

**PRESERVATION OF COCONUT WOOD USING INORGANIC
NANOPARTICLES**

by

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THESIS

Submitted in partial fulfilment of the requirements for the degree of

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2023

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I, hereby declare that this thesis entitled “**PRESERVATION OF COCONUT WOOD USING INORGANIC NANOPARTICLES**” is a bonafide record of research work done by me during the course of research and the thesis has not previously formed the basis for the award to me of any degree, diploma, associateship, fellowship or other similar title, for any other University or Society.

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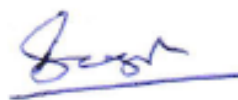
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INTRODUCTION

1. INTRODUCTION

Wood, a versatile natural resource, has been a significant part of human civilization for centuries, primarily used for furniture and construction. Its strength, weight, and flexibility make it a reliable choice for structural applications. Wood's aesthetic appeal and structural stability have influenced architecture from timber-framed homes to modern buildings. Despite the use of steel and other substitutes, the demand for wooden goods has not decreased due to rapid industrialization and population growth (Ramage *et al.*, 2017).

The decline in natural forest harvesting has led to a decrease in timber availability, with only 7% of 294 million hectares of planted forests remaining. However, green cover outside the forest area, including urban trees, orchards, palm trees, and agroforestry landscapes, increased by over 33% from 1990-2020, occupying at least 45 million hectares (FAO, 2022).

According to FAOSTAT (2023), the production of round-wood worldwide has increased by 12% over the past two decades, reaching 3.91 billion m³ in 2020. The construction industry, where demand is projected to nearly quadruple by 2030, and the packaging industry, where demand is projected to more than double by that same year are the key drivers of continued growth in the demand for biomass derived from forests. Also, prediction by Global Forest Products Model (GFPM) expected the increase in the output of industrial round-wood by 28% to 2.5 billion m³ globally between 2020 and 2050 (FAO, 2023).

Sustainable and efficient utilisation of available wood sources and other lignocellulosic materials could possibly reduce the demands and supply gap (Lafleur and Fraanje, 1997; Obata *et al.*, 2005; FAO, 2015; Ramage *et al.*, 2017; FAO, 2022). Wood, despite its attractive appearance, strength, low density, and insulation, also has negative aspects like hygroscopicity, flammability, susceptibility to biological attacks, and aging-induced surface deterioration due to UV-weathering (Jirouš and Miklečić, 1980).

Timber species are naturally resistant to decay caused by microbes and insects, but many are endangered or slow-growing, making them unable to meet market demands (Lebow, 2010). Extractives, produced on trees and deposited in heartwood, provide protection and colour (Troup, 1913). Sapwood from all species is sensitive to decay, while

heartwood from select species is resilient. The most resilient species have inherent resistance and darker-coloured or denser timbers (Richardson, 2002).

Durability of both heartwood and sapwood can be enhanced by the application of certain chemicals i.e., preservatives without altering the physical and mechanical properties of timber (Troup, 1913). The life of wooden structures can be substantially extended by preservation techniques, which lowers replacement costs and allows for more effective use of forest resources. They offer defence against a variety of assaults on wood products, including those from marine borers, insects, and decay fungi (Lebow, 2010).

Some of the traditionally used preservatives includes Oil-based, water-based leachable and water-based fixative, insecticide, fungicide, fumigants, fipronil, hexachloride, dieldrin and other biological products to control wood degradation. These chemicals are applied on or into the wood surface either by non-pressure or pressure method (Zabel and Morrel, 2012). The performance of wood species, chemical preservatives, and treatment techniques over the long term depends on the effectiveness of the products and the chemical penetration and retention. (Lebow, 2010).

Biocides, effective in preserving wood, have drawbacks like unpleasant odors, color, and surface unpaintability. Leaching of harmful chemicals, such as chromium, arsenic, and copper, poses a significant threat to the environment and human health (Troup, 1913). Environmental factors like water volume, flow rate, pH, reduction and oxidation potential, ion adsorption sites, and soluble ligands influence these leaching processes (Lebow, 1996). In CCA, the leaching of arsenic was more likely than chromium and copper (Song *et al.*, 2006), but if pH is higher, the valence of chromium increases, leading to an increase in toxicity (Hasan *et al.*, 2010). As a result, these preservatives are reduced, restricted, or eliminated (Groenier and Lebow, 2006)

Some copper-based preservatives are ammoniacal copper azole (CuAz), ammoniacal copper quat (ACQ), ammoniacal copper citrate (CC), Copper zinc dimethylcocoamine propanoic acid (CZDP), ammoniacal copper zinc arsenate (ACZA) are not effective against all fungi. Some mould or stain fungi so, called copper tolerant which includes *Fibroporia radiculosa*, *Serpula lacrymans*, *Porica placenta*, *Antrodia vaillantii* can grow on copper-treated wood leading to reducing the efficiency of copper

based preservatives (De and Woodward, 1999; Hastrup *et al.*, 2005; Ohno *et al.*, 2015; Lebow *et al.*, 2020).

When applied in suitable concentrations, zinc-based preservatives offer acceptable protection despite being less effective than copper-based ones (Barnes *et al.*, 2004). Similar to copper some zinc tolerant fungi *Poria placenta*, a brown rot fungus as tolerant to zinc based preservative but not to zinc nanoparticles (Németh *et al.*, 2013). Preservatives based on boron have low environmental toxicity and efficiently deter termite and rot fungal attacks (Manning, 2008). However, exposure to the elements can cause borates to be leached out of the wood as they fail to bind to the wood surface (Taylor and Lloyd, 2009).

Due to the above-mentioned issues on the negative effects these conventional approaches have on the environment and the hazards they pose to human health have prompted the creation of creative and sustainable alternatives (Dos *et al.*, 2012). The need for the development of novel solutions for wood preservation is demonstrated by the fact that wood treatments based on CCA and other water based fixative ones have limited commercialization and increased environmental concerns (Groenier and Lebow, 2006).

In the late 1990s, the USDA Forest Products Industry, with the support of the U.S. National Nanotechnology Initiative and American Forestry & Paper Association, initiated national nanotechnology R&D to enhance raw material resistance against insect and pest attacks. This involved developing coatings and solutions related to nano-biocides for wood preservation, recognizing the potential of nanomaterials for innovative, cost-effective preservatives. (Clausen, 2007).

The National Nanotechnology Initiative (NNI) defines nanotechnology as atomic, molecular, or macromolecular scale research and technology development for controlled fabrication and application of structures, devices, and systems with a length scale of 1-100 nanometres (NNI, 2023). Carbon nanotubes (LeRoy *et al.*, 2004) and gold nanoshells (West and Halas, 2003) have distinct physical properties compared to larger carbon or gold particles. The shift from micro particles to nanoparticles can alter various physical properties (Khan *et al.*, 2019).

As wood preservatives nanoparticles such as silver, copper, zinc, boron, silica, titanium oxides are used in many works (Clausen *et al.*, 2009; Kartal *et al.*, 2009; De

Filpo *et al.*, 2013; Künniger *et al.*, 2013; Mantanis *et al.*, 2014) due to greater penetration (Wood pores and pits), used in low concentration, water resistance, durability, photoactivity or UV-Weathering, resistance to scratches, longevity, and antimicrobial properties, termite resistance, minimum leaching, minimum toxicity to environment (Kandelbauer and Widsten, 2009; Kaiser *et al.*, 2013).

Due to the above mentioned benefits, this study was defined to preserve coconut wood using inorganic nanoparticles to increase its durability. Coconut, a tropical tree, is a significant crop in Kerala for its nutritional and medicinal value. Its various uses include water, copra, oil, and wood-based products. Coconut is referred to as the 'tree of heaven' or 'kalpavriksha' due to its diverse products. It has applications in food, medicine, energy, and construction (Sangamithra *et al.*, 2013). Coconut in Kerala has 20.8 percent of the total geographical area under coconut and 33% of the total coconut plantation in India (FSI, 2021). In order to reduce pressure on conventional timber, coconut wood can be utilised effectively as a potential substitute for conventional timber in the tropics (Mosteiro, 1982; Killmann and Fink, 1996; Anoop *et al.*, 2011).

Due to its distinct anatomical characteristics and density, coconut wood differs greatly from typical wood (McQuire, 1979; Mead, 2001). The initial moisture level of coconut palm wood can range from 60% (high density) to 230% (low density), and if it isn't quickly dried out, it can eventually develop blue streaks and mould on the surface (Rehman, 1952). Coconut wood can be increased through primary methods of effective utilisation of timber such as seasoning and preservation (George, 1985). However, for major improvement to meet requirements and exhibits minimal resistance to organisms like termites and stem borers that degrade wood (Mosteiro and Siriban, 1976).

As such, there is no research works on enhancing coconut wood durability, degradability, and biocidal properties using nanoparticles; the present study entitled “Preservation of coconut wood using inorganic nanoparticles” is framed with defined objectives as follows:

1. To synthesize inorganic nanoparticles, their characterization and
2. To test the physico-chemical and biocidal properties of coconut wood treated with inorganic nanoparticles.

REVIEW OF LITERATURE

2. REVIEW OF LITERATURE

2.1. Coconut in India

Coconut in Sanskrit, the ancient Indian language, is called “Kalpavriksha,” which means the thing which provides humans with everything, has been used by man for more than 4000-5000 years. The main significance of coconuts is as a source of food, drink, fuel, shelter, and raw materials for a few cottage and small-scale enterprises (Sangamithra *et al.*, 2013). Along with these, this tree produces coconut wood/timber also known as ‘porcupine wood’ in the lumber trade due to its spotty appearance, grain, colour (Eala 1980; Anoop, 2011).

Coconut is a monocotyledon plant belonging to the Arecaceae family having a thin, straight or slightly curved trunk (0.3-0.4m) and tall height (25-30m) with an average lifespan of around 40-80 years (Kuttankulangara and Megalingam, 2019; Nair and Nair, 2021). It is found in coastal and inland regions of all tropical countries especially between tropic of cancer and capricorn (Jeeshma *et al.*, 2017). It covers an area 12.57 million ha globally with Philippines (3.647 million ha) leading the stand whereas India (2.199 million ha) occupying third. Within India, Southern states namely Kerala, Karnataka, and Tamilnadu, Andhra Pradesh are the leading states in terms of area covering almost 90% of total coconut area in India are found in these states (CBD, 2023).

Kerala is one of the smaller states in terms of area, covering approximately 36% of all coconut plantations in India. Due to this, Kerala is called the 'Land of Coconut' even its name ‘Kera’ in Malayalam means ‘Coconut’ (Ahuja *et al.*, 2014).

2.2. Coconut as an alternative timber source

Historically, the wood from mature coconut palms was used for a variety of applications throughout the world's coconut-producing regions (Killmann and Fink, 1996).

According to botany, the coconut palm is a monocotyledon, not a tree. As a result, its fibres are not considered to be wood strictly speaking. However, under certain conditions, pieces of the stems from old, tall palm trees can be utilised as a substitute for wood (Durst *et al.*, 2004).

Accordingly, there are an estimated 1000 million coconut palms in Asia, of which at least 20% (200 million) senile trees should be replaced. A typical tall variety coconut farm contains roughly 80-100 stems per ha. One tall coconut stem produces roughly 0.7 cubic metres of "roundwood," which can be effectively transformed into an average of 0.28-0.34 cubic metres of sawn coconut "lumber" (GOP, 1992). Based on these data, it is predicted that Asia can create 5-6 million cubic metres of sawn coconut lumber annually from palms that are no longer producing coconut and other products. The majority of tall coconut varieties will have been replaced with the higher-yielding dwarf varieties by the time this level of production is sustained for another 20 to 30 years at present rates of replacement. Although the dwarf species' fibres are not as well adapted for "solid timber" use as the tall varieties are, they can still be used to make wood-based panels (Durst *et al.*, 2004). Also an estimated 371.3 million senile coconut trees, or 111.4 million cubic metres of sawn coconut wood, exist throughout the Asia-Pacific region (CKC, 2023).

In Indonesia, estimated 50-60% of palm trees being over 50 years old, the nation possesses a coconut wood resource of over 185.6 million senile trees that may be removed and replaced with hybrids and other high producing species. According to a sawn timber recovery rate of 0.30 cubic metres per tree, the coconut wood region has access to about 55.7 million cubic metres of sawn wood that can be used economically. According to the supposition that the sawn timber will be utilised to build a typical 60 square metre, two bedroom low cost house with a timber need of 15 cubic metres per house, a total of 3.71 million housing units might be built out of these wood material (CKC, 2023).

Even in India 2.19 million hectare area of coconut is present, the productivity of coconut has been reducing. In Kerala, due to decline in the productivity of coconut palm as a result of diseased, over-mature and senile palm, leading to replant the existing plantation with high yielding varieties of coconut. This creates surplus quantities of coconut palm wood that can be used as a substitute for conventional timbers to meet our growing needs leading to the reduction of pressure on the remaining forest and helps in conserving them (Anoop, 2011; CBD, 2015; Thamban *et al.*, 2016). So, the myriad useful and eco-friendly uses of products and wood taken from these trees are gaining greater prominence especially in tropical countries (Arancon and Apfsos, 2009).

Different parts of the coconut wood trunk can be utilised for different purposes, depending on their density (Desch and Dinwoodie, 1981; Anoop *et al.*, 2011; Srivaro, 2021). Based on density it can be graded into three categories namely High density ($<600 \text{ kg m}^{-3}$), Medium density ($400\text{-}600 \text{ kg/m}^{-3}$) and low density ($>400 \text{ kg/m}^{-3}$) (Killmann and Fink, 1996). Usually for structural and load bearing purposes coconut timber usage is restricted to the harder, denser portions, whereas the lighter portions are modified by adding additives to enhance its load bearing properties (Adkins *et al.*, 2010; Jayabhanu, 2011; Jeeshma *et al.*, 2017).

The study condensed the findings of FPRDI (Forest Products Research and Development Institute) investigations conducted in the 1970s on the characteristics, applications, and upkeep of coconut palm timber as a building material (Mosteiro, 1981).

Mechanical properties of five small transparent samples taken from coconut trees were examined. Compared to solid wood, the strength characteristics of coconut trunks in bending were more varied. The variance in bending of coconut trunks was roughly twice as large as that of various Philippine woods (Espiloy, 1977). According to research, solid wood with a similar specific gravity is more financially competitive with coconut palm timbers as a building material (Ramos and Miciano, 1966).

In order to use full-size coconut trunks as power and communication poles, Espiloy and Tamolang (1977) study tested their strength in bending. Modulus of Rupture (MOR), Modulus of Elasticity (MOE), and Fiber stress at proportional limit were considered as strength qualities. MOR and MOE were equivalent to those of Almon, Mayapis, and Manggasinoro, despite the fact that the Fibre stress at proportional limit of these species was nearly twice as high as that found in the coconut trunk. The robustness of coconut trunks makes them ideal for use as communications and power poles in rural locations.

In comparison to the commonly used *Dipterocarpus* species, which has an average fibre stress of 62.76 MPa, the mechanical property test of a coconut trunk taken about 2000 mm from the groundline level and prepared into standard cross-arms containing the hard and medium portions revealed fibre stresses in MOR of 70.24 MPa and 62.32 MPa, respectively (Parayno *et al.*, 1988). The specific gravity and shrinkage determination investigations were conducted on five coconut trunks that were procured

from Tiaong, Quezon. Apitong and yakal-gisok woods were compared to coconut wood in terms of specific gravity and shrinkage characteristics. Coconut wood experienced radial shrinkage similar to that of apitong and yakal-gisok. Its tangential shrinkage was also lower than that of the two wood species. Coconut wood had a lower specific gravity than two other types of wood. A 19.05 mm x 100 mm coconut board can support an impact load of 1601.28 N and a wind load of 0.001148 N/mm², respectively, while a 12.7 mm diameter coconut dowel can support an eccentrically applied load of 222.40 N, according to theoretical analysis of experimental doors (Zamora and Gesmundo, 1986). The economics of collecting coconut palms, sawing their trunks into timber, and processing T & G for flooring and S. C. for siding were examined in this study. Thus, Recommending Coconut timber is suitable for construction, flooring and other similar applications (Medrano and Laurici, 1977).

Using machine bolts of size 1.3 cm and 1.6 cm, the bolt-bearing stresses of bolt-jointed specimens corresponding to a 4 Lift to drag (L/D) ratio were measured in compression parallel and perpendicular to the grain of green coconut timber. The typical bolt-bearing characteristics of coconut timber were compared to those of Tangile and Apitong species (Floresca, 1978). Suggesting, coconut material has potential applications in low-cost housing, human settlement, rural electrification, and forest conservation in developing countries (Mosterio, 1975).

Coconut trunks were used in the production of school seats in Los Banos, Laguna, Philippines. The process involved sawing, grading, drying, machining, fabricating, and finishing. The chairs were installed in three elementary schools and after 1.5 years, they remained in excellent condition (Mosterio, 1981).

The Small Business Advisory Centre, BSMI, Ministry of Trade and Industry sponsored a training program on manufacturing furniture using coconut wood (Mosteiro, 1986). Based on the ₱ 3.50/bdf retail price of coconut lumber, coconut wood roof shingles were 1.23 percent less expensive than GI corrugated sheets roofing in the cost comparison study. Coconut wood shingles would cost 20.02 percent less than GI corrugated sheets of roofing if the cost of producing coconut lumber was ₱ 1.3/bdf (Floresca *et al.*, 1987). The estimated cost of coconut wood was 20–23% less than that of the commercial hollow block (Laurico 1984).

So, wooden construction, power poles, windows, doors, stairs, flooring, panelling, and other purposes are the main potential end uses for coconut wood. These goods, which are sold on a small scale, might potentially replace the traditional lumber used in building and for structural purposes, as well as for tool handles, furniture, and other objects used in daily life (Mead, 2001; Tamolang 1986).

Thus, Coconut wood has been proven to be a potential substitute for traditional woods and is commercially used in various products.

2.3. Coconut wood uses with respect to its density

Usually, the hard, durable, high density portion of wood can be used in construction of building, furniture, stairs, flooring and panelling, tool handles, railing, and other load bearing structural materials. The moderate hard, medium density can be used in the construction of studs, ceiling joints, window or door frames. The low density is used in the interior decoration parts of buildings as a ceiling and wall lining in the forms of composite and improved woods (Anoop *et al.*, 2011).

2.4. Coconut wood challenges

Low price, hard, assurance, durable, versatile, reduced cost of housing unit, non-branching free from knots, available in different shades, characteristics grains and texture, resistance to indentation and abrasion are some of the advantages of coconut wood (FAO, 1985; Killmann and Fink, 1996). Some potential drawbacks faced during processing, seasoning, biological agents and other degradation factors are discussed here

2.4.1. Processing or working challenges

The processing of coconut palm presents a number of challenges because the anatomy of wood is so distinctive as a result of this, cutting, few sawing operations, even normal steel saws would become dull and useless whereas, these problems faced in other traditional timbers are low. The two main causes of this are friction caused by fine abrasive materials created by the disintegration of parenchymatous ground tissues and the high density of the wood in the outer region (FAO, 1985; Oduor & Githiomi, 2006).

Saw blades, planer blades, shaper knives, drill bits, and other machine tools quickly became dull from the machining of coconut wood into various finished items

using standard machine blades and tool bits. The presence of stigmata cells which contain silica, which are connected to the vascular and non-vascular fibres, may be the cause of the dulling effect. (Mosteiro 1979).

According to Chittenden *et al.* (2007) machining of medium and soft materials can result in chipped grain, but the hard part performed admirably and produced a smooth surface. The thick-walled sclerenchyma fibres and high mineral content of the wood including silica, changes in the angles of vascular bundles makes sawing processes particularly challenging according to Subramanian (2003) research. Hopewell *et al.* (2010) showed that High density makes tools and equipment abrasive. When compared to other conventional wood, coconut is more difficult to saw due to variation in density within the stem.

While grading, the visual method is slow and cautious because it can only be used to roughly estimate observable flaws. It is quite challenging to assess other invisible elements like the material's inherent flaws and strength fluctuation (Arancon and Apfsof, 2009). Also, it is difficult to grade coconut wood based on density due to variation.

2.4.2. Seasoning challenges

The coconut timber needs to be dried and seasoned before use. Depending on the lumber's intended use, the moisture content must be lowered to acceptable limits. The cheapest and simplest way to dry coconut timber is in an open-sided, roofed shed. It's important to guard against mould and fungal attacks while stacking freshly sawn coconut boards for drying (Arancon and Apfsof, 2009).

During drying, cell wall content hardens due to increase in density as a result of decline in moisture content. This makes sawing increasingly challenging. Cell wall content hardens and increases in density as moisture content declines. Coconut trunks should be sawed while still green due to the dermal portion's toughness. The sawing rate is decreased and saw tooth wear is enhanced when the logs are cut dry (Arancon and Apfsof, 2009).

