Synthesis and Characterization of Zinc Oxide Nanoparticles for Wood Protection Application

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A dissertation submitted in partial fulfilment of the requirements for the Degree of Bachelor of Science with Honours

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Declaration

I hereby declare that no portion of this dissertation has been submitted in support of an application for another degree of qualification of this or any other university or institution of higher learning.

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<tr>
<td>AAS</td>
<td>Atomic Absorption Spectroscopy</td>
</tr>
<tr>
<td>ACA</td>
<td>Ammoniacal Copper Arsenate</td>
</tr>
<tr>
<td>ACQ</td>
<td>Ammoniacal Copper Quaternary</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
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<tr>
<td>AWPA</td>
<td>American Wood Protection Association</td>
</tr>
<tr>
<td>CCA</td>
<td>Copper-Chrome-Arsenic</td>
</tr>
<tr>
<td>EN 84</td>
<td>Wood preservative: Accelerated ageing of treated wood prior to biological testing. Part 2: Leaching procedure</td>
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<tr>
<td>FRIM</td>
<td>Forest Research Institute Malaysia</td>
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<tr>
<td>FTIR</td>
<td>Fourier Transform Infrared Spectroscopy</td>
</tr>
<tr>
<td>HMTA</td>
<td>Hexamethylene tetramine</td>
</tr>
<tr>
<td>HRTEM</td>
<td>High Resolution Transmission Electron Microscopy</td>
</tr>
<tr>
<td>ICP-AES</td>
<td>Inductively Coupled Plasma Atomic Emission Spectroscopy</td>
</tr>
<tr>
<td>mm</td>
<td>Millimetre</td>
</tr>
<tr>
<td>Na(DEHSS)</td>
<td>Sodium- di-2-ethylhexylsulfosuccinate</td>
</tr>
<tr>
<td>PAA</td>
<td>Polyacrylic acid</td>
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<tr>
<td>PIXE</td>
<td>Proton-induced X-ray Emission</td>
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<tr>
<td>Abbreviation</td>
<td>Description</td>
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<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
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<td>TEM</td>
<td>Transmission Electron Microscope</td>
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<tr>
<td>UV</td>
<td>Ultra-Violet</td>
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<tr>
<td>WPCs</td>
<td>Wood polypropylene composite</td>
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<td>XRD</td>
<td>X-ray Diffraction</td>
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<td>ZnO NPs</td>
<td>Zinc oxide nanoparticles</td>
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</table>
Mean ZnO nanoparticles size in mixture of distilled water and ethanol at different ratio.

SEM micrographs of Jelutong wood at tangential side (a) and (b) untreated and (c) and (d) treated with ZnO nanoparticles.

SEM micrographs of Jelutong wood at side surface (a) and (b) untreated and (c) and (d) treated with ZnO nanoparticles.

EDX spectrum of synthesized ZnO nanoparticles.

TEM micrographs of ZnO nanoparticles, temperature 50°C (insert a & b) temperature 90°C (insert c & d).

Graph of Absorbance at 357 nm versus Time (day 1 to day 5).

FT-IR spectrum of ZnO nanoparticles prepared by zinc chloride and distilled water at (a) 50°C and (b) 90°C for three hours respectively.

FT-IR spectrum of ZnO nanoparticles prepared by zinc acetate and distilled water at (a) 50°C and (b) 90°C for 3 hours respectively.
FT-IR spectrum of ZnO nanoparticles prepared by zinc acetate with the mixture of distilled water and ethanol in ratio (a) 7:3 (b) 1:1 (c) 8:2 at 90°C for 3 hours.

Mean zinc concentration in wood blocks determined by AAS using acid digestion method.

Zinc leaching (ppm) from nano-zinc oxide treated blocks for different fixation periods.

Mean cumulative zinc leaching (ppm) from nano-zinc oxide treated blocks for different fixation periods.

