tebuconazole or a combination of tebuconazole-propiconazole is used as a co-biocide (Freeman and Mclntyre 2008; Civardi *et al*. 2015). While traditional alkaline copper preservatives are solubilized in aqueous ethanolamine solution, nano-micronized copper compounds are dispersed in water and then the suspension is used to treat the wood with conventional pressure treatment (Kartal *et al*. 2014; Xue *et al*. 2014). Furthermore, the fixation mechanisms of conventional copper systems rely on chemical reactions, such as chelate formation and ion exchange, to form insoluble complexes in treated wood (Temiz *et al*. 2014). However, based on the observation of Matsunaga *et al.* (2009), the fixation of nano-micronized copper occurs primarily through deposition in pit chambers and on tertiary cell wall layers.

Differences in size may influence the extent to which the nano-micronized copper particles can penetrate the microstructure of wood and enhance biocidal action on fungus and termites (Jin *et al*. 2008). Civardi *et al*. (2015) described the fungicidal mechanisms of copper-based nanoparticles in their study. In some cases, Cu-tolerant wood-destroying fungi may not be able to recognize copper nanoparticles. Once nanoparticles enter fungal cell walls through endocytosis or diffusion through the membrane, they form a reactive oxygen species, or have disruptive effects on mitochondria, proteins, and deoxyribonucleic acid (DNA) within the fungus cell. The nanoparticles may also undergo dissolution, and thereby interfere with homeostatic processes within the fungal cell.

Moreover, in a study done by Mclntyre and Freeman (2008), nano-micronized copper quaternary formulation significantly outperformed (achieving a better rating) amine copper quaternary formulations in a five-year field stake test in Finland. This outcome was further verified by a study by Akhtari and Nicholas (2013). The authors found that nano-micronized copper formulation can greatly reduce the weight loss of wood due to termite attack from 46.8% to 0.2%, which is more effective than amine copper formulations (1.0%). Wood treated with micronized copper is less corrosive to metal fasteners and is lighter in color (Kofoed and Ruddick 2010). Additionally, nano-micronized copper systems have proven qualities, such as reduced leaching and reduced selective adsorption of active ingredient, and work effectively in field tests (Freeman and Mclntyre 2013).

Other than copper-based compounds, nano-metals, such as zinc, zinc borate, zinc oxide, titanium dioxide, and silver, have also been utilized on wood (Bak *et al*. 2012; Mantanis *et al*. 2014; Taghiyari *et al*. 2014; Lykidis *et al*. 2015; Harandi *et al*. 2016; Nair *et al*. 2017). Both the mechanical properties and biological efficacy of wood treated with these nanometals have been well-studied and reported in the literature (Akhtari *et al.* 2012; Akhtari and Arefkhani 2013; Lykidis *et al.* 2013). For example, Akhtari and Ganjipour (2013) investigated and compared the effects of nanosilver, nanocopper, and nanozinc oxide on the resistance of Paulownia (*Paulownia fortunei*) wood against white rot fungus (*Coriolus versicolor*). Wood specimens were impregnated with a 400-ppm aqueous suspension of nanoparticles with particle sizes ranging from 10 to 80 nm. A chemical retention of 0.14 kg/m^3 was achieved for all formulations. The results showed that nanosilver, nanocopper, and nanozinc oxide significantly increased the decay resistance of Paulownia against *C. versicolor* by reducing the weight loss of wood from 28% to 2%.

In addition, the effects of nanosilver on white-rot (*Trametes versicolor*) and brown-rot (*Lenzitesacuta*) fungi of several tropical wood species were studied by Moya *et al.* (2017). The treated wood showed high resistance to *T. versicolor* and moderate resistance to *L. acuta*. These studies demonstrated that the nanometals can provide sufficient biological resistance to the treated woods. Other than providing biological efficacy to treated wood, a study conducted by Lotfizadeh *et al.* (2012) revealed a higher drying rate both above and below the fibre saturation point in nanometal impregnated boards. It may then be indicated that nanometal may have the potential to improve drying conditions and decrease drying stresses in convective kilns.