Coconut wood is incredibly prone to seasoning faults due to its lack of consistent radial growth and high level of heterogeneity. According to FAO (1985), low density materials have a higher propensity to collapse during drying. The amount of unusable

wood makes up about 15% of the entire stem. Coconut wood is difficult to nail, and high density timber splits frequently.

If the lumbers were improperly piled for drying, seasoning flaws like cracks and checks would develop on the surface of the coconut wood. As a result, seasoning should be done on the timber before use to bring it into balance with respect to its location (APCC, 2000).

Studies revealed that air drying had a lower failure rate than kiln drying. The core-containing region was particularly prone to the seasoning defect, which was primarily collapse. The most evident seasoning flaw, though, might not be repairable. In the bark-containing region, surface inspections were more noticeable, and twisting was more common in the centre. More often than bows or springs, twist was used. (Laxmana, 1980; Laxmana 1984; APCC, 2000).

2.4.3. Biological agents and other degradation factors

The service life of coconut wood is constrained in tropical areas with high relative humidity because of a variety of environmental factors, including precipitation and high ambient temperatures that encourage biological decomposition. When exposed to certain environmental circumstances, this coconut wood develops a biodegradation vulnerability. Wood possesses hygroscopic features due to its ability to absorb water, which lessens its physico-mechanical characteristics. Untreated wood loses quality and develops a higher moisture content when exposed to rain or snow on a regular basis (Ney *et al.*, 2019).

If exposed to weather, coconut wood has no inherent defences against wood-destroying insects and fungi. Low density materials in the earth will disintegrate in 6 to 18 months, whereas high density materials may endure for 2 to 3 years. Fungi that produce soft rot can speed up the decay of dense wood. Additionally, termites target exposed coconut wood. Coconut stems with bark have served as marine piling for more than 3 years (Mosterio and Siriban, 1976).

Mould and stain-causing fungi are particularly likely to grow on freshly sawn coconut wood. The chopped ends of recently felled stems are likewise diseased. In a tropical setting, it is challenging to avoid stains. Sawn wood should be immediately

dipped in an anti-sapstain preservative solution to reduce staining if rapid kiln drying facilities are not available. Staining fungus only significantly damages the wood's strength qualities by discolouring it (Sulc, 1979).

Ambrosia beetles can attack freshly cut coconut wood while it is drying in the open air. The attack is not severe and stops when the wood is dry, but some uses of the wood may not be able to tolerate the wood's deformity from pinholes. Both kiln seasoning the wood and adding an insecticide to the anti-sapstain formulation can provide protection against ambrosia beetles (Kinninmonth, 1979).

Specific species of *Ganoderma* are responsible for the two primary issues, basal stem rot of oil palms and "anabe roga" or mushroom disease of coconut palms (Govindu *et al.*, 1983; Turner 1965). In order to avoid creating breeding grounds for the rhinoceros beetle the trunks must thereafter be properly disposed of (Meadows *et al.*, 1980).

To assess the natural decay-resistance of coconut trunks when utilised as fence posts, foundations and bridge girders were gathered from four locations, isolates of *Romes sp.*, *Coriolus versicolor*, *Polyporus sanguineus*, *Polyporus sp. A*, and *Polyporus sp. Band Hexagona sp.* were taken from the specimens. From the butt, middle, and top portions, as well as from the exterior and inner regions of each portion, test blocks of coconut wood were gathered. From the exterior to the inner regions and from the butt to the top portions, the resistance of coconut trunks reduced. The responses of the coconut trunks gathered from four different locations to the test fungi also differed (Ballon, 1984).

Similar report has been made in FAO (1985) that higher resistance was seen in test blocks from the outer sections, which reduced towards the inner regions. Additionally, the locations' responses to the resistance against the fungal invasion varied. Due to degradation and a severe infestation of borers, mildew, and fungi, about 16% of the eight-month-old logs that had been stored could not be sawn.

Seasoned coconut wood that is kept from becoming any more wet appears to repel pests that eat wood. Attacks by dry wood termites have been recorded, however they tend to occur in low density materials. Additionally, subterranean termites do not appear to represent a significant threat to dry, sound coconut wood. The solid wood of mature coconut stems is known to provide buildings with satisfactory service for many years.

For a long service life, coconut wood that will come into contact with the ground or will be exposed to weather or other moist conditions needs to be preserved (Findlay, 1985).

The Coconut will quickly decay if left outside. The hard part of the trunk gets consumed by termites and decay fungi when used in ground contact within two to three years. The soft component will rot in a few months. To protect coco wood against insect and decay organism attacks and lengthen its useful life, the proper preservatives must be applied (Bauza *et al.*, 1984).

Insect pests like the red palm weevil and rhinoceros beetle, which lower output by 30%, are the principal biotic constraints preventing the production of coconuts. The borer bugs cause permanent damage to the frond, trunk, and stem of the coconut palm. The palm wood should be free of pest damage and suitable for building applications when it turns senile if integrated pest control measures are implemented against the main pests in the standing crop promptly (Sujithra *et al.*, 2012).

Peters *et al.* (2014) conducted two field trials over a 16-week period to see if they could control the subterranean termites *Coptotermes acinaciformis* and *Mastotermes darwiniensis* using untreated coconut wood of varied densities. It was determined that all densities may be regarded as susceptible, especially to *M. darwiniensis*, and that overall Coconut wood sensitivity to *C. acinaciformis* and *M. darwiniensis* decreased with density.

Freshly cut, untreated wood is susceptible to mould and staining fungi like *Acremonium* species, *Aspergillus* species, *Rhizopus* species, *Fusarium* species, *Penicillium* species, *Paecilomyces* species, and *Scopulariopsis* species (Hopewell *et al.*, 2010; Bahmani *et al.*, 2016), particularly if it is improperly stacked and left out in the elements during the air seasoning process. Seasoning can also make things deteriorate due to pinhole borers and rot fungus. So, surface treatment is needed if the coconut wood is used to make products for export.

2.5. Alternative response to coconut wood problems

Moreover, processing challenges can be reduced by usage of hard facing materials like satellite or tungsten carbide on the saw tooth to overcome the blunting problem of saw blade (FAO, 1985). APCC (2000) suggested that sawing should be carried out in

green condition as a result of this reducing sawing rate and wearing of saw tooth can be reduced.

Arancon and Apfsos (2009) suggested that development of chipped grain in medium and soft portions during machining could be solved by the application of fillers and some extra sanding. Due to difficulty in differentiating soft and hard wood parts, staining increases gradation, making it darker. The dulling effect during sawing solved utilising tools with carbide tips (Mosteiro, 1979).

Arancon and Apfsof (2009), recommended that hardfacing materials, like satellite and tungsten carbide are recommended on conventional saw blades to overcome the rapid dulling of saw teeth. Recovery of coconut trunks and sawmilling properties suggested that timber may be made from coconut. The production recovery in a bandmill with satellite-tipped blades was 49.19% (Siriban *et al.*, 1976).

The dimensions of sawn timber are limited due to the reduced diameter of coconut stems. The maximum width and thickness of sawn timber produced from common logs are 25mm and 50 mm, respectively. But laminating the narrow pieces of wood together can work around the problem and produce timber with wide surfaces (Arancon and Apfsos, 2009; Anoop *et al.*, 2011).

Eala and Tamolang (1976) investigation assessed the suitability of coconut lumber for secondary-wood goods like furniture and fixtures, focusing on its response to common machining operations. Despite its lower turning ability, coconut timber shows potential for turned items, suggesting the possibility of gentle sanding techniques.

Laxamana (1984) study to identify the portion of mature coconut trunks that was susceptible to these defects, as well as to identify the effects of three drying methods (air drying, kiln drying, and forced-air drying) on the development and nature of drying degrades. The butt of the tree was used to produce coconut wood rather than the centre and top of the trunk, which is used for construction of homes and furniture. The board at the top of the trunk that contained the core was most prone to collapse. On the bark-side of the board sawn from the butt, surface checks tended to appear more frequently. Boards from the centre and butt portions tended to twist. Air drying significantly eliminated these flaws

The two hour "reconditioning" process significantly reduced the collapse and distortion that had developed in some boards. When an initial Dry bulb temperature of 63 °C (145 °F) and a Wet bulb temperature of 65 °C (149 °F) were employed, serious surface inspections did not happen (Laxamana and Tamayo, 1976).

2.6. Coconut wood preservation

Coconut is not a particularly resilient material when utilised in conditions that invite assault by decay fungi and wood-boring insects, especially when exposed to the elements and in ground contact. By using adequate wood preservation treatment, for which suitable prescriptions and dose rates have been developed, the low natural durability can be addressed. The level of risk and the expense that can be accepted will determine the treatment to be used (Arancon and Apfsos, 2009)

The use of chemicals or chemical mixtures that are harmful to organisms that destroy wood is typically implied by the term 'Wood preservation'. By allowing us to use wood with the longest possible practical life fix, wood preservation is one of the most crucial ways to maintain our forest resources. Early this century, this was acknowledged, and since then, complex wood treating facilities and organisations have grown all over the world. Coconut wood that has received the proper treatment can last for more than 20 years, but coconut wood that comes into touch, with the ground needs to be preserved. If the wood is mature and has a high density, interior uses like furniture, flooring, and walling do not require chemical treatment. However, in some environments, coconut wood needs to be treated with a preservative to lengthen its useful life (McQuire, 1979). Coconut wood needs to be preserved when it is exposed to the elements or comes into touch with the ground (ISI, 2001).

Debarking round poles and posts was a very challenging task, but it was essential if they were to be treated with hot and cold baths or traditional pressure treatments. Before treatment, the wood must be at least partially air-dried, and this must be done in a covered area. With creosote by hot and cold bath and copper-chrome arsenate by vacuum/pressure, adequate retention and distribution could be obtained, provided the outer zones were well dried. Making absolutely certain that no fungi infected the wood between its felling and final treatment seemed to be the most important aspect (McQuire, 1979).

After treatment, coconut wood that has been adequately loaded with preservative is predicted to have a long service life (Meadows, 1979; Mosteiro and Amaldo, 1980). When preserved properly, coconut wood could be used for a variety of things, including roof shingles, dwellings, piles, and buildings (McQuire, 1979). Even when utilised in ground contact, pressure-treated coconut wood with retentions of 5–10 kgm⁻³ produced good results (George, 1985). Coconut palm wood might be used effectively as building material close to home, which would also help create jobs in rural regions and encourage the preservation of forests.

2.6.1. Methods of preservation

Some of the commonly used Preservative methods that have been mentioned in the study are as follows:

2.6.1.1. Coconut as a green timber

Coconut wood that is in green condition can be preserved using Diffusion process (Mosteiro and Siriban, 1979), Pressure treatment and Pressure-sap displacement (Familton, 1979). The final use of the treated wood and the facilities available for treatment will determine the preservative and treatment method to be used (Findlay, 1985).

2.6.1.2. Coconut as a dry timber

Coconut wood that is completely or partially dry can be preserved using pressure methods (Palomar, 1979; Jeeshma *et al.*, 2017), hot and cold baths (Mosterio and Siriban, 1976; Mosterio and Siriban, 1979), dipping (Mosterio and Siriban, 1979), soaking (Mosterio and Siriban, 1979), and brushing (Mosterio and Siriban, 1979). The final use of the treated wood and the facilities available for treatment will determine the preservative and treatment method to be used (Findley, 1985). Among this Pressure treatment held good efficiency of chemical penetration.

2.6.2. Types of preservation

Due to less literature available on Coconut wood preservation. Some the common preservative used for treating wood throughout the world are Creosote, Inorganic based-NaPCP (Sodium-pentachloro phenate), CCA(Chromium-Copper-Arsenate),

CCB(Chromium-Copper-Boron)/CCF(Chromium-Copper-Fluorine), CC(Chromium-Copper), AAC(Alcyl-Ammonium Components), CU-HDO, MBT(Methylene-bis-Thiocyanate), Boron(Borax, Boric acid), and other water borne Agricultural fungicide (Captafol, Chlorothalonil, Timbafol) were among the regularly used inorganic preservatives identified by Willeitner and Liese (1992).

2.6.2.1. Creosote

Results of exploratory research on the capacity to treat coconut and other common palm species in the Philippines using coal-tar creosote as a preservative were provided. Conditioning by boiling-under-vacuum followed by the Full-Cell technique, out of the three treatment methods used, gave nearly complete preservative penetration in the treated specimens. Due to the high moisture content of the specimens, particularly in the core portion, other treatment procedures demonstrated insufficient creosote penetration and distribution (Mosteiro, 1971).

Mosteiro (1977) study investigated the hot-and-cold bath technique of treatment for round and sawn pieces of partially-seasoned coconut trunks with Moisture content of 35-65 percent in the outer 3.81-5.08 cm from the surface. This method is easy to use, affordable, and flexible in remote locations where access to treatment facilities is limited. Results from the study were satisfactory. After the sawn timbers measuring 12.70 cm x 15.24 cm x 6.1cm, a retention of 272-329.6 kgcm⁻³ was obtained after a 6-8 hour hot-bath and 12-15 hour overnight cooling. A retention of 115.2 -172.8 kgcm⁻³ for the solid round coconut trunk without bark was achieved after 8–10 hours of heating and overnight cooling. Creosote temperature was kept between 92.4 and 97.9°C during the hot bath.

2.6.2.1.1. Draw-back of creosote

Creosote can be applied on wood in ground contact, particularly power poles and fence posts. However, it cannot be painted over, has a strong odour, and is generally more expensive than waterborne preservatives in many nations (Killmann and Fink 1996).

2.6.2.2. Inorganic based

It includes water-borne and water-based fixative types of preservation. Here some of the inorganic based preservatives discussed in the literature.

On over-mature coconut wood, the Tutu Insertion procedure, a potential pressure sap displacement method, was demonstrated. It was determined that it is capable of achieving high penetration of Tanalith NCA, Copper sulphate, Sodium dichromate, Arsenic pentoxide, and Sodium pyro-arsenate and other water-borne preservatives with 90-1100 cm distance from log, 3-290 cm from nearest point of insertion and a high rate of retention of 3-30 kgm⁻³ in core region and 3-10 kgm⁻³ in outer region (Martin, 1978).

Excellent results were obtained with pressure treatment using intermediate retentions of copper-chrome-arsenate. Creosote or copper naphthenate brush-coating of dry wood provided good protection, although retreatment was required every three to four years. A nice and durable surface could be achieved by pre-treating the surface with inorganic salts (such as 12 percent acid copper chromate), followed by one or two coatings of latex emulsion stain (FAO, 1985). If the wood is easily treated with boron by diffusion, protection from insects must be ensured. Unbaled log pressure sap displacement has proven to be impractical (McQuire, 1979).

Tan *et al.*, (1983) recommended adding 2% NaPCP to the treatment solution. Incorporating 2% "Basillt SAB" in the treating solution as an alternative to NaPCP seemed to be somewhat effective against sap-stain. Plackett (1982) discovered that treatments containing 2% land-grown Busan were beneficial against sap-stain and mould. For efficient suppression of sap-stain infection during seasoning. Only a greater dose of "Busan" (1.5%t) shown efficiency against sap-stain and mould fungi in field tests carried out in Brazil over the course of a five-month field experiment (Milano, 1981).

Coconut wood shingles with medium and high density can be treated with CCA or Pentachlorophenol as it is effective withstand outdoor conditions of 1 year, economical and ease of installation (Floresca *et al.*, 1987). A 5% aqueous solution of sodium pentachloro phenate (NAPCP) and 2% borax during the rainy season or a 2.5% NAPCP and 2% borax during the dry season is the preservative that is successfully utilised for dip-treatment. However, it is illegal in many nations due to its severe toxicity (Killmann and Fink, 1996).

Pressure-treating exposed wood is recommended, preferably with water-borne preservatives. These are typically sold as salt-like powders that dissolve in water. Copper-

chrome arsenate (CCA), a salt-type preservative that is particularly effective against termites, is the most often used one in the tropics (Killmann and Fink, 1996).

Jose *et al.* (1989) found that NaPCP was the best preservative for use during preventive treatment and that it effectively inhibited fungal and insect borer attack when combined with boric acid and borax. In addition, they noted that oxycarboxin, together with NaPCP, demonstrated promise in the management of fungi and integration of pesticide. They discovered that using a solution of 1 percent copper sulphate was beneficial for two months against insect borer assault.

Palomar (1989) looked into how different fungicides affected the management of pinhole borer and blue stain during the seasoning of freshly sawn coconut wood. All fungicides were shown to be ineffective in providing the desired protection against the blue stain fungi, according to results after 12 weeks of air drying (Palomar *et al.*, 1989).

The relative performance of three distinct chemicals, including chromic acid (H_2CrO_4), sodium dichromate (Na_2CrO_4), copper sulphate (CuSO_4), and their mixtures, was determined by Palomer *et al.* (1989). The treatment solutions were applied at a rate of 152 g/m to the sawn wood surface of coconut timber and left outside in an inclination facing south. After three years, he discovered that the H_2CrO_4 and Na_2CrO_4 treatment combination outperformed all other treatment options.

Coconut palm sawn wood was preserved using copper chromated arsenate (CCA) under pressure and boric acid under a diffusion process. From mature to juvenile palms, CCA's dry salt retention varied from 5.9 to 8.9 kgm^{-3} , but boric acid's was low. As palm age and wood density increased, the dry salt retention (DSR) of preservative compounds dropped. The high negative correlation between density and DSR discovered in this investigation suggests that preservative solution strength may need to be altered in order to achieve the desired chemical retention. This study demonstrates that the stem wood of wilt-affected coconut palms may be treated to effectively retain chemicals, and that the type of preservative and treatment can be chosen depending on the final application (Gnanaharan and Dhamodaran, 1989).

Heat Pressure Sap Displacement (HPSD) can use CCA preservative to treat Coconut wood poles with a minimum pressure of 1.05 kg/m^2 . The amount of time needed for treatment depended on how long the stems had been cut. At a CCA concentration of

6%, an average retention of 9.81 kg/m³ based on average volume was achieved. Retention would be 14.01 kg/m³, which is close to the minimum standard requirement of 16 kg/m³ if 70% of the entire volume were treated. By increasing the preservative concentration to 8%, retention can be improved (Siriban and Pabuayan, 1990).

The fire resistance of untreated and treated coconut timber was examined using a fire-tube test. A minimum retention of 48–64 kg dry salt per cubic metre of Pyrolith M on coconut timber was found to be efficient in halting the spread of fire, according to test findings using ASTM Standard E69–50. This preservation offered protection that exceeded the BBC, BOCAI acceptance criteria for the fire-tube test of wood, which allowed for a maximum weight loss of 20%. Five different concentrations of a fire-retardant chemical were applied to samples of coconut timber. A treatment efficiency of 50% offered adequate fire hazard defence. To get the necessary loading or retention, treatment by pressure, which outperformed other techniques including brushing, dipping, and soaking, is advised by German *et al.* (1997).

According to Bailleres *et al.* (2010), immediately after sawing, boards should be submerged for 10 seconds in a suitable treatment solution. To find out which stain-control methods are accepted, get in touch with local regulatory agencies and providers of agricultural chemicals. According to research, a treatment solution with 4.5% of chlorothalonil and 1% of carbendazim is effective at reducing discoloration.

Jeeshma *et al.* (2017) investigate the inorganic preservation of coconut wood utilising the treatment methods of pressure and diffusion, medium and high densities, and CCB (Copper Chrome Boron at 1 and 2%), BBA (Borax-Boric Acid at 3, 6 and 10%). The study came to the conclusion that pressure therapy had greater retention and penetration than diffusion treatment. Although the BBA advised a dosage between 6 and 10%, at 3% it demonstrated good retention, making a higher concentration uneconomical. In contrast, CCB obtained the target retention rate at 2%.

2.6.2.2.1. Draw-back of inorganic preservative

In cases where the roof serves as a drinking water catchment, CCA is not recommended for roof shingles. A 1% solution based on the active component Methylene-bis-Thiocyanate has proven to be the most effective of the evaluated substitutes. Chlorothalonil-Captafol combo is another preservative, albeit it is less

effective and less long-lasting, and it has a low toxicity rating for humans. It is a chemical that can be purchased as a powder (Timbafol C) and is applied as a 4% aqueous solution (Killmann and Fink, 1996).

Brands include Boliden K33, Celcure AP, and Tanalith C. Copper-chromium-boron and copper-chromium fluorine are further harmful salts (Killmann and Fink, 1996). CCA and NaPCP were outlawed in many countries, including India, due to their severe human toxicity (Mosteiro, 1971).

Although standard wood preservatives like Creosote, CCA, CCB, ACQ and others have been shown to be quite efficient against wood-eating organisms, some of them have been linked to environmental pollution and some have even been shown to be harmful to both humans and animals (Thompson, 1971)

Numerous negative effects result from the leaching of these inorganic compounds into soil and water systems. Although the endurance of the wood was successfully increased by these compounds, the toxicity to mammals must be disregarded. It was well recognised that chromium and arsenic were carcinogenic substances (Onuorah, 2000).