Mean cumulative zinc leaching (ppm) from nano-zinc oxide treated blocks by EN 11-06 method.
Synthesis and Characterization of Zinc Oxide Nanoparticles for Wood Protection

Application

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ABSTRACT

Zinc oxide has a wide range of applications. Many methods have been developed to prepare ZnO nanopowders which is very costly. Copper-Chrome-Arsenic (CCA) wood preservatives currently being used in Malaysia is not environmental friendly. Leaching of CCA preservatives from treated woods causes environmental pollution. The objectives of this study are to prepare ZnO nanoparticles and to evaluate the effectiveness of zinc oxide nanoparticles for wood protection application. ZnO nanoparticles of hexagonal shape and controlled sizes were synthesized using the sol-gel process with optimized synthesis conditions. ZnO nanoparticles were characterized by SEM, EDX, TEM, and FTIR spectroscopy. The mean size of ZnO nanoparticles was determined to be 130-190 nm in diameters. ZnO colloidal suspension exhibited strong absorption peak at the wavelength of 357 nm. The mean of zinc concentration uptake after the wood blocks treated with ZnO nanoparticles was 456±17.36 ppm. The leaching rate of ZnO nanoparticles from Jelutong wood blocks for the 1-week and 2-week fixation periods were 45% and 21%, respectively. Jelutong wood blocks showed a better retention of ZnO nanoparticles for two weeks fixation period. The potential application of ZnO nanoparticles for Jelutong wood protection was evaluated.

Key words: ZnO nanoparticles, SEM, wood protection

ABSTRAK


Kunci perkataan : Nanopartikel ZnO, IEM, perlindungan kayu
1.0 Introduction

Wood played a crucial role in human life from the beginning of civilization, mainly due to its availability, aesthetic value and its processing properties. It is, therefore, not a surprise that wood has an important place in our cultural heritage. Besides, wood is used in both interior and exterior applications because of its renewability, nice appearance and low cost (Morell et al., 2006). However, wood is dimensionally unstable and continuously vulnerable to deterioration caused by fungi and insects. The special characteristic properties of nanomaterials motivated scientists to explore the simpler together with cheap methods to synthesis nanostructures with advance technology (Shah & Shahry, 2009). Steel, concrete and aluminium have higher energy requirements and material costs in the production process which cause serious water and air pollution when apply to wood (Freeman, 2003). Old wooden objects are always being affected by biological degradation which breaks their structural integrity and weaken their mechanical properties (Evans, 2008). Zinc oxide is crucial because of its wide range of applications. Nano zinc oxide were synthesized using soluble starch and water as a medium and was impregnated onto fabrics which was in cotton form to shield against UV-protection and also inhibit antibacterial activity (Yadav et al., 2006). Besides that, nanotechnology which is unique in textile industry is used rapidly and frequently to improve various properties of textiles especially silver and polypropylene nanocomposites as reported by Xin et al. (2003). Therefore wood preservative is needed to keep wood safe from deterioration by rot, insects or water. Large and various number of different types of chemicals and mechanical processes used to preserve wood. Zinc is a metal that is a key wood preservative. American Wood Protection Association (AWPA) (2010) stated that zinc oxide has large potential as the preservative component of coatings. The most common method used to protect wood from deterioration is surface coating. This method can also be used to improve and
stabilize its distinctive appearance (Evans, 2008). However, even if the surface is being coated with clear coatings of durable properties such as polyurethane coatings, wood photodiscoloration is unavoidable while nano-zinc oxide can improve photostability.

Zinc oxide nanoparticles which acts as a UV stabilizer in coatings and as a wood preservatives had been reported in several previous studies (Auclair et al., 2011). Besides, recent research showed that metal oxide UV absorbers can be used in the form of transparent films and clear coatings (Yang et al., 2005). The growth of the fungi was inhibited by zinc oxide nanoparticles by affecting their cellular functions. This happened because the mycelial mats were deteriorated. Besides that, zinc oxide will also cause the deformation of fungal mats and therefore inhibit the growth of various bacteria (Bouwmeester et al., 2009). The optical transparency of nanoparticulate zinc oxide permits its use in a wide range of applications. Zinc oxide exhibits antibacterial purposes (Vigneshwaran et al. 2006). For the interaction with prokaryotes and eukaryotes system, nano-materials often exhibit physiochemical properties. Anyway, there are few reports regarding impregnating wood with nanometals. If nanotreatment is leach resistance, treated wood with zinc oxide nanoparticles provides advantages for long term nano-coatings of protection from photo-degradation and biological deterioration.