Polymeric Nanocarriers

Polymeric nanocarriers also present the industry with interesting properties in improving the impregnation of wood preservatives. For instance, a polymeric nanocarrier loaded with a hydrophobic active ingredient has excellent colloid dispersity in water (Li and Huh 2014). Furthermore, polymeric nanocarriers are also able to protect the hydrophilic active ingredient from excessive leaching (Peteu *et al*. 2010).

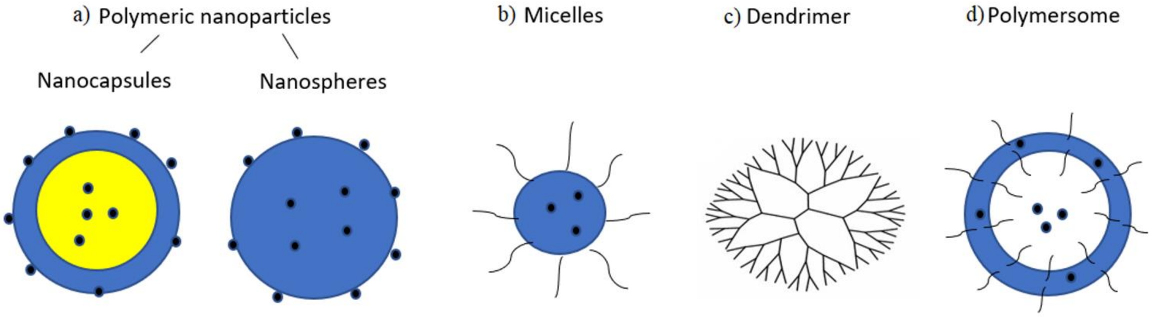

Fig. 5. Types of polymeric nanocarriers for active ingredient delivery. (a) Polymeric nanoparticles: where active ingredients are conjugated to or are encapsulated in polymers. (b) Polymeric micelles: amphiphilic block copolymers that form to nanosized core/shell structure in aqueous solutions. The hydrophobic core functions as a reservoir for hydrophobic drugs, whereas the hydrophilic shell region stabilizes the hydrophobic core and renders the polymer water-soluble. (c) Dendrimers: synthetic polymeric macromolecules of nanometer dimensions, which are composed of hyperbranched polymers that emerge radially from the central core. (d) Polymersome: composed of hydrophilic–hydrophobic block copolymers, arranged in a lipophilic bilayer vesicular system, and with a hydrophilic inner core

Figure 5 shows several types of polymeric nanocarrier that can be used for active ingredient delivery (Mora-Huertas *et al.* 2010; Lee and Feijen 2012; Moreno-Vega *et al.* 2012; Noriega-Luna *et al*. 2014; Zhang *et al.* 2014). Although polymeric nanocarriers have been widely applied in the pharmaceutical sector in controlled drug delivery systems, they have received far less attention from researchers in the wood preservation sector as compared to the attention given to nanometal preservatives.

Encapsulation of the active ingredient into polymeric nanocarriers can be conducted through several techniques such as nanoprecipitation (Gu *et al.* 2015; Sosnik and Raskin 2015; Martínez Rivas *et al.* 2017), emulsion-diffusion (Lee *et al.* 2013), double emulsification (Nabi-Meibodi *et al*. 2013), emulsion-coacervation (Chirio *et al*. 2011), layer-by-layer method (Chai *et al*. 2017), and other techniques (Nagavarma *et al*. 2012; Kulhari *et al*. 2015; Khoee *et al*. 2018).

Liu *et al.* (2001) successfully incorporated biocides (tebuconazole and chlorothalonil) into polyvinylpyridine (PVP) and polyvinylpyridine-co-styrene (PVP-co-St) nanoparticles using a simple and facile impregnation method, where a median particle diameter between 100 and 250 nm was obtained (Liu *et al*. 2001, 2002a). In their studies, sapwood of southern yellow pine (SYP) and birch were treated with active ingredient-containing nanoparticles *via* impregnating in an aqueous suspension of nanoparticles followed by conventional pressure treatments. The treated wood was exposed to brown rot (*Gloeophyllum trabeum*) and white rot wood decay fungus (*Trametes versicolor*) for 55 days. The weight losses of wood after 55 days are given in Table 4.