Previous research (Khan *et al.*, 2006; Moghaddam and Mulligan, 2008) found that chemical leaching raised disposal concerns all around the world.

2.6.2.3. Organic based

As a result of severe drawbacks of conventional wood preservation, there has been a significant increase in interest in creating environmentally friendly wood preservatives that don't harm human health over the past few decades (Onuorah, 2000). There has been an ongoing hunt for various bio-control strategies to preserve wood. Researchers are working to create environmentally friendly preservatives (Xu *et al.*, 2013). Here are some of the organic based preservatives that are discussed in the literature.

By treating with sapwood blocks of *Ceiba pentandra*, extracts from the species *Milicia excelsa* and *Erythrophelum suaveolens* at 40% each and 60% methanol were tested for their ability to prevent attack by *Lenzites trabea* (brown rot-fungi) or by *Polyporus versicolor* (white rot-fungi). The study found that these extracts successfully

prevented the fungal attack with a percent weight loss of less than 20-30% (Onuorah, 2000).

Neem oil's effectiveness with the addition of binding and bittering chemicals was discussed by Subbaraman and Brucker (2001). On *Bambusa vulgaris*, heated neem seed oil was also applied at a temperature of 60 degrees Celsius. For water resistance, anti-swelling effectiveness, and dimensional stability, bamboo is shown improving performance. It was examined whether *Nerium oleander* extract might prevent attacks from white-rot (*Trametes versicolor*) and brown-rot (*Postia placenta*). When the *Nerium* extract from bulb and leaves blended with ethyl alcohol (96%) and treated to Blocks of *Fagus orientalis* (Turkish oriental beech) and *Pinus sylvestris* (Scots pine) wood at a dosage of 0.25, 0.75, 1.50, and 3.0%. The soil block approach was used to expose treated blocks to *P. placenta* and *T. versicolor* attack for 12 weeks. The only extract dosages that were shown to be effective at stopping an attack were 0.25 and 0.75% (Goktas *et al.*, 2007).

Nakayamaa and Osbrink (2009) showed that application of Kukui plant (*Aleurites moluccana*) extractive to southern yellow pine showed resistant to termite damage for concentration more than 27%. Neem extractive and neem extractive with combination with copper sulphate and boric acid showed effective control against *Schizophyllum commune* fungi when treated with Mango and rain tree. As neem extractive alone showed weight loss of 4.7% and 4.1% for mango and rain tree respectively. Whereas with combination with copper sulphate and boric acid showed 3.3% and 3 % weight loss for mango and rain tree respectively. Thus concluding as a promising preservation option for enhancing the durability of wood (Islam *et al.*, 2009).

Tectona grandis heartwood extracts were produced by sequential extractions with ethanol-toluene, ethanol, and dichloromethane. The extracts' biological activity against the fungus *Phanerochaete chrysosporium* was then investigated. According to the findings, ethanol-toluene and ethanol extracts do not have the same antifungal activity as dichloromethane extract (Bhat *et al.*, 2010).

The 'poison food method' was used to investigate the anti-fungal activity of methanol extracts from the leaves and bark of *Prosopis juliflora* and *Cleistanthus*

collinus. The growth of the brown rot and white rot fungus was shown to be effectively inhibited by the extracts (Jain *et al.*, 2011).

The heartwood of *Madhuca utilis* contains farnesol (44%), thymol methyl ether (29%), terpinen-4-ol (38%) and -terpinene (16%). The *Neobalanocarpus heimii*, in contrast, has benzyl carbinol (62%) and benzyl isoamyl ether (34%) in the bark and eicosane (C20) (50%) and cyclopentanone (19%) in the heartwood. This study found that against *Coptotermes gestroi* termites from *Madhuca* and *Neobalanocarpus*, respectively, there was 100% and 99% mortality. Hence, a conclusion about anti-termite activity (Kadir *et al.*, 2014).

Xu *et al.* (2013) treated bamboo with *Cinnamomom camphor* extractive (10%) and Melamine-modified urea formaldehyde (MUF) (10%). The heat resistance and decay resistance to *Gloeophyllum* and *Phanerochaete* fungi was good in case of camphor treated bamboo in combination with MUF. Li *et al.*, (2013) investigation on bamboos demonstrated the efficiency of Camphor extracts against the brown rot decay fungi *Gloeophyllum trabeum* with a mass loss of 13.08% when treated to *Pinus massoniana*.

Oligoporus placenta and *Trametes versicolor* are inhibited by extracts made from sawdust of *Acacia auriculiformis*. It was discovered that 1% inhibited 47.3% and 15.3% when treated with bark extract, while 0.5% inhibited 29.40% and 46.80% (Poonia *et al.*, 2022).

2.6.2.3.1. Drawbacks of organic preservative

From the studies stated above, it is clear that only a small number of trials have demonstrated effectiveness against wood-degrading organisms at low concentrations. In some instances, we can see that it has demonstrated good effectiveness when used in conjunction with other inorganic preservative types. According to several investigations, extracts from the study Onuorah (2000) have been linked to bronchial asthma and skin irritability and also seed in *Erythrophleum guinensis* extractive (Ofori, 1985). In addition, this approach is a low-cost, environmentally beneficial preservative. The following list includes several organic preservative disadvantages.

Xu *et al.* (2013) treated bamboo with *Cinnamomom camphor* extractive and Melamine-modified urea formaldehyde (MUF) in 10% each. The heat resistance and

decay resistance to *Gloeophyllum* and *Phanerochaete* fungi was low in case of camphor treated bamboo whereas with combination with MUF showed good results.

Onuorah (2000) concluded that only extract dosages of 48.056 and 96.11 kg/m³ (3.0 and 6.0 lb/ft³) were found efficacious in suppressing fungal attack. In the study Poonia *et al.* (2022), only bark extract was effective against wood destroying fungi, while leaf extract was not efficient in preserving the wood.

These extracts were also combined with potassium dichromate and copper sulphate to substitute the arsenic ingredient in copper chrome arsenic (CCA) preservative, and their effectiveness against wood rotting fungus was assessed. The outcomes demonstrated that both complex mixes provided reliable defence against wood-decaying fungus (Jain *et al.*, 2011). From the study, Li *et al.* (2013), camphor extract was not as effective in controlling brown rot fungus when compare with borax acid, ACQ (Ammoniacal Copper Quat). Kukui plant extractives at 5.28%, 9.09%, and 16.56% concentration show no resistance to termite attack Nakayamaa and Osbrink (2009).

2.6.2.4. Nano-wood preservation

The search for a novel way of preserving wood in the early 2000 led to the boom of Nanotechnology. By manipulating shape and size at the nanoscale, nanotechnology entails designing, characterising, and developing materials, devices, and systems (Weir *et al.*, 2008). Due to their characteristics, nanoparticles have significant potential for use in a variety of commercial and consumer applications. The paint industry anticipates that nanomaterials will enhance ink's antibacterial, durable, and water-repellent qualities (Kaiser *et al.*, 2013). The study of particles with novel properties and functionalities that range in size from 1 to 100 nanometres is another emphasis of nanotechnology (Freeman and McIntyre, 2008; Kartal *et al.*, 2009). Metal nano-preparations, like zinc copper, silver, titanium, boron and others could have special qualities that set them apart from elemental metals.

Complete penetration and uniform dispersion would be anticipated if the particle size is less than the diameter of the wood window pit (10,000 nm) or the opening of the bordered pit (400–600 nm) (Freeman and McIntyre, 2008). Pyrolysis-produced nanomaterials have regulated particle size, which might enhance their effectiveness in wood protection applications. Although nanoparticles exhibit high dispersion stability,

Van der Waals forces affect them when they are concentrated (Clausen *et al.*, 2011; Németh *et al.*, 2016).

Some of the notable work of nanoparticles in wood preservation is as follows:

The resilience of *Pinus nigra* (black pine) wood to brown rot after being pressure-treated in an autoclave with nanoscale zinc borate and zinc oxide dispersions was examined in this study. Positive outcomes from the two zinc borate-based formulations suggest fungicide actions of the metal nanoparticles on *Coniophora puteana*. The mean weight losses for black pine sapwood treated to this fungus were extremely small, coming up at 0.34% and 0.54% for one with binder (11.1% concentration) and without binder (3% concentration), respectively. On the other hand, impregnating pine wood with nanoscale zinc oxide (2.1% concentration) produced only modest protection has a loss of 35.9% of weight. As a result, zinc borate in Nano formulations can be used to give wood resistance against brown rot (Lykidis *et al.*, 2013).

Using nanoparticles, the study assessed the resilience of black pine wood to mould, decaying fungus, and subterranean termites. The results revealed that although other nanometal preparations did not inhibit mould fungus, nano zinc borate did so marginally. *Trametes versicolor*'s fungal assault was greatly reduced by zinc-based preparations, whereas copper-based therapies had less of an impact on subterranean termites (Mantanis *et al.*, 2014).

The study assessed the *Poria placenta*, a brown rot fungus, resistance of spruce, beech, poplar, and pine wood treated with zinc nanoparticles. According to the findings, brown rot was suppressed by nano-zinc, with softwoods retaining more of it. For both groups, the nano-zinc solution decreased the percentage of mass loss. Zinc nanoparticles demonstrated favourable qualities for wood preservation overall (Németh *et al.*, 2016).

Zinc oxide nanometer-sized coatings were applied to polycarbonate plates, glass plates, and impregnated wood. After 1500 hours of artificial weathering, the wood treatments showed less fading and better optical characteristics (Weichelt *et al.*, 2011).

The study investigates a low-cost, environmentally benign way of creating metal nanoparticles for wood protection. It makes use of copper oxide nanoparticles and plant extracts with wood-preserving characteristics. Termites and wood decay fungi are

examined using an agar and gravel test on the synthesised plant extracts and nanoparticles. A stable, ecologically friendly wood preservative compound is being developed as a result of additional study that has produced preliminary results that are encouraging (Shiny *et al.*, 2019).

Due to its solitary behaviour and protracted lifecycle, *Lyctus africanus*, a significant dry wood pest in tropical climates, is difficult to treat. Despite being effective, today's insecticides, particularly metal salts, can pollute the environment. In this project, metal nanoparticles will be created and used to treat wood in an economical and environmentally responsible manner using plant extracts having wood-preserving capabilities. The combination of *Lantana camara* leaf extract and copper oxide nanoparticles successfully defended treated rubberwood blocks from *Lyctus africanus* attack (Shiny and Sundararaj, 2021).

Holy *et al.* (2022) study examines the use of nanoscale chemical compounds, particularly zinc oxide, titanium oxide, aluminium oxide, and magnesium oxide, in wood preservation. The findings demonstrate that Scots pine wood's physical and thermal qualities can be enhanced by nanoparticles impregnated with 10% polymethylmethacrylate (PMMA), and that these nanoparticles can also biodegrade *Coniophora puteana*, a fungus that causes brown rot. The nanoparticles demonstrated effective defence against the studied fungus, with Al₂O₃ degrading at the fastest rate (3.88%). The nanoparticles are thought to be excellent for enhancing pine wood's durability.

Bak and Németh, (2018) study evaluated the effectiveness of five nanoparticles (zinc-oxide, zinc-borate, silver, copper, and copper-borate) against *Coniophora puteana* and *Coriolus versicolor* in wood species beech and pine sapwood. Results showed diverse fungi tolerance, with the most effective treatments containing borate. Zinc-oxide, copper, and silver nanoparticles showed high resistance to leaching, while zinc-borate and copper-borate showed low resistance.

Leach resistance and UV protection of southern pine specimens treated with nano-zinc oxide were assessed in the study. After a year of exposure to the elements, the results showed no leaching, chemical depletion, or loss of UV protection. Significant

resistance to water absorption was also provided by nano-ZnO treatment, pointing to a potential application in new wood preservative compositions (Clausen *et al.*, 2010).

The study investigates nanomaterials' potential for wood preservation. Termite mortality, termite feeding inhibition, decay inhibition, leach resistance, and other properties of nanozinc oxide, a nanometal, were discovered. The study also discovered that nanoZnO efficiently prevents termite feeding and mould growth but not degradation or mould. Despite these advantages, nano ZnO could not be an adequate stand-alone treatment for mould or deterioration (Clausen *et al.*, 2009).

The effectiveness of southern yellow pine wood treated with copper, zinc, or boron nanoparticles against mould fungi, decay fungi, and Eastern subterranean termites was assessed in the study, as well as its leachability. In comparison to metal oxide controls, the results demonstrated that nano copper, nano zinc, and nano zinc plus silver with surfactant resisted leaching. Additionally, *Trametes versicolor*, a white-rot fungus, acquired significant resistance to nanometals, which also prevented termites from feeding (Kartal *et al.*, 2009).

Clausen *et al.* (2011) examines the role of leach resistance and nano-zinc oxide particle size in termite mortality following exposure to wood treated with ZnO particles. For leach resistance and termite resistance, sapwood from southern yellow pine was compared to wood that had been treated with soluble zinc sulphate. Less than 4% of the wood treated with particle nano-ZnO leached, while 13 to 25% of the wood treated with soluble ZnO leached. Less than 10% of the leached nano-ZnO-treated wood was devoured by eastern subterranean termites, who also had greater mortality rates.

The study examines the impact of nanoparticle size on copper leaching in treated southern pine sapwood. Results show that size significantly influences leaching, with 50nm nanoparticles leaching slightly less than the control while 10nm leached more. Polymer stabilizers also increase nanoparticle leaching (Ding *et al.*, 2013).

The study tested the Scots pine wood's resilience to rot, mould fungus, and subterranean termites after being treated with nanoparticles of ZnO, B₂O₃, CuO, TiO₂, and SnO₂. While other chemicals leached out, the nano-ZnO compound proved resistant to leaching. Nano-ZnO and nano-B₂O₃ prevented weight loss brought on by fungus-related attacks on mould. Beneficial termite resistance was developed by nano-CuO and

nano- B_2O_3 treatments. Nano-compounds did, however, marginally lessen the effect of weathering (Terzi *et al.*, 2016).

On samples of European beech and Scots pine sapwood, the study examined the antifungal properties of copper and silver nanoparticles. According to the findings, copper nanoparticles typically leach between 15 and 35 percent from pine sapwood, which had the highest retention value. The leaching rate for silver nanoparticles was 15% lower. The treatment with nano-copper had the strongest antifungal impact, but after leaching, this effect was minimal (Pařil *et al.*, 2017).

MATERIALS AND METHODS

3. MATERIALS AND METHODS

The present study entitled “Preservation of coconut wood using inorganic nanoparticles” was conducted at the Department of Forest Products and Utilisation, College of Forestry, Kerala Agricultural University in 2022-23. Various tests were conducted to characterize the nanoparticles and to quantify the effect of nanoparticles as wood preservatives on the coconut wood following standard procedures are discussed in this section. The workflow adopted is discussed under the following heading as shown below.

3.1. Collection of wood samples

For the study, seasoned coconut (*Cocos nucifera* L.) stem wood scantling and planks were collected from the coconut wood workshop, College of Forestry, Vellanikkara (Plate 1A & 2A). The coconut wood was aged between 30-40 years old.

3.2. Preparation of wood samples

Collected wood scantling and planks were converted to cubes and planks of varied standard sizes to carry out different experiments. First, scantlings were planned to remove uneven surfaces and texture. After it was marked to the required dimension, it was cut to remove low-density wood and later reduced to the size (Plate 1A) as prescribed by ASTM (American Society for Testing Materials) and AWPA (American Wood Protection Association) standards (Table 1). Similarly, the larger planks were sanded to remove uneven surfaces and flakes and reduced to the size prescribed by the ASTM and AWPA standards (Plate 1B). Converted wood samples were sanded using sandpaper to make the surface smooth by removing grains, and flakes formed during conversion (Plate 2A).

3.2.1. Cleaning of wood samples

Coconut is a monocot wood as no vessels, only tracheid's with border pits for conduction (Murry, 1977). To remove extractives and blockage present in tracheid's, wood samples were soaked in warmed Acetone for 24 hours (Ding *et al.*, 2011). As a results of this it improves the penetration of nanoparticles into the wood. The Plate 2B shows a clear demonstration of coconut wood samples before and after cleaning.

Table 1: Standard sample sizes of coconut wood prescribed for various tests.

Sl. No.	Test	Type of sample	Size of the sample	References
1.	Leachability	Blocks	1.9 cm × 1.9 cm × 1.9 cm	AWPA E11 (2012)
2.	Chemical retention	Blocks	1.9 cm × 1.9 cm × 1.9 cm	AWPA A21 (1983)
3.	Subterranean termite test (no-choice test)	Blocks	2.5 cm × 2.5 cm × 2.5 cm	AWPA E1 (2009); ASTM D3345 (2022)
4.	Weatherability	Planks	7.2 cm × 5.2 cm × 1.4 cm	ASTM D2898, (2017); Visual examination (Clausen <i>et al.</i> , 2010)
5.	Water absorption (24 hrs.)	Planks	7.2 cm × 5.2 cm × 1.4 cm	ASTM D1037 (2020)

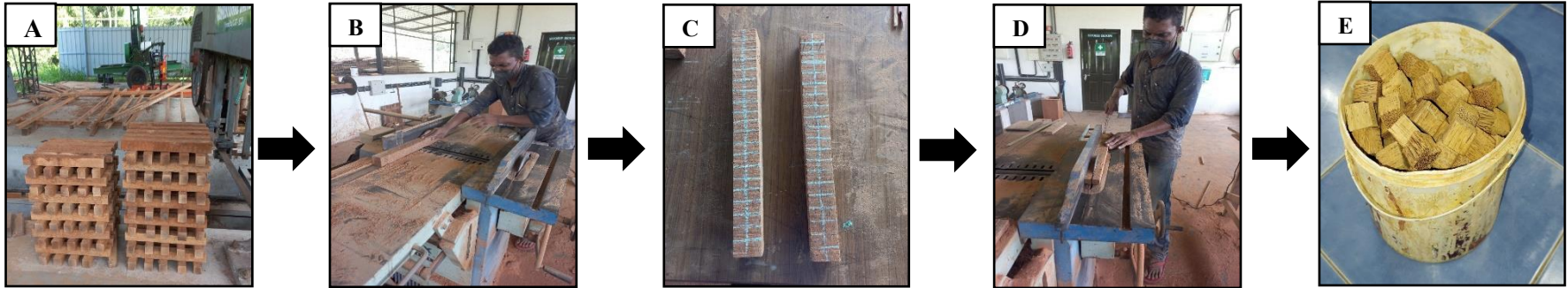


Plate 1A: Conversion of coconut wood scantling into cubes

A) Coconut wood scantlings; B) Planning of scantling; C) Marking of scantling; D) Cutting of scantling; F) Un-sanded coconut cubes

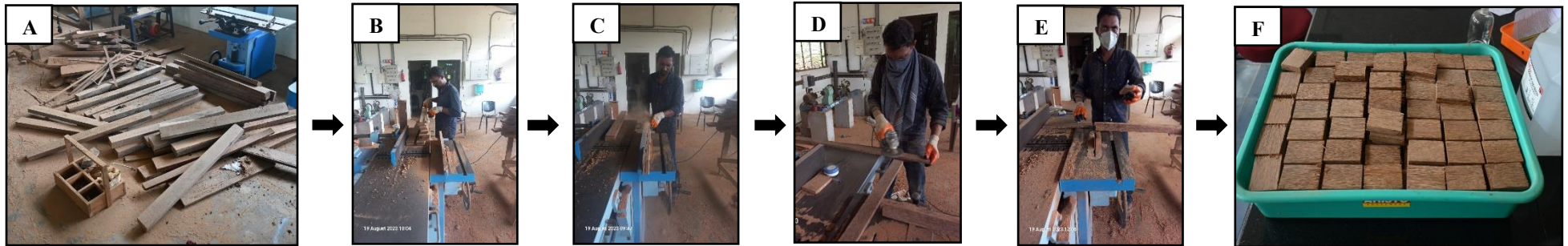


Plate 1B: Resizing of coconut wood planks

A) Coconut wood planks used to make cubes; B) Measuring of planks; C) Sizing of planks; D) Sanding of planks; E) Conversion to definite size; F) Un-sanded planks