Studies have shown that with the reduction of particles size, would lead to increase of antibacterial activity and the screening efficiency of zinc oxide. This allows for reduced loadings when compared to larger particles sizes (Ammala et al., 2002). However, various type of wood were treated with a specific size of zinc oxide nanoparticles in previous study and the effect of nanoparticles sizes could not be determined. The concentration of precursors, effect of reaction temperature, and the growth of nanoparticles plays crucial role on its properties. The method used to synthesis zinc oxide nanoparticles should has several advantages. Systematic studies was required to optimize the efficiency of the
conditions in order to obtain nanoparticles with desired dimensions. Thus, the aim of this study was to prepare ZnO nanoparticles and to optimize the efficiency of zinc oxide nanoparticles for wood protection.

1.1 Problem Statement

Inorganic types of Copper-Chrome-Arsenic (CCA) preservatives currently being used for the protection of wood is not environmental friendly (Hingston et al., 2001). Leaching of CCA preservatives from treated woods causes environmental pollution. Therefore, research on zinc oxide nanoparticles fixation and its leachability from the wood will be carried out to evaluate the effectiveness of zinc oxide nanoparticles preservatives for wood.

1.2 Objectives of Study

1. To synthesize ZnO nanoparticles by using suitable synthesis approaches.

2. To characterize the chemical and physical properties of ZnO nanoparticles prepared.

3. To evaluate the effectiveness of ZnO nanoparticles for wood protection application.
2.0 Literature Review

2.1 Technology Used to Protect Wood

Due to the environmental pressures and limited sources of naturally durable wood supplies, wood preservative are commonly used to extend the lifespan of less durable wood species (Freeman et al., 2003). Natural durability of timber can be defined on its ability to fight against the attacks of foreign organisms for example insects, fungi and also marine borers. Studies on natural durability of timber have been carried out by numerous researchers all over the world. Natural durability or natural resistant is defined as the ability of the heartwood of timber species to resist biological deterioration in service (Wong et al., 2005a). The measurements for these procedures followed a rating scale of AWPA on the longevity of wooden stakes in field tests and rate timbers into an acceptable durability rating class. It is useful to the timber trade in assigning a value to wood species although the relative durability rating cannot confirm the service life of the life-size structures in service (Zabell & Morell, 1992). Durability class of Malaysian Wood Species can be divided into durability of treated wood and durability of untreated wood. Among the 113 wood species minority of Malaysian wood species classified as durable timber (Class 1 & 2) and these timbers are commonly utilized in heavy construction of roof trusses and beam. Several factors such as wood density, lignifications of heartwoods and also the toxicity of its extraneous material. The heartwood extractives of durable timbers are known to contain in sufficient quantity of specific extractives which is the mixture complex of toxic and non-toxic organic compound in order to prevent destruction of wood destroying organisms commonly. 80% of Malaysian wood species are susceptible to biodeterioration and even the very durable timbers susceptible to light decay by rot eventually. On the other hand, usage of naturally durable timbers for construction such as the roof trusses is always limited. This is due to the scarcity of the durable wood supplies in Malaysia (Wong et al.,
It is important to apply preservative treatment on those less durable timbers for long term period in order to protect the wood from biodegradation. Hazard class system for preservative at prescribed loadings in other word, retention for wood product utilization in different situation serves as a guide to the application in many parts of the world as well as in Malaysia. CCA preservatives is still commonly used for the wood construction in order to protect from wood destroying organisms in Malaysia (Wong et al., 2000). According to Sandu (2002), when the wooden objects are seriously altered where its integrity and authenticity no longer being ensured a consolidation treatment is necessary which provide object with mechanical resistances and properties. A number of decisions must be made regards to materials and methodology once the necessity for consolidation method is determined which include the choice of consolidant, the solvent type, suitable method of application, and the solution concentration. The factors depend on the functional requirement of the object, nature of the objects, and type and condition of materials (Unger, 2009). Three techniques involve include reversibility, compatibility, and re-treatability to consolidate wood materials. These methods can help to identify in the aspect which related to shrinking phenomena, penetration depth, uniformity of distribution, wood swelling, toxicity levels, and consolidant retention (Timar, 2009). Consolidation material should have two main properties which are adhesion and cohesion which can impart sufficient strength to provide mechanical properties while also to ensure cohesion of disrupted structure. One of the stability includes resistance to weathering and aging. Soluble resins, thermoplastics synthetic polymers in solvent solution is the latest treatment of consolidation due to its ease of application and the reversibility of the consolidation product (Unger, 2001).