Table 4. Fungi Resistance of SYP and Birch Treated with Different Formulations of Nanoparticles

Fungi	Wood	Biocide	Matrix	Weight loss (%) at Different Active Ingredient Suspension Loadings (kg/m³)			
				0.0	0.1	0.4	0.8
T. versicolor	Birch	Tebuconazole	PVP	43.0 (0)	18.6 (0)	13.0 (0)	7.0 (1)
			PVP-*co*-10% St	36.0 (0)	23.0 (0)	12.8 (0)	10.4 (0)
			PVP-*co*-30% St	35.0 (0)	7.0 (0)	6.1 (0)	7.0 (0)
			PVP/HBP	37.0 (0)	28.0 (8)	7.0 (3)	7.0 (4)
		Chlorothalonil	PVP	43.0 (0)	20.1 (0)	14.6 (0)	12.4 (1)
			PVP-*co*-10% St	35.0 (0)	21.7 (0)	14.0 (0)	10.5 (1)
			PVP-*co*-30% St	35.0 (0)	15.4 (0)	11.5 (0)	10.6 (0)
			PVP/HBP	37.0 (0)	21.0 (4)	14.0 (7)	10.0 (9)
T. versicolor	SYP	Tebuconazole	PVP	17.0*	7.0*	3.7*	2.1*
		Chlorothalonil	PVP	18.5*	15.6*	12.7*	11.1*
G. trabeum	SYP	Tebuconazole	PVP	17.0 (3)	7.0 (1)	4.0 (2)	2.0 (1)
			PVP/HBP	10.0 (3)	6.1 (1)	3.1 (1)	2.8 (1)
		Chlorothalonil	PVP	17.0 (3)	16.0 (0)	13.0 (0)	11.0 (0)
			PVP/HBP	10.0 (0)	9.0 (4)	7.0 (1)	5.0 (2)

The standard deviations are in parentheses.
*Standard deviation is not provided in original papers

Table 4 shows the fungi resistance of SYP and birch treated with different formulations of nanoparticles based on the study of Liu *et al.* (2001). The most interesting observation was that the active ingredient-containing nanoparticles were found to provide good resistance against fungal attack on treated wood, even at very low levels of active ingredient incorporation (0.1 to approximately 0.8 kg A.I/m^3). This observation indicated that an active ingredient introduced into wood using the nanoparticles had unexpectedly high activity. Such unique properties have the potential to reduce cost and minimize the side effects of active ingredients on non-target organisms. However, the cause of this observation is still unknown.

Additionally, the same authors, Liu *et al.* (2001) also studied and compared the efficacy of different biocide-containing nanoparticles against wood decay fungi as shown in Table 4, where the nanomaterial matrices were made up of polyvinyl chloride (PVC), PVP, PVP-co-St, and blends of PVP/hyperbranched polyesters (HBPs) (Liu *et al*. 2002a). Different biocides, such as 4,5-dichloro-2-n-octyl-4-isothiazolone (RH-287) and chlorpyrifos, were included in these robustness studies. The method was generally robust where nanoparticles could be prepared from several different polymers, copolymers, and polymer blends. However, it was found that decreasing the hydrophilicity of polymers decreased the active ingredient release rate, and concurrently increased the size of the nanoparticles. In contrast, the delivery efficiency of nanoparticles on wood decreased with increasing suspension loading and matrix hydrophobicity. The delivery efficiency of birch was also lower than that of SYP, because birch has smaller pit pores than SYP. Large-sized nanoparticles blocked the pit pores of wood and thus, prevented the further introduction of nanoparticles into the wood. Undelivered nanoparticles were found to have undergone aggregation. Greater aggregation occurred in the more hydrophobic formulations than in the hydrophilic formulations.