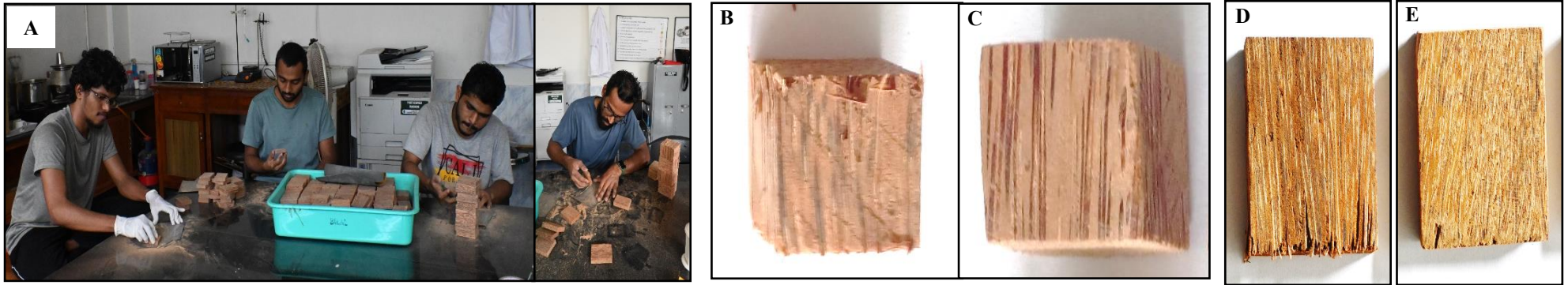


Plate 2A: Sanding of coconut wood samples

A) Coconut wood samples sanding; B) & C) Coconut Wood cubes before and after sanding, respectively; D) & E) Coconut wood cubes, planks before and after sanding, respectively

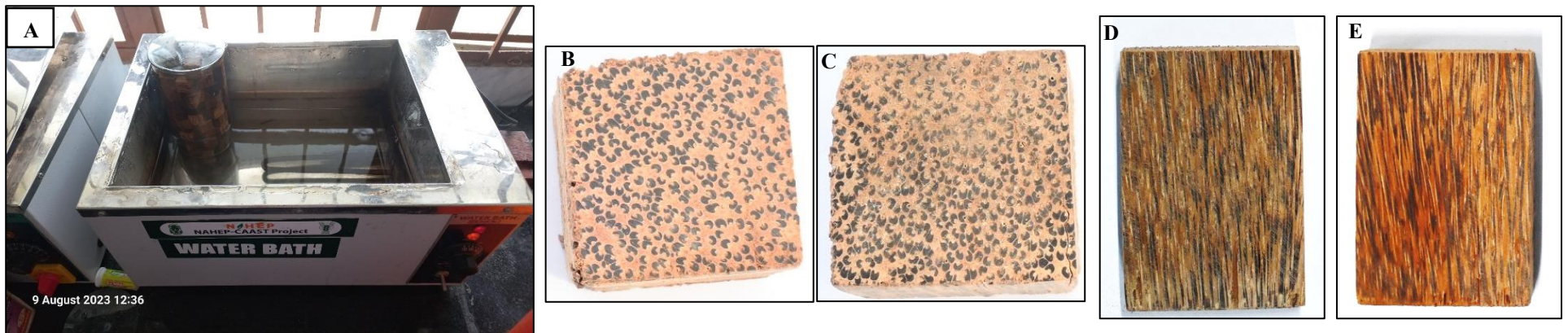


Plate 2B: Experimental setup for cleaning of wood samples

A) Coconut Wood sample soaked in Acetone; B) & C) Coconut wood cube before and after cleaning with acetone; D) & E) Coconut wood plank before and after cleaning with acetone

3.3. Measurement of wood samples

3.3.1. Estimation of green volume (V_g)

The green volume of the few randomly selected samples was calculated according to ASTM D2395 (2017), which defined green volume as the volume of the wood specimen before any shrinkage occurs due to drying of moisture content below the fiber saturation point (about 30%). The partially dried wood samples were soaked in distilled water for about 24 hours until they reached a fully swollen condition above the fiber saturation point, and the sample volume was estimated by measuring dimensions using digital vernier calliper (Plate 3A).

3.3.2. Estimation of density (ρ_o):

After green volume estimation, wood samples were dried in a hot air oven at a temperature of 103 ± 2 °C till they reached constant weight. The final weight was measured using a weighing balance (Shimadzu electronic balances AUX220) with 0.001g accuracy and noted as oven-dry weight (Plate 3B). The basic density for both cubes and planks were measured based on the oven dry mass/weight of a specimen and its green volume (ASTM D2395, 2017). It is expressed as a kilogram per meter cube (kg m^{-3}).

$$\text{Density } (\rho_o) = W_o/V_g$$

Where, W_o =Oven dry mass (kg)

$$V_g=\text{Green volume (m}^3\text{)}$$

3.3.3. Grading of wood samples:

Based on the basic density, wood samples were categorised into high-density wood ($> 600 \text{ kg m}^{-3}$) and medium-density wood ($450\text{-}600 \text{ kg m}^{-3}$) for further treatment application (Plate 3B).

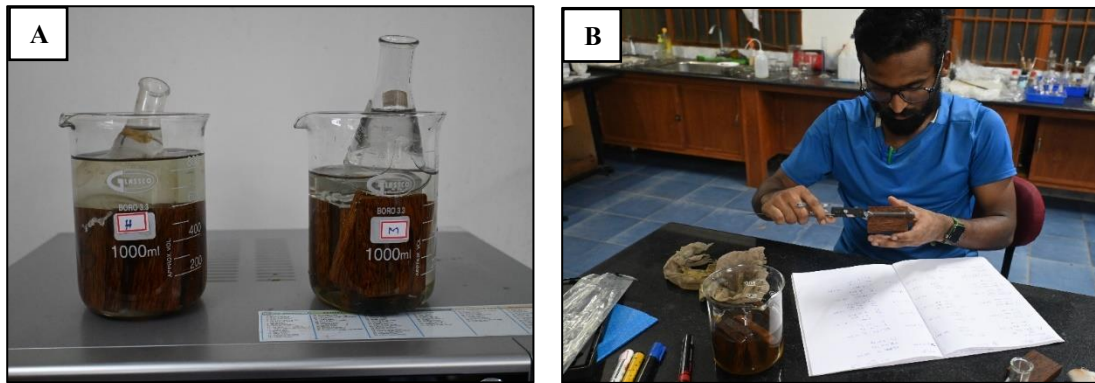


Plate 3A: Experimental setup of estimation of green volume

A) Wood samples immersed,
and

B) Measuring of coconut wood sample dimensions

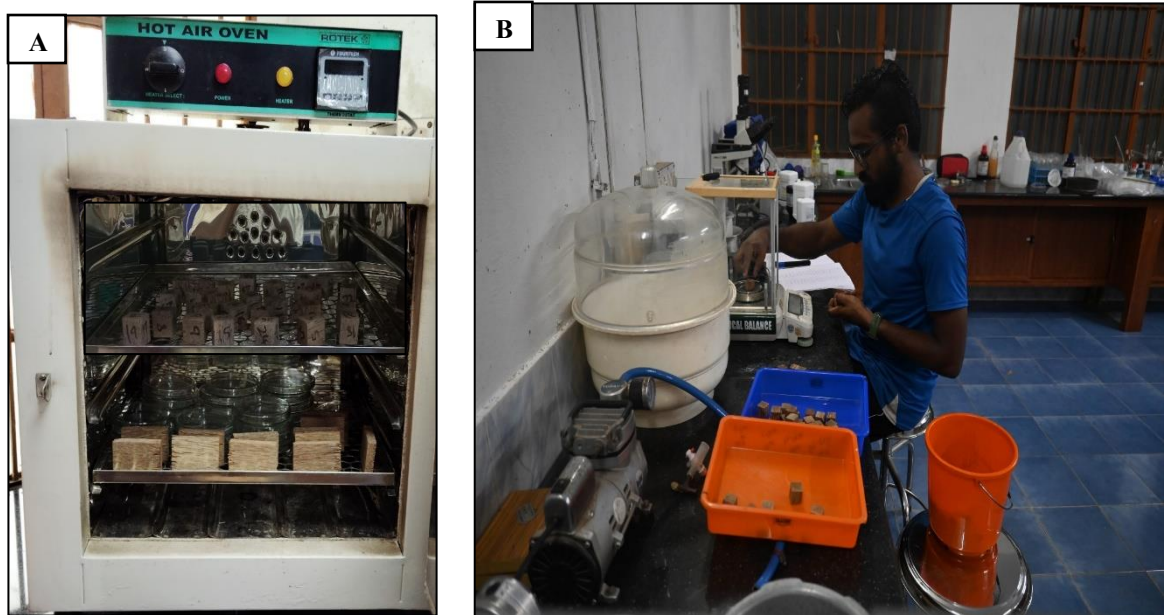


Plate 3B: Estimation of oven dry weight and grading of samples

A) Oven drying of cubes & planks; B) Weighing of coconut wood
samples and their grading

3.4. Preparation of nanoparticles and fixative solution

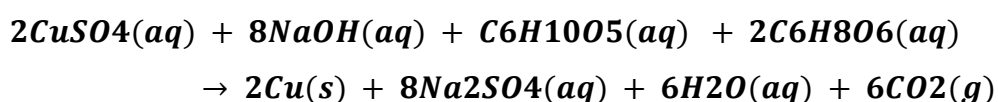
In this experiment, two nanoparticles, namely Copper and Zinc were synthesized and used for wood treatment. The procedure for the preparation of respective nanoparticles is as follows:

3.4.1. Copper nanoparticles synthesis

Copper nanoparticles were prepared by employing a chemical reduction procedure using copper sulphate pentahydrate as a precursor salt, starch as a capping agent, and sodium hydroxide as a reducing agent (Khan *et al.*, 2016).

The first step of the preparation is to prepare a starch solution (1.2%) by adding 1.2g of starch to 120 mL of warm deionized water under magnetic stirring. Allow the solution 5-10 minutes for complete dissolution of starch. After completely dissolving, 0.1 M (2.49g) copper sulphate pentahydrate solution was added with continuous stirring for 30-40 min. The synthesized mixture solution was mixed quickly by adding 50 mL of a 0.2 M (1.7613 g) L- ascorbic acid solution until it turned lemon-yellow colour. The resulting solution was then progressively supplemented with 30 mL of a 1 M sodium hydroxide solution (1.2 g) dropwise while being heated at 80°C for two hours until the solution changed colour from green to ochre and completion of the reaction was marked if the solution remains constant with ochre colour. A pictorial representation of copper nanoparticles is shown in the Plate 4A.

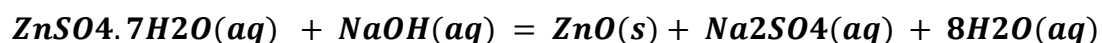
The reaction during the process is as follows:



3.4.2. Zinc nanoparticles synthesis

Zinc nanoparticles were prepared using the modified procedure prescribed by Kumar *et al.* (2013). Sodium hydroxide solution was gradually added to the aqueous zinc sulphate heptahydrate solution dropped-wise in a molar ratio of 1:2 while vigorously stirring for 6 hours leading to the formation of zinc nanoparticles (Plate 4B).

The equation of the reaction during zinc nanoparticle preparation is as follows:



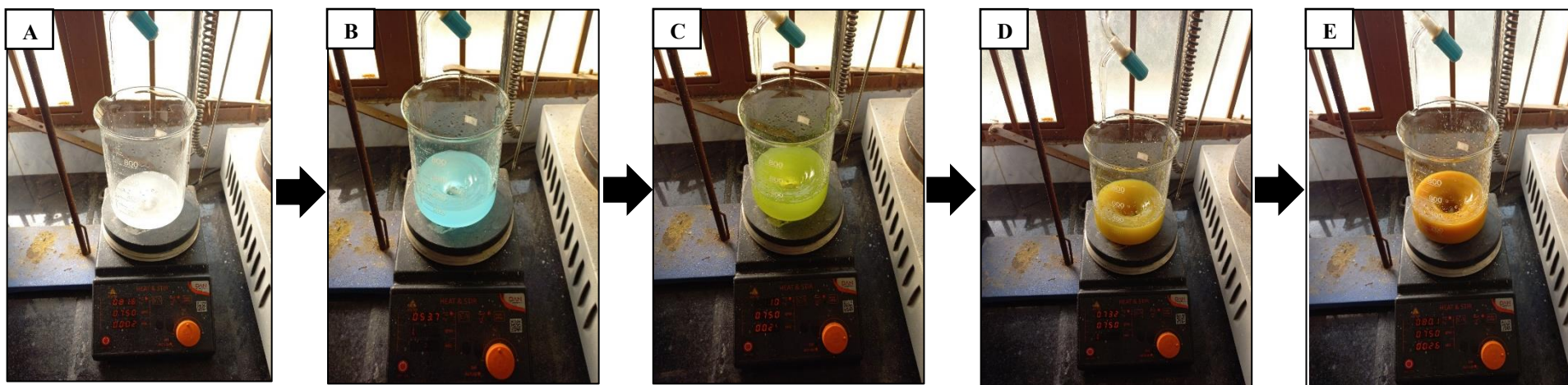


Plate 4A: Flowchart showing preparation of copper nanoparticle

- A) Starch solution (1.2%) under stirring; B) Blue colour solution after copper sulphate solution (0.1M) addition; C) Addition of ascorbic acid (0.2M in 50ml) turns solution into lime yellow;
 D) Reduction of size after adding sodium hydroxide (1M) under stirring; E) Ochre colour copper nanoparticles formed after 2 hr.

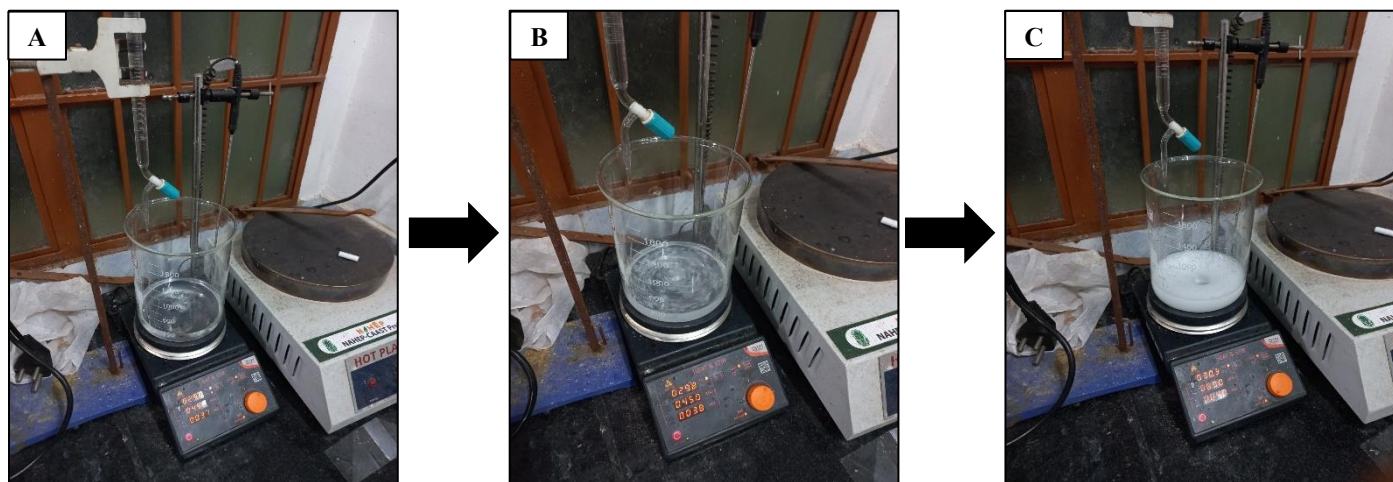


Plate 4B: Flowchart showing preparation of zinc nanoparticles

- A) Zinc sulphate solution (0.1M) under stirring; B) Addition of sodium hydroxide (0.2M); C) Zinc nanoparticles formed after 6hr

3.4.3. Zinc and Copper nanoparticles yield

In order to prepare different concentrations of nanoparticles, the quantity of nanoparticles needs to be known. In order to calculate the quantity of different nanoparticles required, yield of different nanoparticles obtained with respect to the quantity of precursor salts used was determined.

To obtain copper nanoparticle powder, the resultant solution was filtered in what's man Filter paper (No.1) and dried at room temperature or in a hot air oven at room temperature as copper is sensitive to heat. Later it was pulverized into a fine powder using a tube driver. (Plate 5A).

To obtain zinc nanoparticles the precipitate obtained was then filtered using what's man filter paper (No.1) and carefully cleaned with deionized water. The precipitate was dried in a 100°C oven and it was undergone calcination at 500-600 °C pulverized to remove any impurities and later it was freed to fine powder using an tube driver (Plate 5B).

Later, both these powdered nanoparticles were weighed using shimadzu's electronic balances (AUX220) with 0.001g accuracy and yield of respective nanoparticle recorded in grams.

3.5. Characterization of nanoparticles

The characteristics of prepared nanoparticles were analysed using various instruments. For all the characterization tests a sample of 10ml solution was used. To prepare this 10ml solution, 1g of the respective nanoparticles were diluted in 100ml supernatant of the respective nanoparticles. After it was stirred using magnetic stirrer for uniform dispersion. Later, from this solution 10ml was taken using pipette (Plate 6A).

3.5.1. Scanning Electron Microscopy-Energy Dispersive X-ray spectrometer (SEM-EDX)

Scanning electron microscopy (SEM, JEOL JSM-6390 LA) with a detector equipped with an energy-dispersive X-ray spectrometer (EDX) (6390 LA) was used to analyse the morphology and chemical composition of the synthesized nanoparticles. EDX was carried out at an acceleration voltage of 15.0 kV. The powdered, dispersed

nanoparticle samples were dusted onto a piece of double-sided sticky carbon conductive tape attached to a tiny copper stub. Afterwards, an ion sputtering apparatus is used to sputter-coat the sample with gold in order to avoid surface charges and to give a homogenous surface for analysis and imaging.

3.5.2. X-ray diffraction (XRD)

To analyse the degree of crystallinity of prepared nanoparticles XRD analysis was performed on a Bruker D8 Advance diffractometer operated at 40 kV and 30 mA with Cu(K α) radiation (1.54 Å) as a source. With a step width of 0.020°, a sampling time of 57.6 s, a step scan mode was used at 25 °C temperature. The 2 θ had a scanning range of 10 to 80°. To ensure a large surface area would be exposed to the X-rays during data collection, the nanoparticle solution was dried to obtain powder, deposited on top of an aluminium slide, and spread out to cover the designated area.

3.5.3. Fourier Transform-Infrared (FT-IR) Spectroscopy

To determine the presence of functional groups on the produced nanoparticles, FTIR spectroscopy was used. Using Nicolet iS50 spectroscopy coupled with a detector (DTGS KBr) in the range of 100-4000 cm⁻¹ at a 4 cm⁻¹ resolution of 6-32 scans, the sample's FTIR spectrometer was acquired at room temperature and the spectra were captured.

3.6. Preparation of fixative for nano-coating

Fixative is a substance used to lock or fix the chemicals in the wood (Morrell and Lebow, 2005). In this study, D-sorbitol and citric acid can be used as a fixative in wood preservation (Lee *et al.*, 2020). The fixative was prepared according to Larnøy *et al.* (2018) where, D-sorbitol and citric acid were mixed in a 3:1 molar ratio and stirred at 80°C, to obtain a semi-liquid solution (Plate 6B).

For the known quantity of fixative, known grams of copper and zinc nanoparticles were added and stirred to prepare the required concentration of nano-coating. The nano-coating of different concentrations prepared (Plate 6B). As a control, fixative without nanoparticles was used. 1%, 2.5% and 5% are three different concentrations of each zinc and copper nanoparticles were prepared under this study.