Chemically treated wood provides more advantages compare to untreated wood. The lifespan of treated wood can be extended compare to untreated wood. Therefore, the
frequency of the replacement of the wooden structures can be decreased and it conserves our forest. Hingston et al. (2001) stated that untreated wood can only be used in limited situations. Preservation processes is therefore very important to be practiced for wood construction (Wong et al., 1997).

The preservation process onto timbers or wood products caused the cost of wood product to increase. Biodegradation is a problem in which preservation is needed in order to conserve forest (Wong et al., 2005). Indeed, wood preservation also applied by wood importing countries (Richardson, 1993). In Malaysia, wood preservative started in 1918 by The Forest Research Institute (which now known as The Forest Research Institute Malaysia, FRIM) with natural durability stake tests. There are a few types of wood preservative used to protect timbers from biodeterioration (Wong et al., 2000) which are tar oil, organic solvent preservatives and water borne preservatives. For tar oil preservatives, creosote is the most commonly used. It was widely applied to railroad sleepers due to its ability to protect against fungi and insecticide. This method can protect wood and increases the life span of wood to 30 years and more (Becker et al., 2001). Organic solvent preservatives composed biocidal compound which dissolved in volatile or non-volatile and non-polar organic solvent. This type of preservatives mostly used in treatment of wood in buildings. The methods used in such preservative include spraying, dipping, brushing, or double vacuum treatment (Richardson, 1993). For waterborne preservatives, aqueous solution of toxic salts used in housing constructions, treatment of boardwalks and recreational use construction. Copper-Chrome-Arsenic-CCA, Ammoniacal Copper Arsenate-ACA, Borate and Ammoniacal Copper Quatenary-ACQ are the preservatives method used to protect timbers or wood from deterioration. Anyway, CCA is the most commonly used (Wong et al., 2000) and replaced organic preservatives type like creosote (Hingston et al., 2001; Wong et al., 2000). Fixation can be defined as complete
chemical reactions and interactions between preservative components and wood substrate that preservative components are able to be bound permanently in the wood (Kartal & Lebow, 2000). Complete fixation of CCA wood preservative onto wood take days, weeks and months for preservative components to be permanently left in wood. Various studies on these reactions have been done by many researchers to elucidate the fixation mechanism. The fixation mechanisms are different between softwoods and hardwoods which was indicated by Greaves (1974). The withdrawal of CCA preservatives from several countries resulted in the worldwide decrease usage of this wood preservatives to protect wood (Kartal et al., 2004). According to Hingston et al. (2001) this is due to the environmental contamination. Therefore, studies on nanoparticles fixation and leachability become important and various research had been carried out to evaluate the efficiency of nanoparticles preservative.

2.2 The Effect of Nano-ZnO on Wood Protection

Wood polymer composites exposes outdoors were susceptible to degradation from moisture, sunlight, fungal attack and microbial colonization (Imamura et al., 1998). Wood always susceptible to fungal decay while polymers generally resistant to fungal attack (Schmidt, 2007). Fungi can always accessed to cell wall within wood of wood polymer composites. Therefore, wood polymer composites need to be protected against fungi and UV irradiation when they are used in outdoors. According to Poda et al. (2013), many applications used nanotechnology. Impregnation of solid wood with metal nanoparticles suspension for heat treatment and wood preservation were introduced (Taghiyari, 2012). It was followed by applying mineral nanofibers as fungicide and fire-retardants (Karimi et al., 2013), spectroscopy analysis (Akhtari et al., 2013), as well as improving thermal conductivity in wood-composite panels (Taghiyari et al., 2013). The exploration of inorganic nanocomposites adsorbed to organic polymers has attracted much attention due
to such nanocomposites which provided an effective way to improve the decay resistance, physical and mechanical properties and UV stabilizer (Devi et al., 2013). However, the fix adsorption of nanoparticles during the synthesis process of nanocomposites was highly tedious and complex due to the high probabilities for nanoparticles to agglomerate. In order to avoid the agglomeration of nanoparticles in polymers, the adsorption of inorganic particles with polymers always associated through alteration of the surface (Hong et al., 2009). It was an effective way to graft the polymers onto the modified nanoparticles in order to upgrade its dispersion in matrix of polymer which could enhanced the properties of the resulting composites (Matei et al., 2008). Nano-zinc oxide had been introduced as naturally occurring elements in the environment that having a long history of UV stabilization, antimicrobial properties, antibacterial and etc (Clausen et al., 2010). ZnO nanoparticles has also been used in wood plastic composite for protection (Farahani et al., 2013). Previous research showed that aqueous soluble metal formulations is the most residential wood preservatives. Two particles sizes of ZnO with three concentration was impregnated with Southern Yellow Pine (SYP) sapwood and were used for leach and termite resistance. From the treated wood samples, there were range from 14% to 24% of the zinc sulphate leached and less than 4% leached from the nano zinc oxide treated specimens (Clausen et al., 2011). Nano-zinc oxide has great potential to be used in wood preservation because of its thermal stability at high temperatures, protection of wood against UV, and also against being attacked by fungi and termite (Clausen et al., 2009).