In another work by Liu *et al.* (2002b), a surfactant-free method was used to synthesize active ingredient-containing polymeric nanoparticles. In this work, the authors used a self-stabilization method (eliminated the surfactant component) by preparing a nanoparticle matrix *via* a free radical initiator. In this method, the initiator fragments serve to ionically stabilize the nanoparticles. They also incorporated additional polar or potentially ionic moieties, such as acrylic acid or methacrylic acid *via* a copolymerization method to further stabilize the nanoparticles. The team found that surfactant-free formulations afforded nanoparticles with significantly smaller median particle diameters and more stable aqueous suspensions (6 months) than their surfactant-stabilized counterparts (few weeks). Although surfactant-free suspensions had significantly broader particle size distributions, the surfactant-free nanoparticles were still delivered more efficiently into the wood than the surfactant-stabilized formulations. The surfactant-free nanoparticles appeared to afford more resistance to decay than the surfactant-stabilized formulations. The SYP lost < 5% of its mass after 55 days of exposure to *G. trabeum* when the active ingredient content in the wood was only 0.4 kg/m^3, while an active ingredient content of 0.8 kg/m^3 in birch wood was sufficient to bring its mass loss to < 5% after 55 days of exposure to *T. versicolor.* This observation was ascribed to faster active ingredient release from the surfactant-free nanoparticles than those stabilized with the surfactant, which would allow the active ingredient to reach threshold levels more rapidly.

Other potential nanocarriers

Nanotubules can be a promising material for use as a carrier for biocides, due to their hollow structure and high contact surface area. However, it appears that no research has been done on the combination of other nanocarriers with biocide in wood preservation.

Carbon nanotubes (CNTs) are one of the best known nanotubules. The CNTs are the third allotropic form of carbon-fullerenes that are rolled into cylindrical tubes (Kushwaha *et al*. 2013). They offer several advantages that include high carrying capacity, high biocompatibility, and high surface area to volume ratio (Rastogi *et al*. 2014;

Tavakolifard *et al*. 2015). Currently, the chemical and physical properties of CNTs, such as water dispensability, cytotoxicity, and active ingredient loading, can be improved through functionalization (Heister *et al*. 2012). Surface functionalization of CNTs may be covalent or non-covalent. The backbone or sidewall of CNTs can be covalently modified with hydrophilic functional groups (Cheng *et al*. 2011; Azqhandi *et al*. 2017). Meanwhile, non-covalent functionalization exploits favorable interactions between the hydrophobic domain of an amphiphilic molecule and the CNTs surface. Shao *et al*. (2015) worked on a novel single-walled carbon nanotube-based delivery system for an antitumor agent, Paclitaxel. This nanocarrier exhibited excellent intracellular drug delivery properties in cancer cells.

Another potential carrier is halloysite. Halloysite is a naturally occurring aluminosilicate clay nanotubular material. Aside from its low cost, it is routinely utilized in the sustained release of chemical agents in pharmaceutical areas due to its non-toxicity. (Rabisková 2012; Lvov *et al*. 2016). The release rate of active agents can be controlled by adjusting inner lumen of halloysite and clogging the tube ends with end-stoppers such as calcium phosphate (Cavallaro *et al*. 2017). Scarfato *et al.* (2016) developed a halloysite nanotube-based carrier with a commercial biocide, namely Biotin T, for the protection of construction materials. The mortar containing the biocide-loaded nanotubes showed reduced water capillary absorption and prolonged resistance to microbiological growth after being exposed to natural contamination in outdoor conditions for two years. The authors attributed these results to the treatment's prevention of leaching and environmental degradation, a phenomenon resulting from the entrapment of the active agents within the halloysite nanotubes.