Plate 5A: Steps in estimation of copper nanoparticle yield

A) Filtering of sample; B) Drying of sample at room temperature; C) Recovery of dried nanoparticle by scrapping; D) Grinding using tube drive; and E) Weighing of nanoparticles

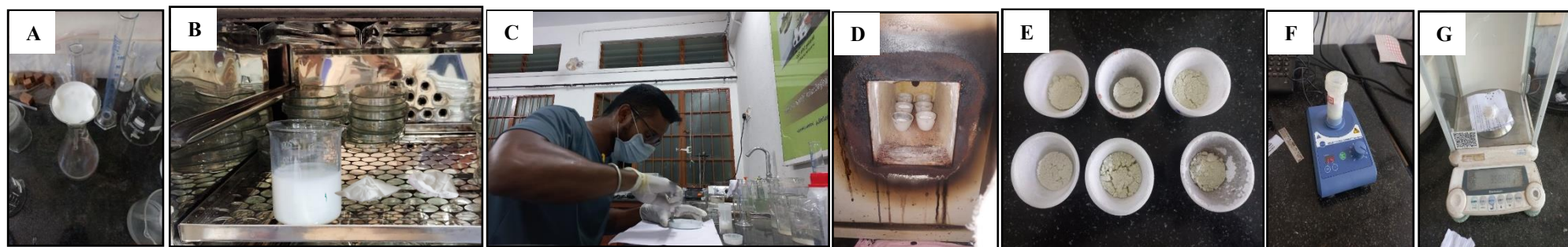


Plate 5B: Steps in estimation of zinc nanoparticle yield

A) Filtering of sample; B) Drying of sample; C) Recovery of dried nanoparticle by scrapping; D) Calcination; E) Recovery of calcined nanoparticles; F) Grinding using tube drive; and G) Weighing of nanoparticles

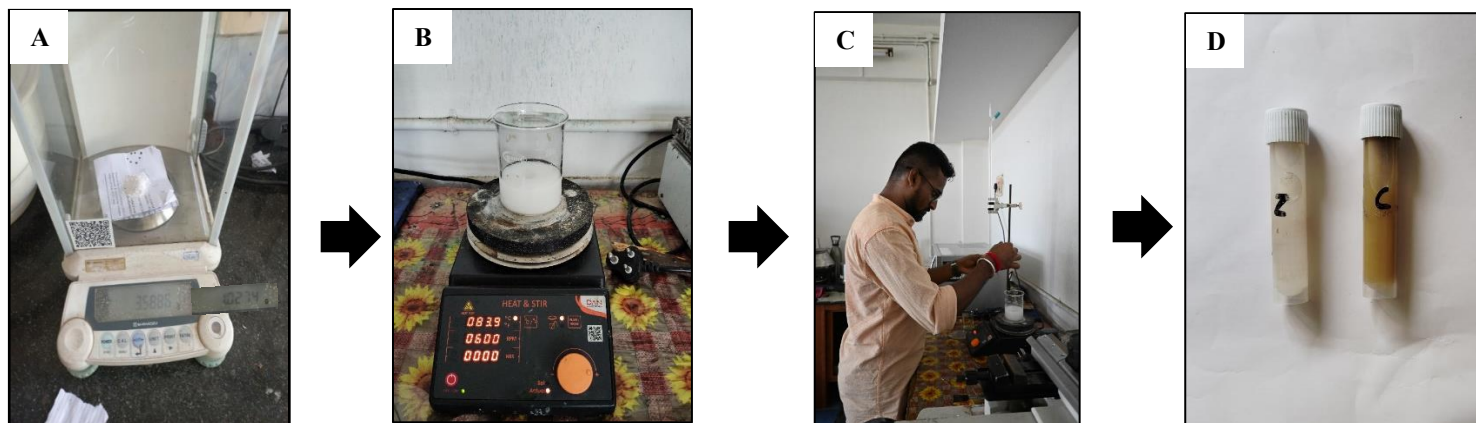


Plate 6A: Sample preparation procedure for nanoparticle characterization

A) Weighing of nanoparticles, B) Stirring of nanoparticle with the supernatant using magnetic stirrer, C) Taking 10ml of solution using pipette, and D) Zinc and copper stored in plastic container before analysis

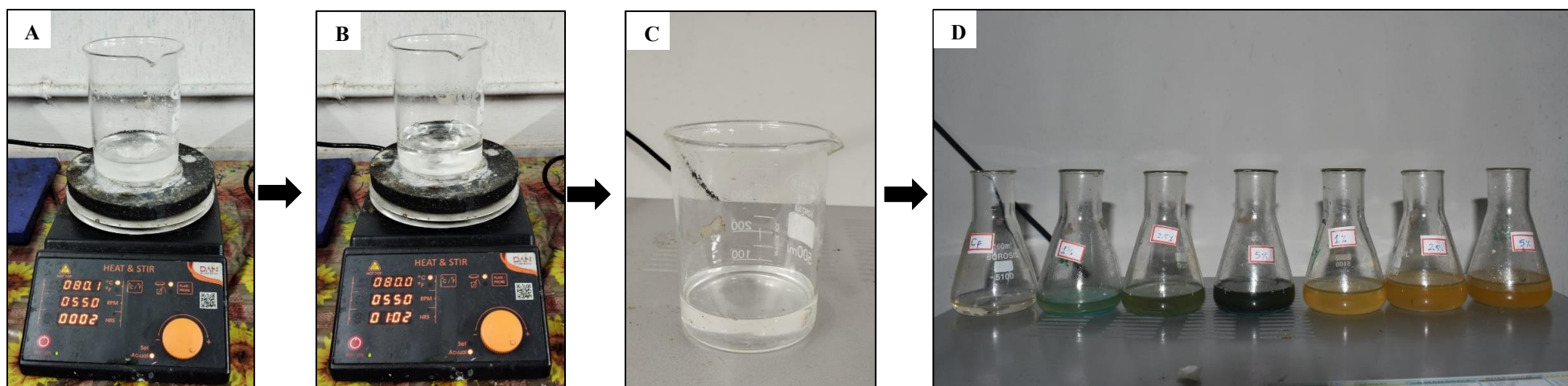


Plate 6B: Images showing steps in preparation of fixatives

A) D-sorbitol dissolved in distilled water at 80°C, B) After adding of citric acid and heating it for 1hr, C) Fixative solution after cooling, and D) Prepared fixative solution used for coating mixed with different concentration of copper, zinc nanoparticles along with control

3.7. Preparation of solution

Based on the yield obtained using respective amount of precursor salts, 1%, 2.5% and 5% concentration of copper nanoparticles were prepared and dispersed with supernatant as a solvent using an Ultrasonic Probe Sonicator (ULTRA AUTOSONIC LLP) to avoid agglomeration, to decrease the size of the nanoparticles and its uniform dispersion in the fluid before being it for pressure treatment (Sandhya *et al.*, 2021). Zinc nanoparticles were also prepared with different concentrations after preparation using Ultrasonic Probe Sonicator in the similar procedure as followed in copper nanoparticle (Plate 7A & 7B).

3.8. Impregnation with nanoparticles

The preservation work was carried out with two major methods of preservative application with three different concentrations, namely 1%, 2.5%, and 5%. The procedures followed in these techniques are as follows:

3.8.1. Pressure treatment

Coconut wood was treated with different nanoparticle concentrations without using a fixative in the pressure treatment chamber. This is suitable for nanoparticles having a size range of 50-100 nm or wood with small pits/pores, usually of size 50-100 nm. Oven dried wood samples of both classes (Medium and High density) with weight difference not exceeding 0.5g among the blocks were subjected to pressure treated with prepared copper and zinc nanoparticles as per modification of ASTM D1413 (1999); AWPA E10 (2012). The chemical treatment plant used in the study is shown in Plate 7C.

Wood samples were placed in a pressure cooker with a weight on them to avoid eventual floating of sample after pouring preservative into the cooker as shown in Plate. First, known concentration of preservative was poured into the pressure cooker (Plate 8B). After closing the pressure cooker's lid, attach the cooker to the vacuum pump and open the valve that separates it from the vacuum. A vacuum-pressure is applied in the treating chamber, maintained at 80-100 mmHg and hold it for 30-45 minutes. At the end of the vacuum period (holding period) the valve of the vacuum pump was closed by followed a pressure of 30-45 psi was applied using cycle pump and hold it for 1-2 hrs. At the end of the pressure period, release the pressure valve and leave the blocks submerged in the treating solution for 30-60 minutes. Take each block out of the solution and use

tissue paper to remove any leftover surface preservative. Keep the sample for further conditioning in a tray. Plate 8 procedure of pressure impregnation of nanoparticles into coconut wood.

3.8.2. Nano-coating treatment

Coconut wood was coated with different nanoparticle concentrations with fixative as a medium using Brush. This is suitable for wood with small pores/pores, usually of broad size of 400-600 nm or slightly even less. Oven dried wood samples of both classes (Medium and High density) were coated with prepared copper and zinc nanoparticles with the help of brush using nano-coating prepared (Section No. 3.4.5). After coating, it was allowed to dry for 12-24 hr.

3.7.3. Conditioning of wood samples

All the impregnated wood samples placed them in tray, in a well aerated, room conditions for 48-72 hours ASTM D1413 (1999); AWP A E10 (2012).

3.7.4. Distribution of nanoparticles in treated wood samples

3.7.4.1. Using SEM-EDX Mapping

The distribution of nanoparticles in treated wood samples was studied by SEM-EDX Mapping for inside the wood when treated with pressure and on the surface of the wood when treated as a coating. Scanning electron microscopy (SEM, JEOL JSM-IT500LA) with a detector equipped with an energy-dispersive X-ray spectrometer (EDX) (500LA) was used to analyse the morphology and chemical composition of the wood samples treated with nanoparticles. EDX was carried out at an acceleration voltage of 20.0 kV.

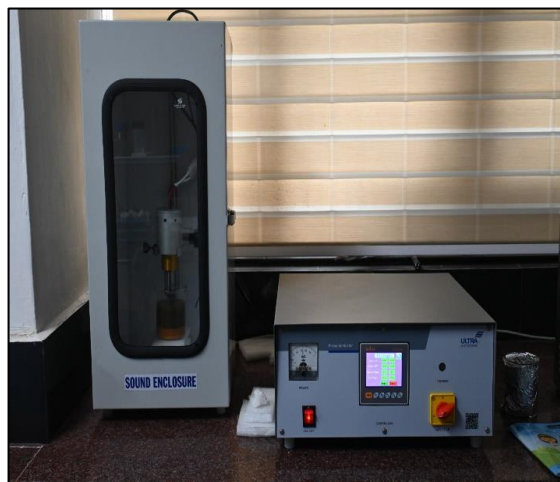


Plate 7A: Ultrasonication of prepared Nanoparticles

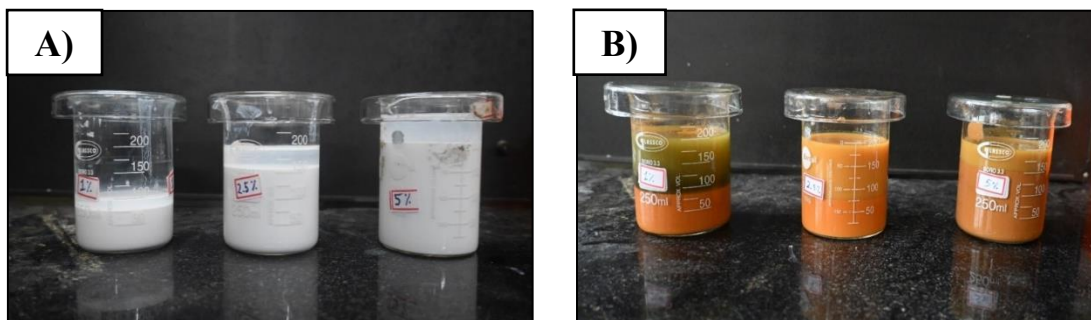


Plate 7B: A) Zinc and B) Copper nanoparticle solution after ultrasonication



Plate 7C: Pressure treatment plant designed and used for the study

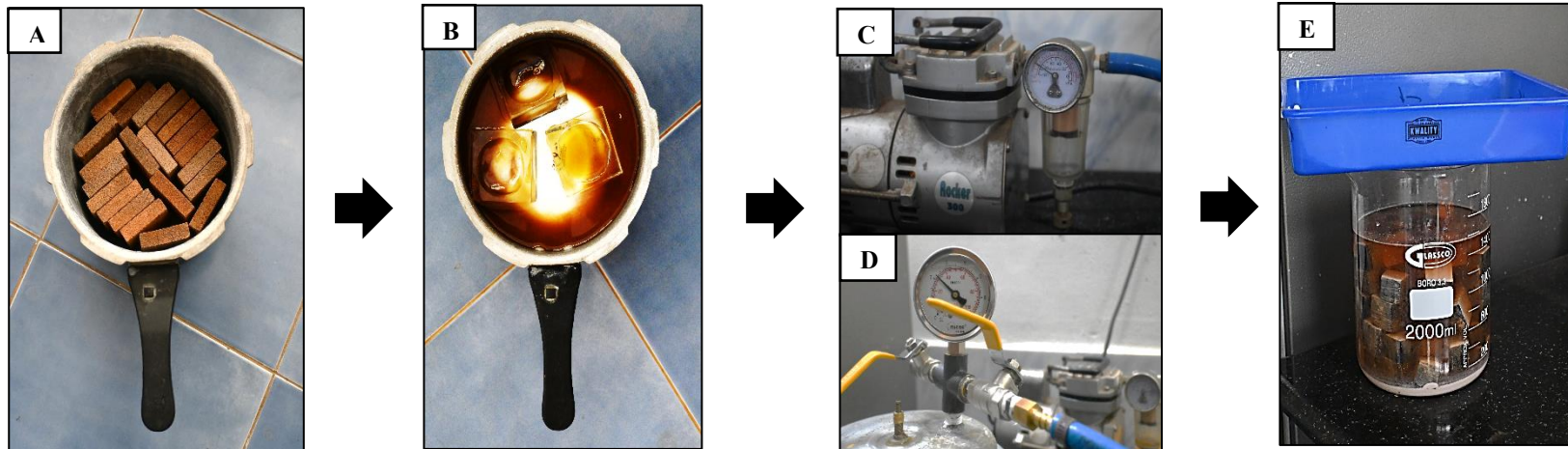


Plate 8A: Steps in pressure treatment of wood

- A) Wood sample stacked inside treatment chamber; B) Wood samples immersed in nanoparticles solution and weighed them down; C) Application of initial vacuum with vacuum pump;
 D) Pressure application; E) Wood samples immersed in nanoparticles solution after removing it from pressure chamber



Plate 8B: Drying of nano-coated coconut wood samples

- A), B) Drying of coconut wood after applying coating of copper and zinc respectively

3.9. Experiments

In order to understand the effectiveness of nanoparticles for wood preservation various tests were carried out according standards recommended by AWPA and ASTM.

Before carrying out the experiment conditioned weight (W_2) of samples was recorded.

Conditioned weight (W_2): Weight of the wood block plus remaining preservative after conditioning and before exposure to various kinds of tests.

3.9.1. Chemical Retention:

In order to quantify the copper and zinc nanoparticles impregnated in different wood samples, conditioned samples of all treatments were selected and grounded using a Willey mill and passed through a mesh screen (Mesh number 30). After it was digested according to di-acid digestion of AWPA A7 (2012) Standards as given below.

0.5g of wood powder was taken in a conical flask with 100ml of water. 10 ml of the acid mixture (mixture of nitric acid concentrated (HNO_3), perchloric acid (HClO_4) in a 9:4 ratio) was added, and thoroughly mixed by swirling. The mixture in a flask was placed in a digestion chamber with low heat. The flask was heated to a high temperature until no longer producing red fumes. Further the volume is decreased to roughly 3-5ml, by evaporation. The liquid turning colourless is a sign that digestion is complete. Add 20 ml of distilled water to the flask after it has cooled, then transfer it to a 100 ml volumetric flask to make up the volume. After filtering the solution, the concentration of copper and zinc was measured separately using AWPA A11 (1983) standards and AAS (Atomic Absorption Spectroscopy). Procedure to estimate chemical retention is shown in the Plate 9. After taking the reading from AAS in ppm (parts per million), deduct the value of blank sample for zinc and copper respectively from each treatments retention values. Blanks sample refers to reading of sample solution without any wood sample in it. Recorded 'ppm' value was converted to kg m^{-3} using the formula given below.

$$\text{Chemical Retention (kg m}^{-3}\text{)} = \text{ppm Value} \times \text{DF} \times \text{SG} / (5000 \times W_g)$$

Where,

$$\text{ppm Value} = \text{Recorded value in AAS,}$$

DF = Dilution factor, = 20 (After digesting the sample, 20 times more volume of water was poured to make it dilute)

SG = Specific gravity of wood

W_g = Weight of the original wood sample

3.9.2. Chemical leaching:

In order to assess the laboratory evaluation of the leaching of nanoparticles from wood expressed as a percentage of the original preservative retention, an accelerated leaching test was conducted according to AWWA E11 (2012) standard procedure.

Five samples per treatment were placed in a 250 ml beaker and submerged in 100 ml of deionized water. The submerged samples were agitated for 14 days continuously with collecting the leached water after 6hr, 1, 2, 4, 6, 8, 10, 12, and 14 days and stored in a glass container. A total of nine readings were taken per treatment. Each collected leached samples were analysed for copper and zinc with AAS (Atomic Absorption Spectroscopy). Total quantities of leached copper and zinc were calculated over the course of time. Percent leaching was calculated by averaging the leaching rate of 5 specimens. Before AAS analysis, it was filtered to remove dirt particles using mull or muslin cloth. Detailed procedure to estimate chemical leaching is shown in the Plate 10.

3.9.3. Subterranean termite test

The laboratory assessment of treated and untreated wood for its resistance to subterranean termites is covered by ASTM D3345 (2022); AWWA E1 (2009).

No-choice test: Termites were given a single option in a test, and the termite's ability to ingest the material was determined by the amount of mass loss. Data on avoidance is required for this test in order to calculate avoidance based on consumption of the treated material, which is frequently treated with substances thought to be insecticidal.

In this study, no-choice test was performed, to evaluate what will be the minimum threshold concentration level for avoiding termites' infestation. A natural, active, large colony of termites was identified in the Bio-resource Park, College of Forestry, Vellanikkara. The weighted samples were tied with metal wire as shown in the Plate 11A.

A clean plastic container was taken with its base removed and a hole was made on the top. The plastic container was covered at the place of slight opening made on top of the termite mound. The samples were tied to the metal wire and placed inside the mound through the small hole in the cap in such a way that, wood samples could be accessed easily by pulling the metal wire for observation without disturbing the colony as shown in the Plate 11.

In each plastic box, one sample block from each treatment was placed. A total of five samples per treatment was used as a replication. Observations were taken in an interval of 7 days to analyse wood degradation. Its population was also monitored by taking photographs periodically. The duration of the experiments was 6-8 weeks. The was concluded when control wood degradation was more than 50%.

At the end of the incubation, wood specimens were oven dried, reconditioned at 27° C and 70% RH, and reweighed to calculate weight loss. The termite avoidance was estimated by presence or absence of live termites after the completion of four weeks. Following incubation, wood specimens were visually rated on a scale of 0-10 with ten being sound and zero for complete degradation according to ASTM D3345 (2022); AWPA E1 (2009) standards (Table 2).

Table 2: Visual scoring or rating used for subterranean termite test (ASTM D3345, 2022; AWPA E1, 2009)

Score	Description
10	Sound
9	Slight attack, up to 5-10% of cross-sectional area affected
7	Moderate attack, 10-30% of cross-sectional area affected
4	Severe attack, 30-50% of cross-sectional area affected
0	Failure, >50-75% of cross-sectional area affected

Final weight (W₃) - At the end of each experiment weight of the test block after test and after final conditioning.

Calculation of weight/mass loss:

At the end of each experiment, the mass change is recorded as shown in the given below formula.

$$\text{Mass Loss (\%)} = \frac{W_3 - W_2}{W_3} \times 100$$

Where,

W_3 = Weight of wood sample after water absorption test

W_2 = Weight of wood sample before water absorption test (Conditioned weight)

3.9.4. Weatherability:

The weatherability test was carried out following modifying standards method C of ASTM D2898 (2017). The treated coconut wood planks along with control both coated and pressure treated were weathered outdoors for 6 weeks and visually evaluated for rain exposure and Natural weathering (*i.e.*, greying) (Plate 12A). Five samples per treatment were placed horizontally on the cemented floor facing direct sunlight (Clausen *et al.*, 2011). The specimen surface in direct light was considered the exposed surface, and the underside of each specimen was considered the unexposed surface for reporting results. At the end of the experiment, mass loss, greying due to natural weathering was done.

Final weight (W_3)- At the end of each experiment weight of the test block after test and after final conditioning.

Calculation of weight/mass loss:

At the end of each experiment, the mass change is recorded as shown in the below formula.

$$\text{Mass Loss (\%)} = \frac{W_3 - W_2}{W_3} \times 100$$

Where,

W_3 = Weight of wood sample after water absorption test

W_2 = Weight of wood sample before water absorption test (Conditioned weight)

3.9.5. Water absorption test

Water absorption test was carried out according to method B-Single continuous 24 hours submersion in water of ASTM D1037 (2020) standards.

After conditioning, recorded W_2 , and dimensions of the sample such as length, width and thickness (one centimetre from the sample's edge on each side, four points were measured for thickness and the average of these measurements will be utilised) to nearest 0.01cm. The samples were submerged vertically under deionized water maintained at room temperature continuously for 24 hours. After 24-hour, weight (W_3) and dimensions of the samples were measured after whipping surface water. The following equation was used to calculate the values of volume swelling percentage (VS %) and water absorption percentage (WA %). Detailed procedure for water absorption test is shown in the Plate 12B.

$$\text{Water Absorption \% (WA \%)} = \frac{W_3 - W_2}{W_3} \times 100$$

$$\text{Volumetric Swelling \% (VS \%)} = \frac{V_3 - V_2}{V_3} \times 100$$

Where,

W_3 = Weight of wood sample after water absorption test

W_2 = Weight of wood sample before water absorption test (conditioned weight)

V_3 = Volume of the wood sample after water absorption test

V_2 = Volume of the wood sample before water absorption test (conditioned volume)

3.10. Statistical Analysis

The present study was carried out to quantify nanoparticles as an effective way to preserve coconut wood. The method of application of preservative pressure without fixative and coating with fixative has been considered as a different entity. Coconut Wood blocks and planks were prepared and sorted into high and medium densities. Copper and zinc with three different concentrations 1%, 2.5% and 5% as a nanoparticle and nanoparticle concentrations was considered as other two factors along with density of wood. Five replications were used in the study and the data was analysed by Nested ANOVA model using R-Studio (Ver. 4.3.0) for different treatment. Control samples were treated with deionized water.