Besides, decay resistance of wood polypropylene composite (WPCs) treated with zinc oxide nanoparticles against white and brown rot fungus were investigated. The composites were exposed to decay subsequently according to a modified standard. The distribution of nano-zinc oxide was studied by scanning electron microscope combined with energy dispersive analysis of X-rays (EDAX). The decay resistance of the composite was
improved by using nano-zinc oxide against the fungi because there was no clear evidence of nano-zinc oxide agglomeration at a loading of 3% (w/w) using SEM-EDAX based on the results. Selective toxicity of nanomaterials was reported. Brown rot fungi is able to produce organic acids and therefore they are heavy metal tolerant (Reddy et al., 2007). Such acids can form insoluble metal salts and detoxify the preservatives (Eaton & Hale, 1993). Green and Clausen (2003) stated that *C. puteana* was reported to be copper-sensitive. The ability of the nanoparticles to improve the decay resistance against the brown rot fungus proved that nanometal is not restricted to white rot fungi only. For weathering process, untreated and treated specimens will be weathered at outdoors and the specimens will be examined visually for UV damage such as checking, splitting, and graying. The treated and untreated specimens will be horizontally placed on a tray for the exposure of sunlight. The surface that expose to sunlight will be known as exposed surface while for the inside surface will be known as unexposed surface in order to report the results. Water absorbed in the specimens will be evaluated by American Society for Testing and Materials (ASTM) while for water repellency, it was based on beading of water on the surface of specimens and it will be examined through visual. All the specimens will be placed at room temperature, weighed, and submerged in deionized water for one day and reweighed to determine the absorption. The specimens will be examined through inductively coupled plasma atomic emission spectroscopy ICP-AES for zinc retention and grain checks will be counted for all specimens (Clausen et al., 2010).
2.3 Synthesis of ZnO Nanoparticles

Zinc oxide is no longer stranger to scientific study. It has featured as subject of thousand of research papers. ZnO enters arena with several advantages. Solochemical technique stands out due to its rapidity among the various routes for chemical synthesis of ZnO. Besides than rapidity, it also has the properties of low cost, mild experimental conditions, simplicity, and applicability to industrial scale. Zinc oxide complex and a heated alkaline solution with the controlled temperature is prepared for this technique (Gusatti et al., 2010). ZnO nanoparticles cause cytotoxicity which was indicated by a study on eukaryotes involving mammalian cells (Reddy et al., 2007). Many methods such as laser ablation (Scarisoreanu et al., 2005), hydrothermal methods (Ni et al., 2005), electrochemical deposition (Chang et al., 2002), sol-gel method (Ristiac et al., 2005), thermal decomposition (Wong et al., 2009), and combustion method (Badhuri et al., 1997) used to produce ZnO nanostructures. The most recent method were anodization (Shetty & Nanda, 2012), ultrasound (Khorsand, 2013), electrophoretic deposition (Vazquez et al., 2013) and co-precipitation (Singh et al., 2013).

2.3.1 Chemical Process

2.3.1.1 Sol-Gel Process

The precursors such as ZnCl₂, Zn(NO₃)₂ and NaOH were prepared for synthesis. The process was ran out in a condition at 90°C in a specific time. Dripping process carried out in such method from sodium hydroxide solution to zinc nitrate solution and the procedure was performed with constant stirring. In addition, the temperature was maintained at the desired value which resulted in precipitation of ZnO and color of the solution changes from transparent to milky white (Ravi et al., 2004). The product characterized by using scanning electron microscope, X-ray diffraction, and UV-vis spectrophotometer. Morphology and