Furthermore, there are many other nanocarriers that can be used, such as montmorillonite clay (Wanyika 2014), calcium carbonate (Qian *et al*. 2011), titanium dioxide (Wang *et al.* 2016a), titanium alloy (Doadrio *et al*. 2015), and silica nanoparticles (Khamsehashari *et al*. 2018). All these nanocarriers exhibited superior performance in drug controlled-release formulation, albeit in the pharmaceutical sector. It is obvious that the application of nanotechnology in wood preservative requires more attention from researchers and industry, if the development of a safer, more efficient, and low-cost biocide delivery system is to become a reality.

Wood Modification (Coating Treatments)

Employing nanotechnology in wood modification, especially coating treatment, also can result in next-generation products having hyper-performance and superior service ability. The coating treatment can be done through physical or chemical approach. In physical approach, pre-synthesized nanomaterials can be used directly or added into existing wood coating. The nano-based coating is then applied on wood surface by spraying coating, brushing or dipping (Rassam *et al.* 2012; Havrlik and Ryparová 2015). The applied nanomaterials also can be *in-situ* synthesized on wood surface by chemical reactions such as hydrothermal method and sol-gel deposition methods (Wang and Piao 2011; Liu *et al.* 2015). The chemical approach offers an advantage by addressing distribution and interaction issues compared to physical approach (Mishra *et al.* 2017).

One of the popular coating materials in wood preservations is the development of hydrophobic surfaces on wood. The nanomaterials can be act as water repellents (control the rate of water sorption) and/or as dimensional stabilizers (control swelling from moisture sorption). For example, Soltani *et al*. (2013) treated beech wood (*Fagus orientalis*) with zinc oxide nanoparticles. Their study showed there was a reduction in water absorption and volumetric swelling which suggested that nano-ZnO provided substantial water resistance and dimensional stability.

Moreover, superhydrophobic surface with a water contact angle higher than 150° have recently attracted attention. Nanomaterials such as silica-polymer nanocomposites (Chang *et al.* 2015), tungsten trioxide (Sun and Song 2018), titanium dioxide nanoparticles (Pánek *et al.* 2017) and zinc oxide nanorods (Wang *et al.* 2011) were used to achieve this superior ability. As water droplets cannot adhere to such superhydrophobic surface but easily roll off, it can take away pollutants and thus, exhibits self-cleaning effects. For example, a transparent self-cleaning surface for cellulose-based materials was fabricated by Wei *et al.* (2018). In their study, silicon dioxide nanoparticles were first modified with poly-(dimethylsiloxane) and (heptadecafluoro-1,1,2,2-tetradecyl) trimethoxysilane. The modified SiO_2 nanoparticles were then well-distributed on the microscale rough surface of the cellulose-based materials by spray-coating to form a micro/nano two-tier structure. Consequently, the spray-coated superhydrophobic surface can resist water, dust, and thoroughly prevent fungal attachment to treated wood (Yao *et al.* 2017).

Furthermore, wood polymers in surface of natural wood will undergo dissociation and cause rapid color changes when exposed to solar radiation. In a study conducted by Salla and co-authors (2012), maleic anhydride graft polypropylene (MAPP) based coating was blended with 7.5% of zinc oxide nanoparticles. Rubberwood (*Hevea brasiliensis*) specimens were treated with the coating and then exposed to UV light to assess its effectiveness for protection of wood against UV degradation. MAPP contains acid anhydrides groups that can associated with wood constituents by reacting with hydroxyl groups of wood. In addition, polypropylene content in MAPP may make wood surfaces hydrophobic. The photostability test showed dispersion of ZnO nanoparticles in MAPP restricted the color changes and photodegradation of wood polymers. This UV-absorbing material is essential for outdoor applications to increase their effective operation life or durability.

Additionally, nano-based coatings with strong bactericidal properties such as titanium dioxide nanoparticles (Zuccheri *et al*. 2013), polymers (Du *et al.* 2001; Ong *et al.* 2006), and zinc oxide nanoparticles (Nosáľ and Reinprecht 2017) also can provide decay resistance to wood products. There are also other nano-based coatings that can improve scratch and abrasion resistance (Kanokwijitsilp *et al.* 2013), and fire retardancy (Soltani *et al.* 2016) of wood.