Table 3: List of factors and their levels used in the study

Sl. No.	Factors	Levels
1.	Density	<ol style="list-style-type: none"> 1. Medium density 2. High density
2.	Nanoparticles	<p>Each density had following chemicals</p> <ol style="list-style-type: none"> 1. Copper 2. Zinc 3. Distilled water (control)
3.	Chemicals concentration	<p>Each nanoparticle had following concentration</p> <ol style="list-style-type: none"> 1. 1% 2. 2.5% 3. 5% 4. Control used as a pure form

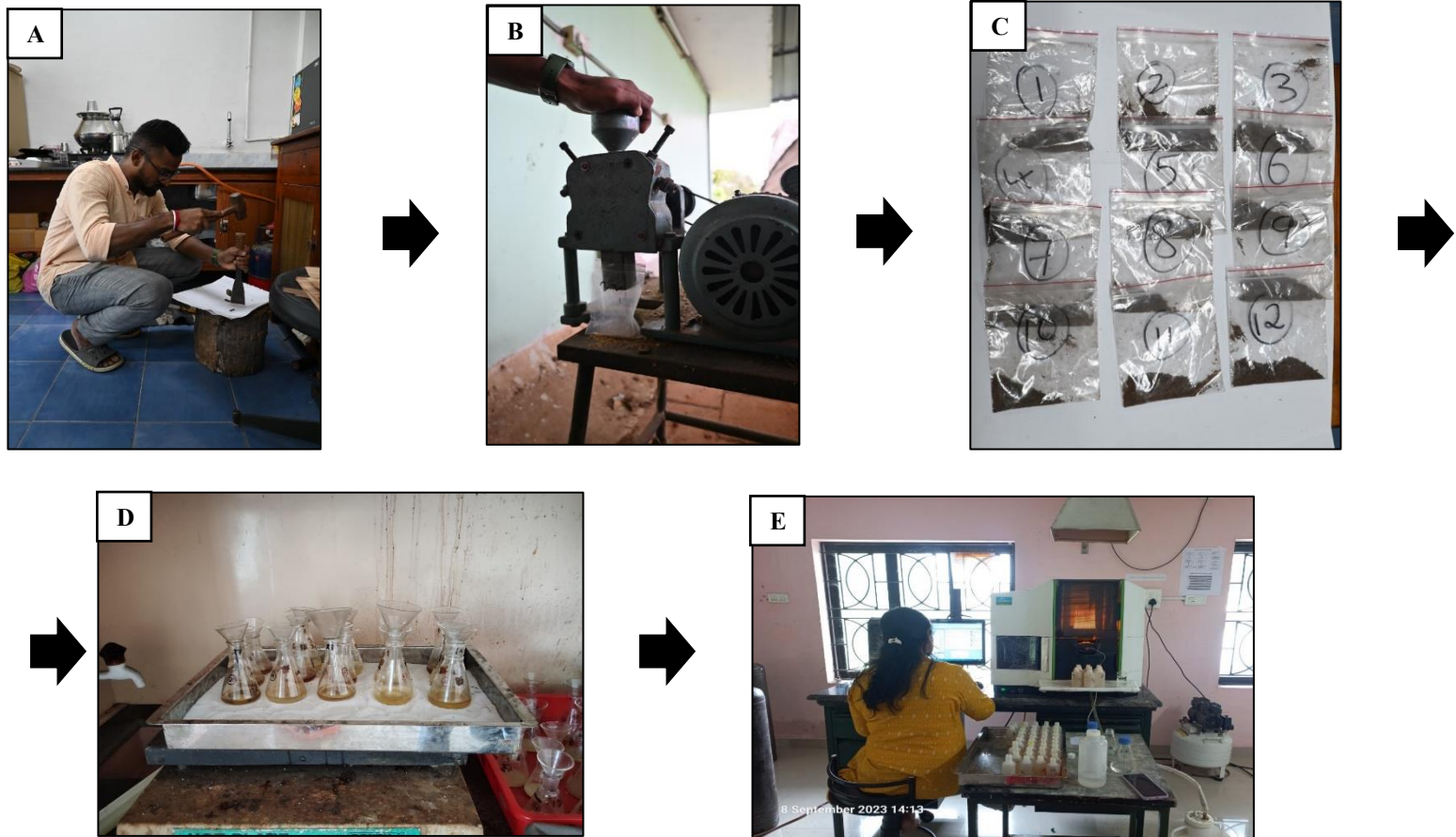


Plate 9: Procedure for chemical retention test

A) Cutting of sample using chisel and hammer; B) Grinding of sample using willey machine; C) Packing of grounded sample; D) After acid digestion of the sample and E) Recording ppm value using Atomic Absorption Spectroscopy (AAS)

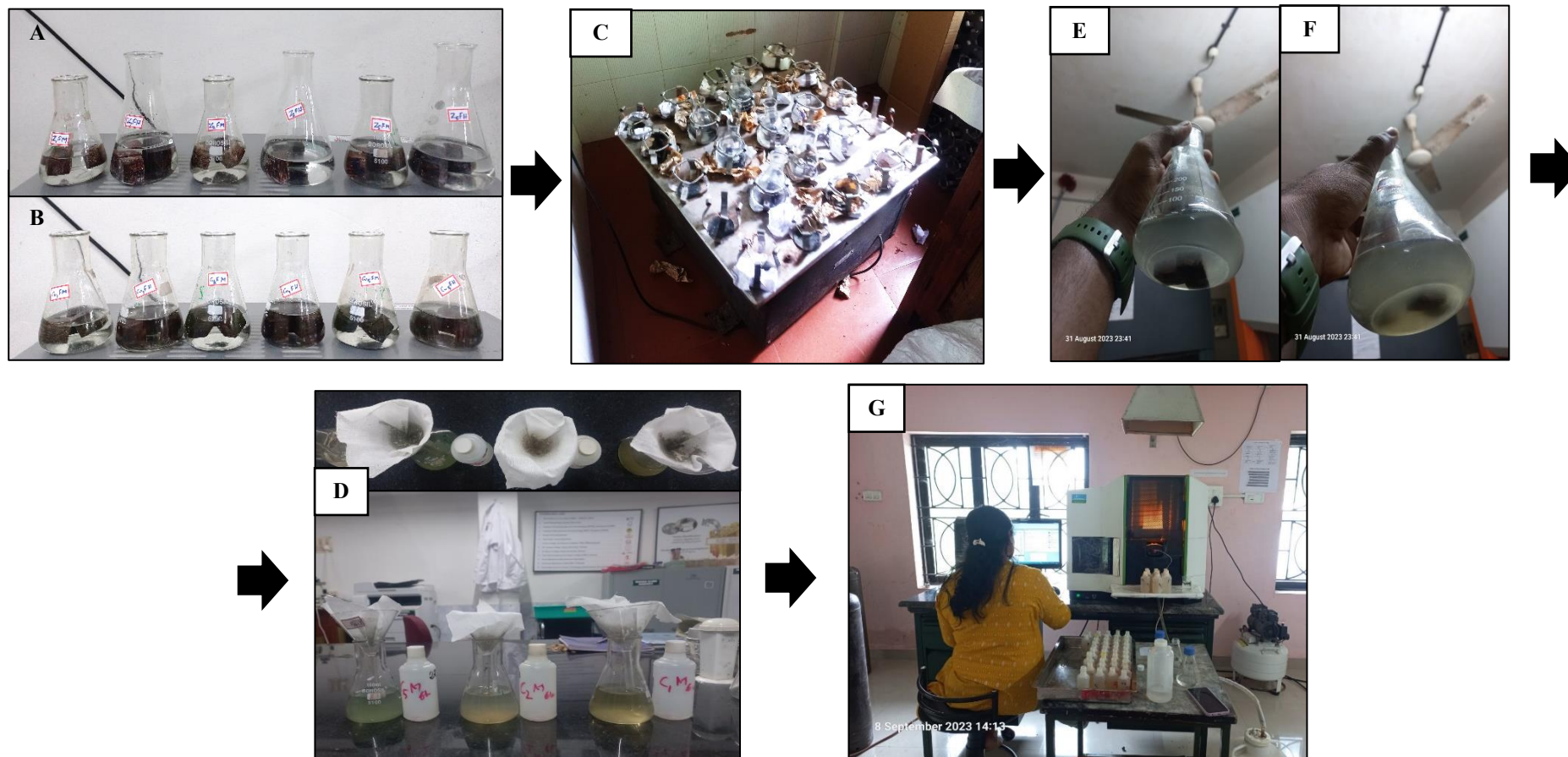


Plate 10: Images showing general procedure of leaching test

A) & B) Wood blocks immersed in deionized water in beaker; C) Shaking of immersed wood blocks using orbital shaker; D) Filtering of nanoparticle leachate; E & F) Leached zinc and copper nanoparticles after leaching experiment; and G) Analysis of leachate using atomic absorption spectroscopy

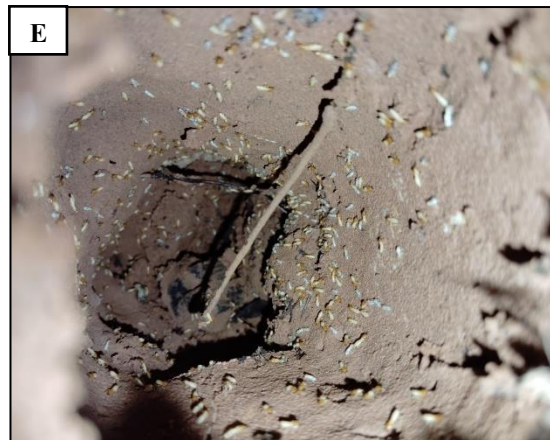


Plate 11: Images showing experimental setup for subterranean termite test
A) Sample tied using galvanized wire; B) Artificial mound; C) No choice test setup; D) Recording observation; E) Termites infestation in the artificial mound; and F) Cleaning of recovered samples



Plate 12A: Weatherability test experimental setup

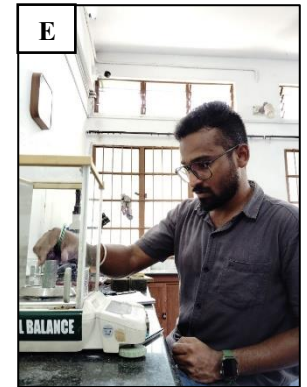


Plate 12B: Water absorption test experimental setup

A) Sample placed in a beaker; B) Weighing it down before pouring deionized water; C) Filling the beaker with deionized water; D) Soaking it for 24 hr; and E) Recording weight

RESULTS AND DISCUSSIONS

4. RESULTS AND DISCUSSION

The key findings of experiments conducted in the study entitled “Preservation of coconut wood using inorganic nanoparticles” are presented in this chapter under the following sub-headings.

4.1. Yield of nanoparticles

The yield of copper and zinc nanoparticles prepared during the study is presented in the Table 4. The copper nanoparticles prepared using copper sulphate pentahydrate (2.49 g) as a precursor salt under chemical reduction process yielded 1.62 whereas, zinc nanoparticles produced from zinc sulphate heptahydrate (14.4 g) precursor salt yielded 10.1 g of zinc nanoparticles by employing precipitation method..

Table 4: Yield of nanoparticles prepared

Sl. No.	Nanoparticle	Precursor salt	Quantity of precursor salt (g)	Average yield of nanoparticles (g)
1	Copper	Copper sulphate pentahydrate	2.49	1.62±0.082
2	Zinc	Zinc sulphate heptahydrate	14.4	10.1±0.635

4.2. Characterization of nanoparticles

The physical and chemical properties of prepared nanoparticles were studied using various techniques such as SEM (Scanning Electron Microscope) and EDX (Energy Dispersive X-ray Spectroscopy), XRD (X-ray Diffraction), and FTIR (Fourier Transform Infrared Spectroscopy). The results obtained during the investigation are presented as follows.

4.2.1. SEM-EDX Analysis

4.2.1.1. SEM

The surface morphology of synthesised copper and zinc nanoparticles were analysed using SEM (Plate 13A. and 13B respectively). The synthesised copper

nanoparticles were irregular cuboidal shaped with a mean size of 173.66 nm. While the synthesised zinc nanoparticles are irregular flake shaped with a mean size of 105.44 nm.

The size and shape of nanoparticles prepared varies with the method of preparation like physical, chemical, and biological methods (Mukherjee *et al.*, 2001), preparation conditions include temperature, pH, time, and solvent (Din and Rehan, 2017), and various chemicals used during preparation like precursor salts, reducing agent, capping or stabilizing or surfactant (Din and Rehan, 2017). In the present study, the copper nanoparticles had an average size of 173.66 nm with an irregular cube shape. With the similar procedure followed by Khan *et al.* (2016) recorded the lowest size of around 25-30 nm, having a cube shape. In the hydrothermal method of synthesis, Samson *et al.* (2019) reported nano-copper with irregular cube-shaped copper oxide nanoparticles with an average of 30 nm size. Following the similar method Sasmal *et al.* (2016) and Mohamed *et al.* (2014) larger sized particles *i.e.*, around 200-500nm with a polygon spherical shape, and 200-800 nm having nano belt shape, respectively. While, by employing the biological method of nanoparticle preparation using the leaf of *Enicostemma axillare*, Mali *et al.* (2019) obtained nano forms of copper having a spherical shape with size ranging from 50-100nm.

Using the precipitation process in the study yielded zinc nanoparticles of flake form and an average size of 105.44 nm. While, Mahalakshmi *et al.* (2019) following the same procedure with different precursor salt (zinc acetate) and reducing agent (sodium carbonate) reported spherical shaped zinc nanoparticles of an average size of 45 nm . A comprehensive study of combination of different precursor salts and reducing agents by Shankar and Rhim, (2019) reported the various shapes and sizes of nanoparticles. Zinc acetate and Zinc chloride as precursor salt with sodium hydroxide as reducing agent produced nanoparticles with 50-100 nm with irregular shape, while zinc chloride as precursor salt and potassium hydroxide as reducing agent reported 50-100nm nanoparticles with irregular shape while, zinc nitrate precursor salt with potassium hydroxide and sodium hydroxide reducing agents have yielded flower shape with cluster of rods of size 25-30 nm and 100-150 nm, respectively. A biological/green synthesis method followed by Yücel *et al.* (2020) using *Lactobacillus reuteri* E81 bacteria to prepare zinc nanoparticle have resulted 96-155 nm nanoparticles with spherical to

hexagonal shape. In the similar manner, Yedurkar *et al.* (2016) reported 80-130 nm nanoparticles with spherical shape using *Ixora coccinea* leaves.

Even after following the same methods the various sizes and shapes of nanoparticles are produced due to varied preparation conditions include temperature, pH, time, solvent, chemicals used and like different precursor salt (copper and zinc based chemicals), surfactant/capping/stabilizing agent (includes cetyltrimethylammonium bromide, polyvinylpyrrolidone, citric acid or others) and reducing agents (includes sodium borohydride, ascorbic acid, sodium hydroxide or others) and their respective concentration and molar ratios (Din and Rehan, 2017).

4.2.1.2. EDX

The energy dispersive spectra of the samples from the SEM-EDX analysis utilised to determine the elemental composition of the synthesised nanoparticles are shown in Figure 1.

The presence of Copper (Cu), and Oxygen (O) was confirmed by the EDX spectrum of the Cu nanoparticles shown in Fig. 2C-A. The peak at 0.5 keV is associated with the binding energy of oxygen (OK_{α}), while the peaks at 0.94, 8.05, and 8.91 keV are associated with CuL_{α} , CuK_{α} , and CuK_{β} , respectively. The weightage percentage of Cu and O in copper nanoparticles is 58.63 % and 41.37 %, respectively (Table 5).

The EDX spectra of the Zinc (Zn) nanoparticles is as shown in Fig 13C-B. The peak at 0.5 keV is associated with the binding energy of oxygen (OK_{α}), while the peaks at 1.01, 8.64, and 9.57 keV are associated with ZnL_{α} , ZnK_{α} , and ZnK_{β} , respectively. While the weightage percentages of Zn and O in zinc nanoparticles are 81.88 % and 18.12 % (Table 6).

Results demonstrated that produced nanoparticles had a maximum percentage of respective elements, *i.e.*, Copper and Zinc.

The presence of different forms of copper nanoparticles (CuK_{β} , CuK_{α} , CuL_{α} , and OK_{α}) were confirmed based on the Khan *et al.* (2016), Sasmal *et al.* (2016), Baqer *et al.* (2018) and Mali *et al.* (2019). Khan *et al.* (2016) confirmed the CuK_{β} , CuK_{α} , CuL_{α} , and OK_{α} particles at an EDX spectroscopy values of 8.94 keV, 8.04 keV, 0.94

keV, and 0.85 keV. In some cases, an addition peak at 2.2 keV indicates the presence of Au (Gold) due to the gold coating of the sample (Khan *et al.*, 2016; Mali *et al.*, 2019) as it improves the conductivity of the sample and results in high-quality resolution image (Leslie and Mitchell, 2007). Sometimes, peak at 0.27 keV represent the presence of $CK\alpha$ which may be due to presence of carbon compounds in chemicals used for preparing nanoparticles or may be due to carbon tape used to mount the sample on the stub or due to interaction of reducing agent or capping agent with copper during bioprocessing (Khan *et al.*, 2016; and Mali *et al.*, 2019).

Table 5: EDX analysis of copper nanoparticles

Element	Weightage %	Atomic Absorption %
Oxygen	41.37	73.7
Copper	58.63	26.3
Total	100	100

Table 6: EDX analysis of zinc nanoparticles

Element	Weightage %	Atomic Absorption %
Oxygen	18.12	47.48
Zinc	81.88	52.52
Total	100	100

The elemental composition using EDX analysis of prepared copper nanoparticle was 58.63% of copper and 41.37% of oxygen. The weight percentage of copper (83.75%) was less than the value reported by Khan *et al.* (2016) but more than the value reported by Mali *et al.* (2019) with copper had 25.14%. In contrast, Khan *et al.* (2016) reported oxygen had 11.05%, less value than our study while, Mali *et al.* (2019) had 56.06%. The variation in the weight percentage of Cu and O may be due to the oxidation of copper when exposed to air while preparing (Feng *et al.*, 2012; Baqer *et al.*, 2018) or an increase in the concentration of the reducing agent leads to the oxidation of copper or copper oxide to cuprous oxide as shown in the below equation (Zaafarany and Boller, 2009).

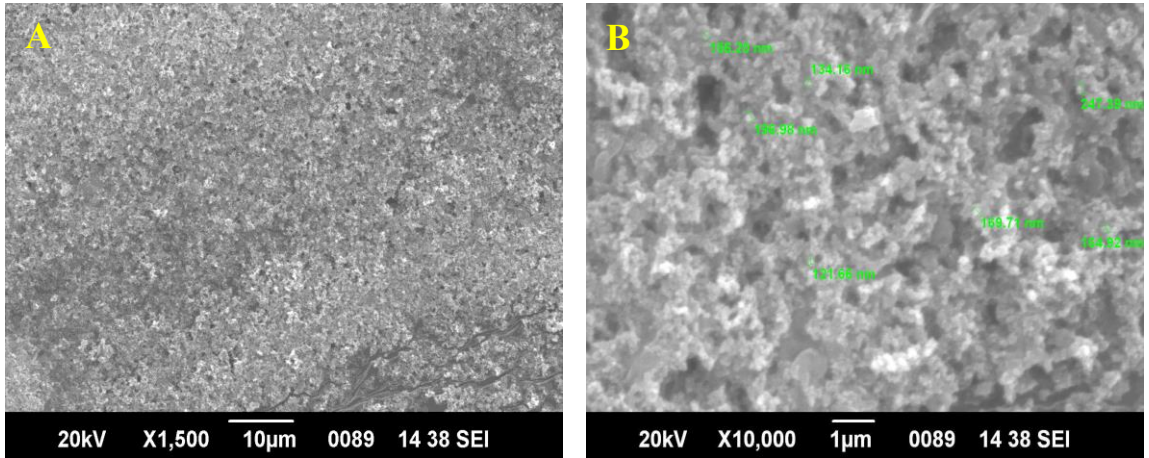


Plate 13A: SEM image of copper nanoparticles at magnification A) 1500x, B) 10000x

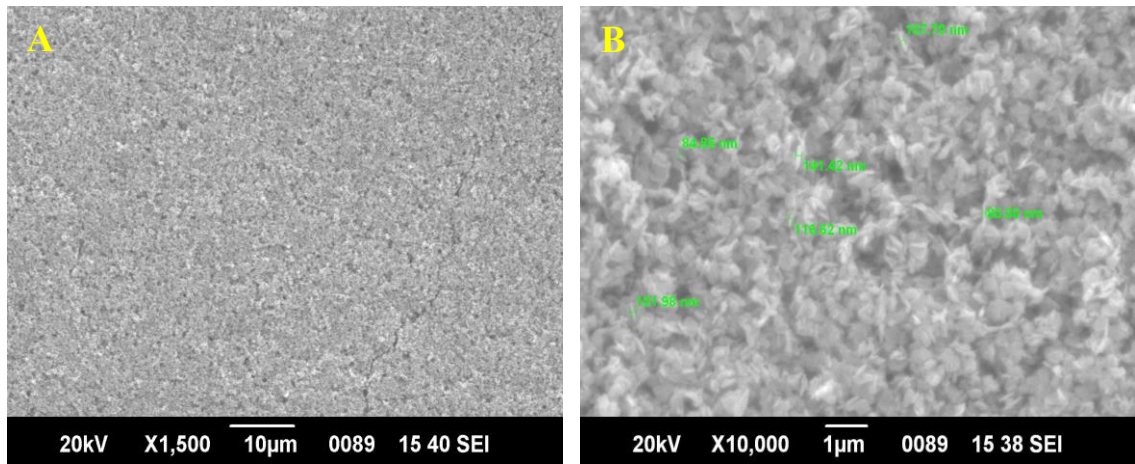


Plate 13B: SEM image of zinc nanoparticles at magnification A) 1500x, B) 10000x

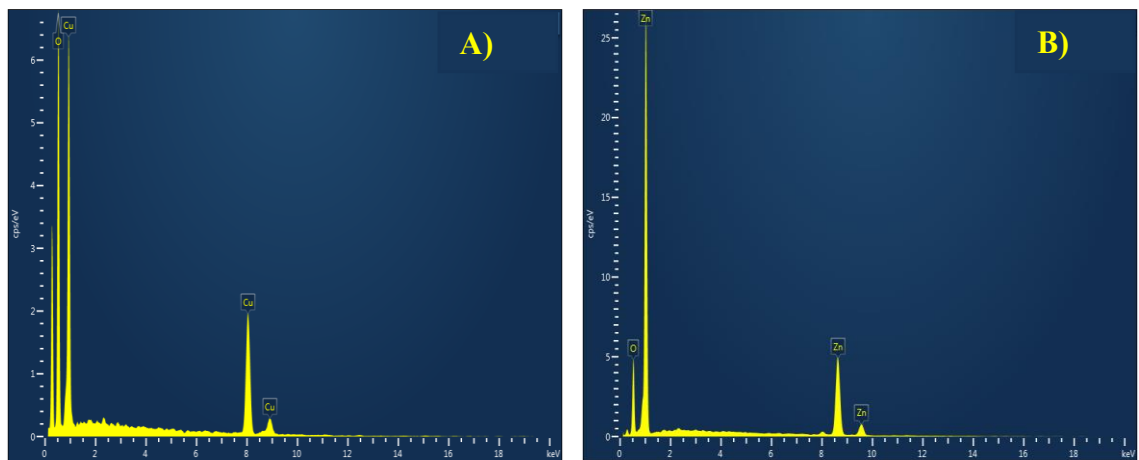
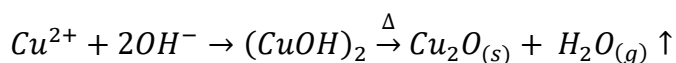


Fig 1: EDX spectrum of nanoparticles A) Copper, B) Zinc

Formation of cuprous oxide



The occurrence of OK_{α} , ZnL_{α} , ZnK_{α} , and ZnK_{β} in the zinc nanoparticles were confirmed with the peak value of EDX analysis at 0.5 keV, 1.01 keV, 8.64 keV, and 9.57 keV respectively based on (Fig. 2) were Hasanapour *et al.* (2012), Yedurkar *et al.* (2016), Mahalakshmi *et al.* (2019), Shankar and Rhim, (2019) and Yücel *et al.* (2020).

The elemental composition using EDX analysis of prepared zinc nanoparticle had 81.88% of copper and 18.12% of oxygen. Compared to EDX analysis reported by Mahalakshmi *et al.* (2019), where they found zinc 79.29% and oxygen 20.71% elemental compositions. While using biological method employed by Yücel *et al.* 2020 obtained 63.02% of zinc and 36.38% oxygen using zinc oxide. Almost the same concentration of elements were also reported by Yedurkar *et al.* (2016).

According to EDX analysis, the produced zinc nanoparticles did not contain any additional impurities. This could be attributed to calcination process, which leads to removal of any impurities present in the sample except zinc-oxide as it remains stable at high temperature (Baharudin *et al.*, 2018).

4.2.2. FTIR

The Fourier Transform Infrared Spectroscopy (FTIR) was carried out to determine the chemical composition and molecular structure of the nanoparticles. The FTIR was acquired from 400-4000 cm^{-1} wavelength at room temperature. The FTIR data was smoothed to remove noise and plotted using Origin lab software (Ver. 9.55).

4.2.2.1. FTIR-copper nanoparticle

The FTIR spectrum of copper nanoparticles is showed in the Figure 2. The peaks at 3442.92 cm^{-1} correspond to the -OH group. The peak value at 2921.63 cm^{-1} and 2851.72 cm^{-1} corresponds to either symmetric or asymmetric C-H group of alkyls. The peak at 1628.59 cm^{-1} corresponds to C=O stretching while the peak at 1379.82 cm^{-1} , 1157.08 cm^{-1} , 1081.57 cm^{-1} and 1017.25 cm^{-1} corresponds to C-O groups. The metal-oxygen stretching vibration mode is at peak value at 861.9 cm^{-1} (Cu_2O), 764.59 cm^{-1}

$^1(\text{Cu}_2\text{O})$, $624.32 \text{ cm}^{-1}(\text{Cu}_2\text{O})$, $526.47 \text{ cm}^{-1}(\text{CuO})$, and $434.67 \text{ cm}^{-1}(\text{CuO})$. The predicted results for the FTIR peak value for copper nanoparticle are based on the earlier studies of as shown below (Table. 7).

Table 7: Predicted result of FTIR spectrum for copper nanoparticle

Wavelength (cm^{-1})	Predicted results	Reference
3425.01	OH- Group bond	Padil and Cernik (2013)
2921.45	C-H of alkyl group	Salavati and Davar (2009)
2851.65	C-H of alkyl group	Salavati and Davar. (2009)
1628.51	C=O stretching	Karthik and Geetha (2011)
1380.05	C-O stretching	Mohammed (2020)
1157.7	C-O stretching	Mohammed (2020)
1082.07	C-O stretching	Sankar <i>et al.</i> (2014)
1018.19	C-O stretching	Sankar <i>et al.</i> (2014)
861.9	Cu_2O	Jadhav <i>et al.</i> (2011)
764.59	Cu_2O	Jadhav <i>et al.</i> (2011)
624.32	Cu_2O	Jadhav <i>et al.</i> (2011)
526.76	CuO	Padil and Cernik (2013)
434.67	CuO	Padil and Cernik (2013)

FT-IR is the mostly used for the greater understanding of elemental composition of small sized samples. Hydroxyl group (OH) stretching is shown by the wide peak value of $3200\text{-}3800 \text{ cm}^{-1}$ (Padil and Crenik, 2013). It was confirmed from other authors such as from Mohamed (2020) mentioned the peak value at 3337 cm^{-1} , Jadhav *et al.* (2011) at 3298 cm^{-1} , Kalyani *et al.* (2015) at 3478 cm^{-1} , Luna *et al.* (2015) at 3445.89 cm^{-1} , and Karthik and Geetha (2011) at 3570 cm^{-1} .

The C-H vibration corresponds to a peak ranging from $2800\text{-}3000 \text{ cm}^{-1}$ (Salavati and Davar, 2009). The similar findings were reported by Mohamed (2020), observed at the peak value of 2921.72 cm^{-1} , Salavati and Davar (2009) at 2921 cm^{-1} and 2865 cm^{-1} , Sankar *et al.*, (2014) at 2926 cm^{-1} , Luna *et al.* (2015) at 2922.21 cm^{-1} .

Karthik and Geetha (2011) at 1620 and 1429 cm^{-1} , Padil and Cernik (2013) at 1630 cm^{-1} , Kalyani *et al.* (2015) at 1623 cm^{-1} , Khatami *et al.* (2017) at 1634 cm^{-1} and,

Baquer *et al.* (2018) at 1652 cm^{-1} had reported the presence of C=O stretching in the mentioned region. Thus, it was verified that the C=O group was present at 1628.51 cm^{-1} inside our research.

The peak at 1380.05 cm^{-1} , 1157.7 cm^{-1} , 1082.07 cm^{-1} and, 1018.19 cm^{-1} corresponds to the C-O group was confirmed from Mohammed (2020) at 1398.69 cm^{-1} , 1163.39 cm^{-1} and 1097.44 cm^{-1} indicates C-O stretching vibration while peak at 1384 cm^{-1} describe C-O bond (Kalyani *et al.*, 2015). Similar to this peak value for C-O stretching was recorded by Karthik and Geetha (2011) at 1429 cm^{-1} , Padil and Cernik (2013) at 1074 cm^{-1} , Sankar *et al.* (2014) at 1087 cm^{-1} , Khatami *et al.* (2017) at 1067 cm^{-1} and, Renuga *et al.* (2020) at 1046 cm^{-1} .

The peak value of 400-600 cm^{-1} corresponds to CuO stretching (Padil and Cernik, 2013; Keabadile *et al.*, 2020). While 600-900 cm^{-1} corresponds to Cu_2O (Jadhav *et al.*, 2011; Luna *et al.*, 2015). To support this Saif *et al.* (2008) at 522 cm^{-1} , 580 cm^{-1} for CuO and 610 cm^{-1} for Cu_2O , Sankar *et al.* (2014) at 473 cm^{-1} for CuO, Kayani *et al.* (2015) recorded peak values at 473 cm^{-1} corresponding to CuO and 624 cm^{-1} to Cu_2O , Luna *et al.* (2015) at 533.33 cm^{-1} , 585.41 cm^{-1} for CuO, Baquer *et al.* (2018) ranging from 414-434 cm^{-1} for CuO, Sagadevan *et al.* (2019) CuO at 432 cm^{-1} , 611 cm^{-1} , and 886 cm^{-1} for Cu_2O , Samson *et al.* (2019) 911 cm^{-1} and 841 cm^{-1} indicate Cu_2O while Cu-O for 452 cm^{-1} , Renuga *et al.* (2020) 620 and 686 cm^{-1} CuO, Keabadile *et al.* (2020) at 444, 494, 579, 595 cm^{-1} . Thus, the above-mentioned citation, concluded the presence of CuO at peak value 526.76 and 434.67 cm^{-1} and Cu_2O at 861.9, 764.59 and 624.32 cm^{-1} .

4.2.2.2. FTIR-zinc nanoparticles

The FTIR spectrum of zinc nanoparticles is presented in the Figure 3. The Peak values at 3414.83 cm^{-1} and 2920.66 cm^{-1} are related to the stretching vibration of the strong and weak hydroxyl (-OH) group, possibly due to containing zinc oxide or zinc hydroxide or other hydroxyl groups. Peak values show the existence of the C-H group in alkane at 2850.76 cm^{-1} . Peak readings at 1136.83 cm^{-1} , in either vibration or plane bend of primary or secondary alcohol. While, the peak at 995.09 cm^{-1} and 619.52 cm^{-1} corresponds to metal-oxygen i.e., zinc hydroxide (Zn-OH) and zinc-oxide (ZnO) stretching vibration mode. The predicted results for the FTIR peak value for zinc nanoparticle are based on the earlier studies of as shown below (Table. 8).

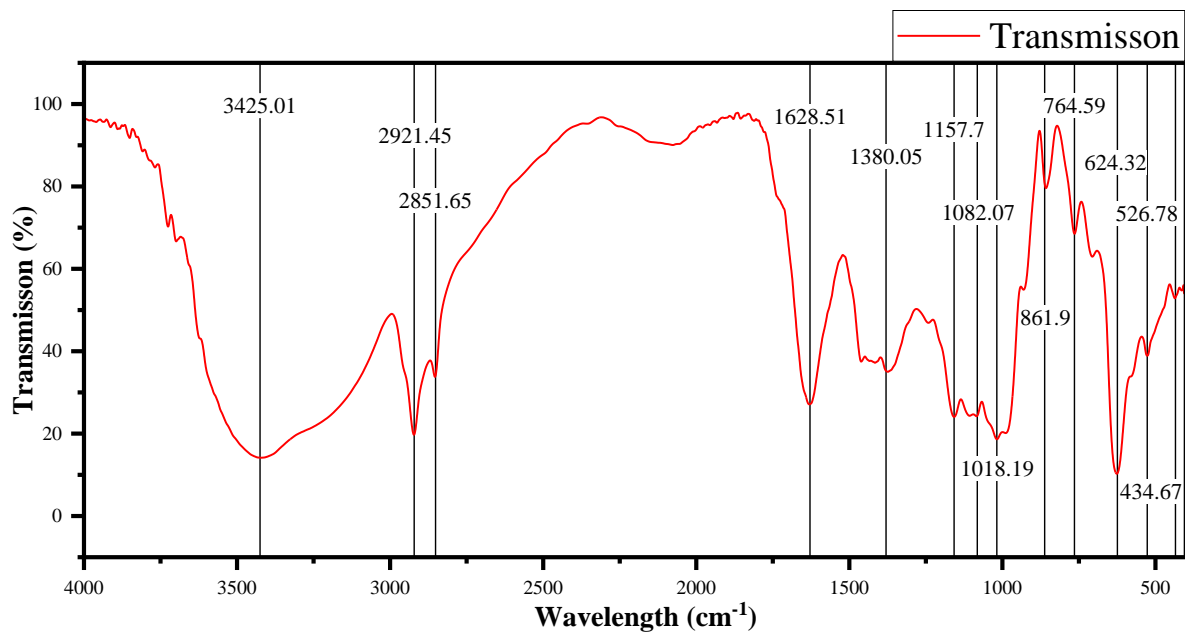


Fig 2: FTIR spectrum of copper nanoparticles

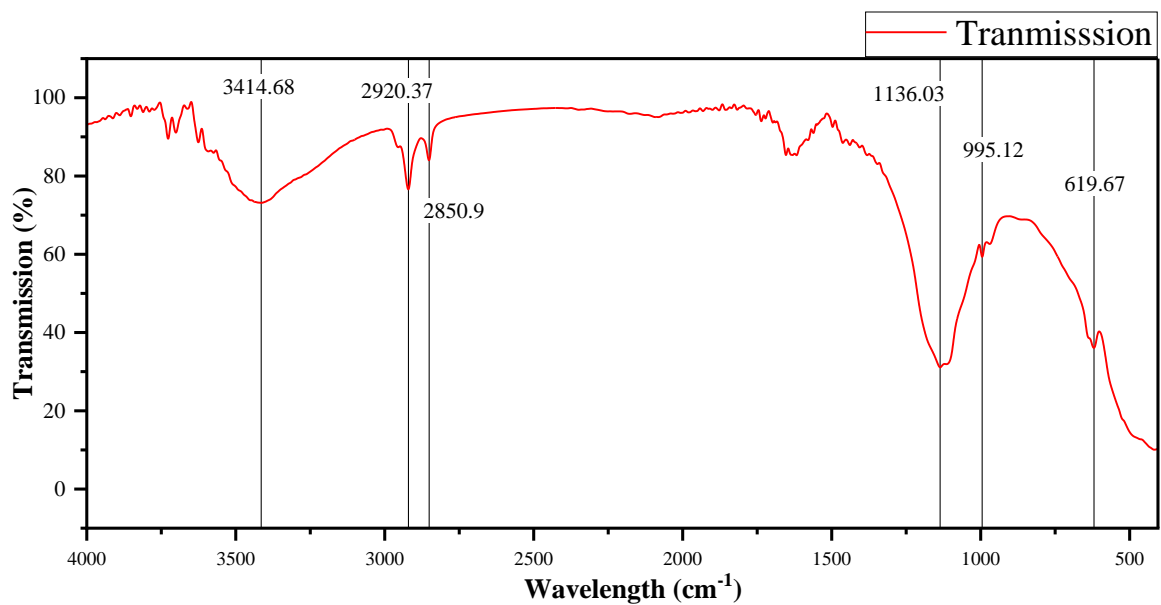


Fig 3: FTIR spectrum of zinc nanoparticles

Table 8: Predicted results of FTIR spectrum for zinc nanoparticle

Wavelength (cm ⁻¹)	Predicted results	Reference
3414.83	OH- Group bond	Srivastava <i>et al.</i> (2013).
2920.66	OH- Group weak bond	Jayarambabu <i>et al.</i> (2014)
2850.76	C-H group in alkane	Gnanasangeetha and SaralaThambavani (2015)
1136.83	Primary or secondary alcohol	Mehralizadeh and Gharbani (2018); Mahalakshmi <i>et al.</i> (2019)
995.09	Zn-OH	Mahalakshmi <i>et al.</i> (2019)
619.52	Zn-O	Jun <i>et al.</i> (1998)

A broad peak value between 3000 and 3500 cm⁻¹, which is indicative of a strong hydroxyl group, was reported by Srivastava *et al.* (2013). Wahab *et al.* (2008) at 3455 cm⁻¹; Azizi *et al.* (2015) at 3349 cm⁻¹; and Chaudhary *et al.* (2019) at 3369.34 cm⁻¹ and 3358 cm⁻¹ have all referenced this study that is comparable with peak 3414.83 cm⁻¹ mentioned in our study indicating the presence of hydroxyl group or binding of these group on Zinc oxide or Zinc hydroxide. A weak hydroxyl group might cause the peak in 2900–3000 cm⁻¹ range (Jayarambabu *et al.*, 2014). To bolster this, peak value at 2925 cm⁻¹ was reported by Azizi *et al.* (2015) as weak -OH stretching. Some have stated that absorption of -OH group on zinc oxide or zinc hydroxide when it is water is the cause for the peak at 2920.66 cm⁻¹ (Kulkarni and Shirsat, 2015; Rajendran and Sengodan, 2017).

C-H stretching is indicated by a peak value between 2800 and 3000 cm⁻¹ (Gnanasangeetha and SaralaThambavani, 2015). Mishra *et al.* (2013) at 2850 cm⁻¹ and Janakiraman and Johnson (2015) at 2854 cm⁻¹ have reported similar results indicating the presence of C-H group. According to studies by Mehralizadeh and Gharbani (2018) at 1190 cm⁻¹, PP *et al.* (2020) at 1117 cm⁻¹, and Jayarambabu *et al.* (2014) at 1154 cm⁻¹, the peak value of 1136.83 cm⁻¹ correlates to bending or stretching vibration of primary or secondary alcohol groups.

Zinc-oxide is assigned a peak value range of 400–600 cm⁻¹ (Jun *et al.*, 1998; Sangeetha *et al.*, 2011). According to Poovizhi and Krishnaveni (2015) analysis, the zinc oxide stretching mode is responsible for 610.08 to 692.44 cm⁻¹. The results of

Mahalakshmi *et al.* (2019) at 620 cm^{-1} and 950 cm^{-1} corresponding to zinc oxide and zinc hydroxide, respectively. This supports the existence of zinc oxide (619.52 cm^{-1}) and zinc hydroxide (995.09 cm^{-1}) in the present study. Taufiq *et al.* (2018) found similar results at 690 cm^{-1} and about 900 cm^{-1} , whereas Kołodziejczak *et al.* (2012) found similar results at 650 cm^{-1} and 919 cm^{-1} , respectively.

4.2.3. XRD

The crystal structure was verified using X-ray diffraction analysis (XRD). The sample was examined at room temperature at 2θ range of 10° - 80° with a wavelength (1.5406Å). The XRD data was smoothened to remove noise and plotted using Origin Lab software (Ver. 9.55).

4.2.3.1. XRD-copper nanoparticle

The XRD spectrum of copper nanoparticles is presented in the Figure 5. The peak observed at 2θ values of 29.6° , 36.4° , 42.2° , 43.3° , 50.4° , 61.3° , 73.5° , and 77.4° corresponds to 110, 111, 200, 111, 200, 220, 311, and 222 crystalline structure lattice planes of copper nanoparticles respectively. Peak value 29.5° , 36.4° , 42.2° , 61.3° , and 77.4° are related to Cu_2O while, 43.3° , 50.4° , and 73.5° are related to CuO . For, Cu_2O , the major peak was recorded at 36.4° while the minor peak was at 77.3° . For CuO , the major peak was recorded at 43.3° while the minor peak was at 73.5° .