Although these nano-based coatings provide exterior protection to wood, maintenance frequency and their appearance need to be take into consideration. The coating also needs to be flexible and has strong adhesion with wood as exterior wood shrinks and swells with moisture changes. Both short- and long-term effects of nano-based coatings is important for the safety of consumers.

Risk Assessment of Nanomaterials

The application of nanotechnology in wood preservation has raised concerns over the potential for human exposure to nanoparticles as well as their release into the environment (Seaton *et al.* 2010; Schrand *et al.* 2010). Recently, ecotoxicological studies for nanomaterials especially metallic nanoparticles such as silver (Sambale *et al.* 2015), zinc oxide (Ng *et al.* 2017), titanium dioxide (Iavicoli *et al.* 2012), and copper (Civardi *et al.* 2015) have been published. These literatures are essential for developing standardized risk assessment methods for wood preservation industries. The nanomaterials are likely to accumulate in soils and sediments and reach a critical concentration over time when nano-treated wood products are disposed into the environment (Auffan *et al.* 2014; Wang and Nowack 2018).

Furthermore, effect of these nanomaterials on human health will still remain as a critical question with the commercialization of nanotechnology. As such, these nanoparticles may be inhaled or ingested by consumers when they are in contact with the nano-treated wood surfaces. For instance, copper-based nanoparticles which are the most common nano-based formulation used in wood preservation market today are hazardous when being inhaled and will mainly exert their toxicity on the respiratory tract of humans (Hristozov *et al.* 2018). In addition, these nanoparticles can interact with the cell membrane and eventually penetrate it (Navya and Daima 2016; Contini *et al.* 2018). Besides, nanoparticles can also be transported *via* lymphatic and circulatory systems to different tissues and organs, where accumulation can result in severe injuries and damage to living cells (Blanco *et al.* 2015). For example, zinc oxide nanoparticles can interfere with the antioxidant defense mechanism of cells by inducing oxidative stress that lead to blood–brain barrier dysfunction (Feng *et al.* 2015; Saliani *et al.* 2016).

Even though a number of reports have been published lately, the actual exposure level of nanoparticles is difficult to be determined, leading to inconclusive findings. This problem has become more apparent along with other challenges such as difficulties in data generation as well as the quantitative determination of risks (Lee *et al.* 2010). Moreover, nano-based formulation is not adherently more dangerous than conventional formulation. In a study conducted by Platten III and co-workers (2016), the dermal release of copper and copper particles was examined from the surfaces of lumber pressure-treated with micronized copper. The authors found that the micronized copper azole and copper azole formulations released similar quantities of total copper, resulting in similar exposure levels. Therefore, more studies are required to provide regulators a scientific foundation for environmental and human health policy regarding the application of nanoparticles in wood products in the future.

CONCLUSIONS

Wood preservation can be achieved by treating wood with various wood preservatives or by forcing it to undergo thermal or chemical modification. Generally, wood preservatives can provide biological resistance, while wood modification can enhance physical properties of wood by altering the chemical structure of the lignocellulose component. Suitable wood preservation methods by necessity are adopted based on the end-product applications.

Nanotechnology has been observed to have a great potential for wood preservation applications. The use of nanosized metal preservatives allow for deeper penetration and a more homogenous uptake of particles in the wood. In addition, the incorporation of biocides in nanocarriers allows biocides to be safely stored within the interior of nanoparticles, where loss due to factors, such as leaching and the random degradation of biocides, can be avoided. Therefore, the development of more effective, safe to use, and environmentally friendly preservative formulations are encouraged to safeguard the environment in the long run. Moreover, nano-based wood coating can provide exterior protection such as UV resistance and hydrophobicity for wood. In short, it can be concluded that nanotechnology is likely to have a major impact on the wood protection industry, through the future design of nanomaterials with the necessary unique properties to enhance the performance of wood preservatives, prolonging the wood product service life. However, consideration towards the environmental impact and potential health risk of the nanotechnology is essential and crucial to ensure this emerging market is sustainable.

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