Table 9: Predicted result of XRD analysis for copper nanoparticles

Peak value (2θ)	Predicted CuO and Cu ₂ O Lattice phase
29.572°	110-Cu ₂ O
36.405°	111-Cu ₂ O
42.288°	200-Cu ₂ O
43.297°	111-CuO
50.415°	200-CuO
61.351°	220-Cu ₂ O
73.518°	311-CuO
77.356°	222-Cu ₂ O

The Table 9. indicates the predicted copper oxide or cuprous lattice phase. According to JCPDS (Joint Committee on Powder Diffraction Standards) card no. 04-

0836 reported by Khatami *et al.* (2017), the peak value at 29°, 36°, 42°, 43°, 50°, and 61° assigned to 110, 111, 200, 111, 200, and 220 planes of copper nanoparticles. Peak values at 29°, 36°, 42°, and 61° are related to Cu₂O group (JCPDS card no. 05-0667). On the other hand, peak value at 43°, and 50° belongs to the CuO group (JCPDS card no. 04-0836). While, Kooti and Matouri (2010), recorded peak value at 73.6° and 77.6° corresponding to CuO and Cu₂O groups, respectively of 311 and 222 crystal planes, respectively. With peak at 29.6° (110), 36.5°(111), 42.4°(200), 61.5°(220) corresponds to Cu₂O.

Similar reports have told by Salavati and Davar (2009) with JCPDS card no. 08-1326, Mohamed *et al.* (2014) with JCPDS card no. 80-1916, Kayani *et al.* (2015) with JCPDS card no. 48-1548, Khan *et al.* (2016) with JCPDS card no. 05-667. Sasmal *et al.* (2016) with JCPDS card no. 04-0836, Baqer *et al.* (2018) with JCPDS card no. 80-1917, and Mali *et al.* (2019) with JCPDS card no. 05-0667.

From the above-mentioned cases, it is confirmed that CuO and Cu₂O had a monoclinic phase. It also confirmed that the synthesized nanopowder was almost free of impurities as it does not contain any characteristics of XRD peak other the copper oxide or cuprous oxide peaks, except few minor peaks may be due to noise/other impurities (Mohamed *et al.*, 2020).

Thus, it demonstrates that the copper nanoparticles generated using the aforementioned method contain copper oxide (CuO) and cuprous oxide (Cu₂O).

4.2.3.2. XRD-zinc nanoparticles

The XRD spectra of Zinc nanoparticles is depicted in the Figure 6. The peak observed at 2 θ values of 31.8°, 34.4°, 36.2°, 47.5°, 56.6°, 62.9°, 66.4°, 68.0°, 69.1°, 72.6°, and 77.0° corresponds to 100, 002, 101, 102, 110, 103, 200, 112, 201, 004, and 022 crystalline structure lattice planes of Zinc-oxide respectively. The major peak was recorded at 36.2° while the minor peak was at 77.0°.

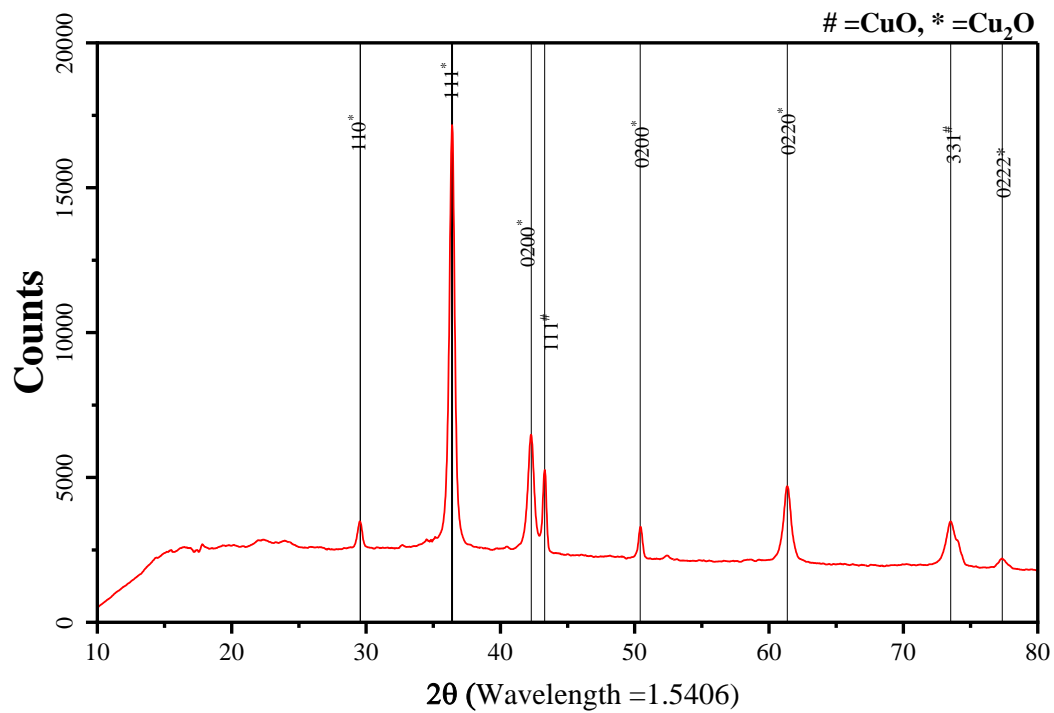


Fig 4: XRD curve of copper nanoparticles

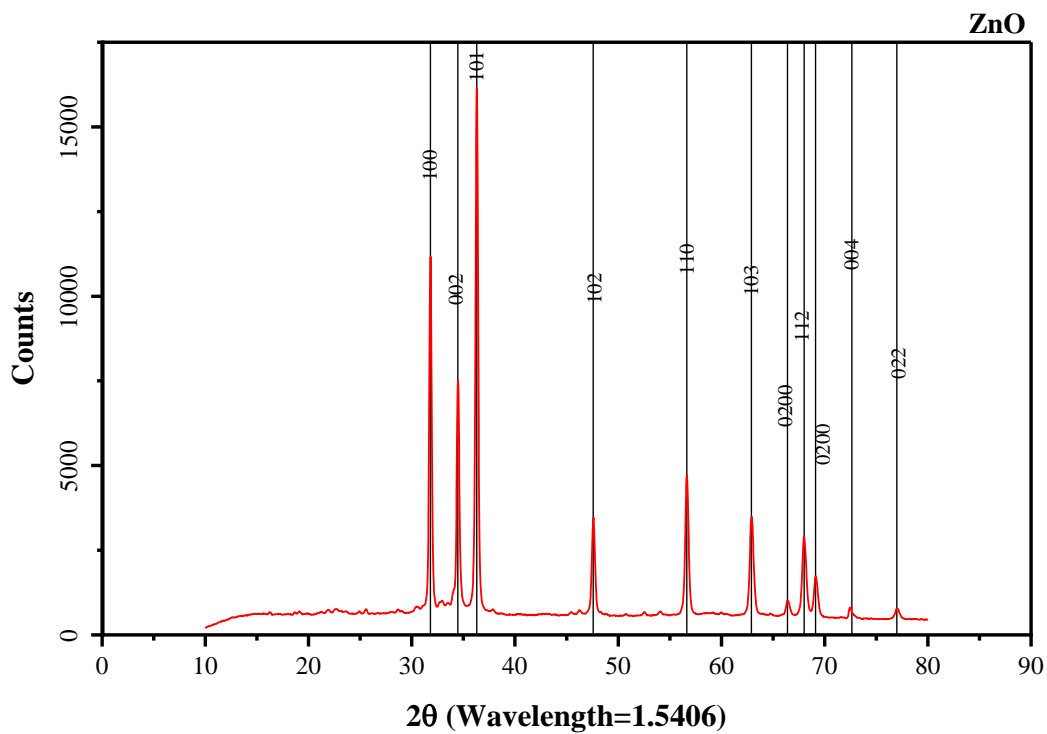


Fig 5: XRD curve of zinc nanoparticles

Table 10: Predicted result of XRD analysis for zinc nanoparticles

Peak value (2 θ)	Predicted Zinc Lattice phase
31.8°	100
34.4°	002
36.2°	101
47.5°	102
56.6°	110
62.9°	103
66.4°	200
68.0°	112
69.1°	201
72.6°	004
77.0°	022

The predicted Zinc lattice phase is indicated in the Table 10. The XRD peak value was confirmed by Taufiq *et al.* (2018) with JCPDS(Joint Committee on Powder Diffraction Standards) card no. 36-1451 which recorded peak at 31.7° (100), 34.4° (002), 36.2° (101), 47.5° (102), 56.6° (110), 62.8° (103), 66.3° (200), 67.9° (112), 69.1° (201), and 72.5° (004), and 76.9° (022).

Similar results were found in Wahab *et al.* (2008), Hasanpour *et al.* (2012), Kołodziejczak *et al.* (2012), Kumar *et al.* (2013), Jayarambabu (2014), Yedurkar *et al.* (2016), Rajendra *et al.* (2017), Mehralizadeh and Gharbani (2018), Chaudary *et al.* (2019), Yucel *et al.* (2020), Hamouda *et al.* (2023) who verified with JCPDS no. 36-1451 and Top and Cetinkaya (2015) with PDF card no 76-1778.

The zinc crystalline possesses a wurtzite hexagonal structure, was validated from above mentioned reports. It also confirmed that the synthesized nanopowder was almost free of impurities as it does not contain any characteristics of XRD peak other the zinc oxide peaks, except few minor peaks may be due to noise (Hamouda *et al.*, 2023).

Thus, zinc nanoparticles prepared using the aforementioned method have pure Zinc-oxide (ZnO) phases.

4.3. Distribution of nanoparticle in treated wood samples

4.3.1. SEM-EDX Mapping

Figure showing the SEM- EDX Elemental mapping of the internal image of coconut wood pressure treated with copper and zinc nanoparticles and the surface image of coconut wood coated with zinc and copper nanoparticles is depicted in the Figure (6-9) showing an area of 50 μ m.

The parrot green shade in the wood indicates the presence of copper in the pressure treated coconut wood (Figure 6). Its corresponding EDX Spectra are shown in Figure 6B indicating the abundance of elements. From EDX Spectra it shows an abundance of copper after carbon and oxygen elements. Similarly in the nano-coated wood also the presence of parrot green shade in the wood represents copper in the surface of wood, and its corresponding EDX Spectra are shown in Figure 7A & 7B, indicating the abundance of elements. EDX Spectra shows an abundance of copper after carbon and oxygen elements.

Zinc is present in the pressure-treated coconut wood, as evidenced by the parrot green hue of the wood (Figure 8A). The corresponding EDX Spectra for it are displayed in Figure 8B, along with the elemental abundance. Zinc is abundant after carbon and oxygen elements, according to the EDX spectrum. The zinc present on the surface of the wood in the nano-coated wood is also represented by the parrot green shade, and Figure 9A & 9B displays the corresponding EDX spectra, which demonstrate the elemental abundance. Following carbon and oxygen elements in the EDX spectrum is an abundance of zinc.

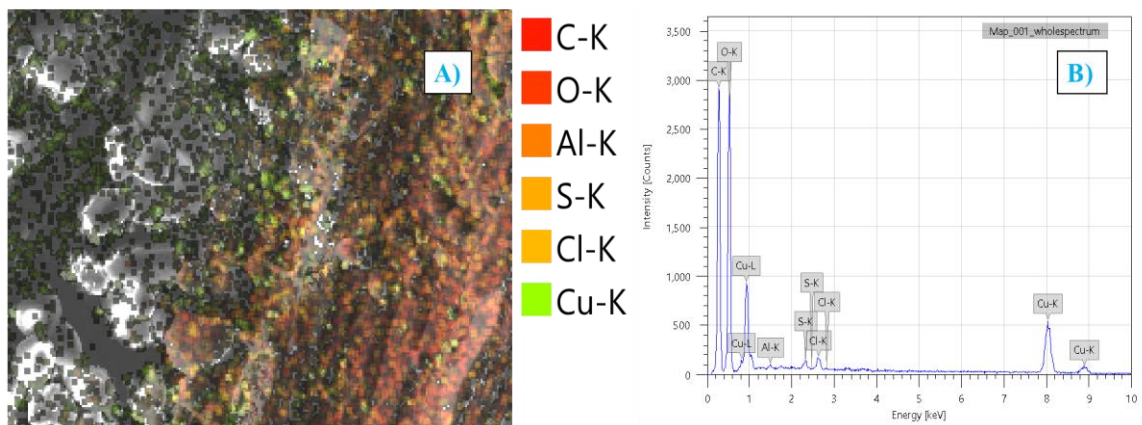


Fig 6: A) Internal SEM image of coconut wood pressure treated with copper nanoparticles B) Corresponding EDX spectra

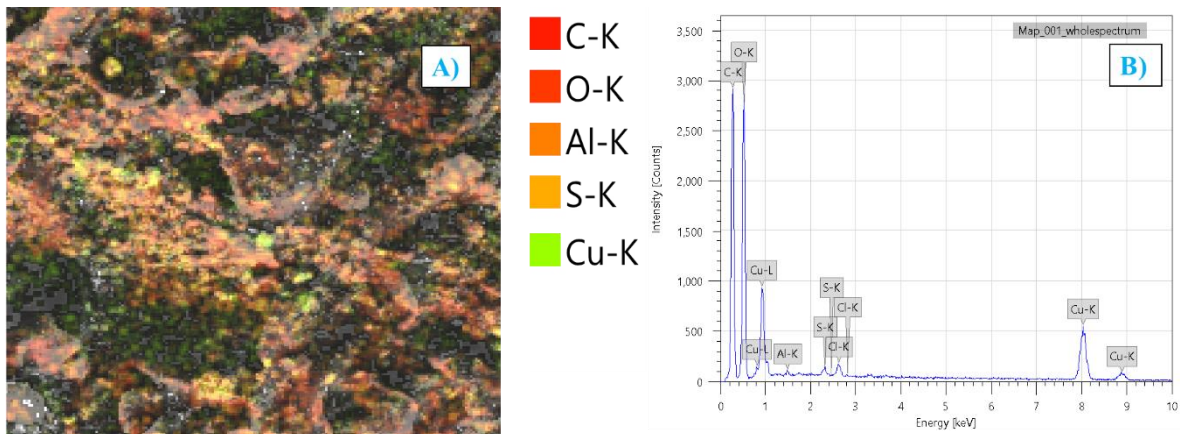


Fig 7: A) External SEM image of coconut wood pressure treated with copper nanoparticles
 B) Corresponding EDX spectra

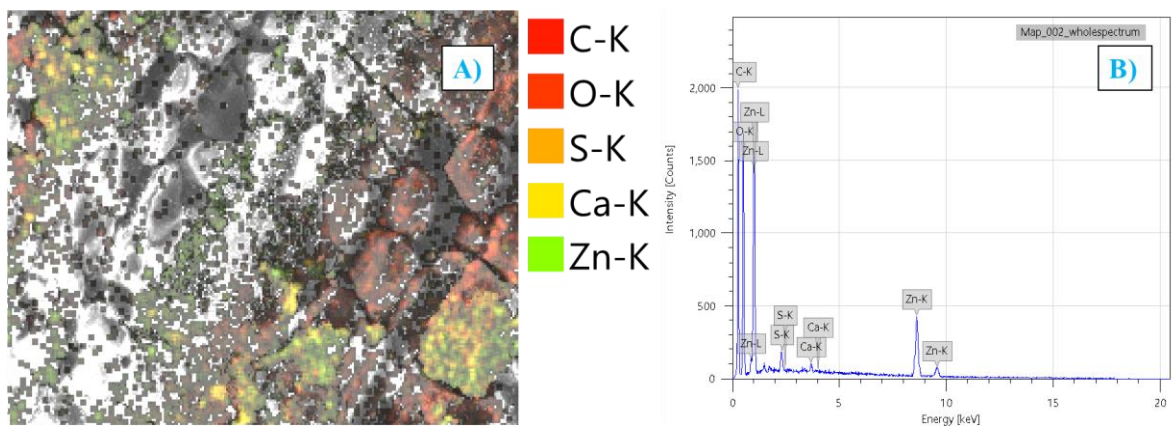


Fig 8: A) Internal SEM image of coconut wood pressure treated with zinc nanoparticles
 B) Corresponding EDX spectra

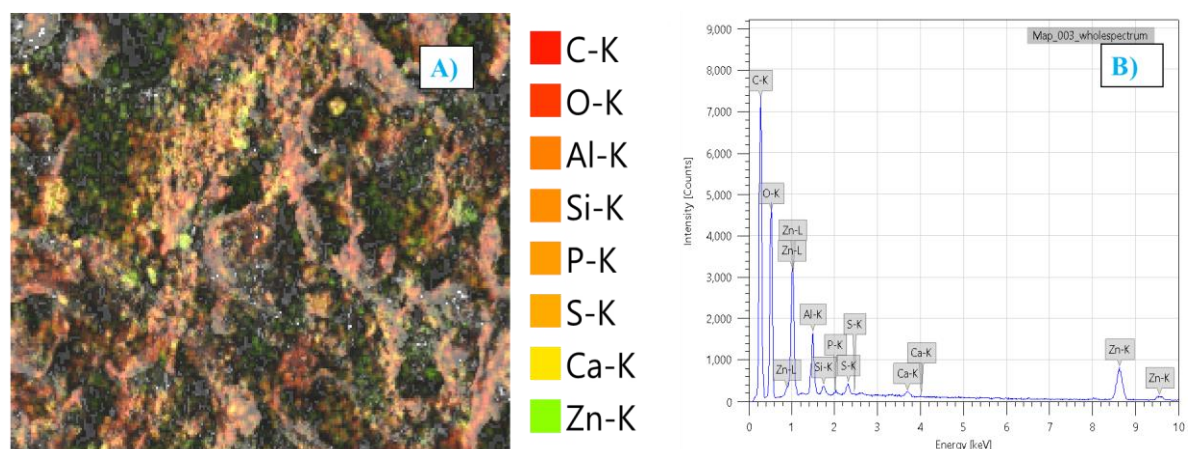


Fig 9: A) External surface SEM image of coconut wood coated treated with zinc nanoparticles
 B) Corresponding EDX spectra

4.4. Experiments

4.4.1. Chemical Retention Test

Chemical retention was carried out to investigate the effective embedding of nanoparticles into coconut wood. The chemical retention of coconut wood blocks treated to different density with different nanoparticle and its concentration by pressure and coating methods used in the study is shown in the Table 11. These are the average values obtained after digestion of the five wood samples from each treatments.

The chemical retention analysis of coconut wood treated with nanoparticles using various application methods has shown significant differences among various densities and nanoparticle concentrations used in the process but no significant difference was observed among the nanoparticles (Table 11).

In case of pressure method of application, among density, medium density noticed a maximum retention of 4.4 kgm^{-3} compared to high density (3.87 kgm^{-3}). Among the nanoparticles, zinc and copper treated to medium density showed high retention (4.54 kgm^{-3} and 4.26 kgm^{-3} , respectively), while copper and zinc high density recorded low retention of 3.67 kgm^{-3} and 4.08 kgm^{-3} respectively, showing no significant difference among nanoparticles used. A significant difference was recorded among various concentrations, with 5% zinc treated to medium density showing maximum retention of 6.49 kgm^{-3} which was slightly comparable with 5% copper treated to medium density (5.69 kgm^{-3}). While, 1% copper and zinc treated to high density showed minimum retention of 2.37 kgm^{-3} and 2.2 kgm^{-3} .

While, coating as a method of application, among density medium density noticed the maximum retention (4.32 kgm^{-3}) compared with high density of 3.78 kgm^{-3} . A significant difference was not observed between the nanoparticles treatments applied to coconut wood. The zinc treated to medium density exhibited high retention of 4.43 kgm^{-3} and 4.21 kgm^{-3} , respectively, whereas the copper treated to high density showed poor retention of 3.59 kgm^{-3} and 3.97 kgm^{-3} , respectively. There was a notable variation observed between the different concentrations; Highest retention of 6.34 kgm^{-3} was observed for 5% copper treated to medium density, and 6.16 kgm^{-3} for 5% zinc treated to

Table 11: Chemical retention value (kgm^{-3}) of various density of coconut wood block treated with nanoparticle concentrations by various methods

<u>Pressure Treatment</u>					<u>Coating Treatment</u>				
Nanoparticles and its concentrations	Density				Nanoparticles and its concentrations	Density			
	Medium		High			Medium		High	
	Copper	Zinc	Copper	Zinc		Copper	Zinc	Copper	Zinc
1%	2.54	2.59	2.37	2.2	1%	2.46	2.65	2.09	1.96
2.50%	4.54	4.55	4.28	4.01	2.50%	4.0	4.3	3.04	4.31
5%	5.69	6.49	4.62	5.76	5%	6.34	5.64	6.16	5.64
Average of nanoparticle	4.26	4.54	3.67	4.08	Average of nanoparticle	4.21	4.43	3.59	3.97
Average of density	4.4		3.87		Average of density	4.32		3.78	
Average of density-Significant at 0.05%					Average of density-Significant at 0.05%				
Average of nanoparticle-Non-Significant					Average of nanoparticle-Non-Significant				
Average of concentration-Significant at 0.001%					Average of concentration-Significant at 0.001%				

the same medium density. Conversely, the minimum retention was shown by 1% zinc and copper treated to high density (1.96 kgm⁻³ and 2.09 kgm⁻³, respectively).

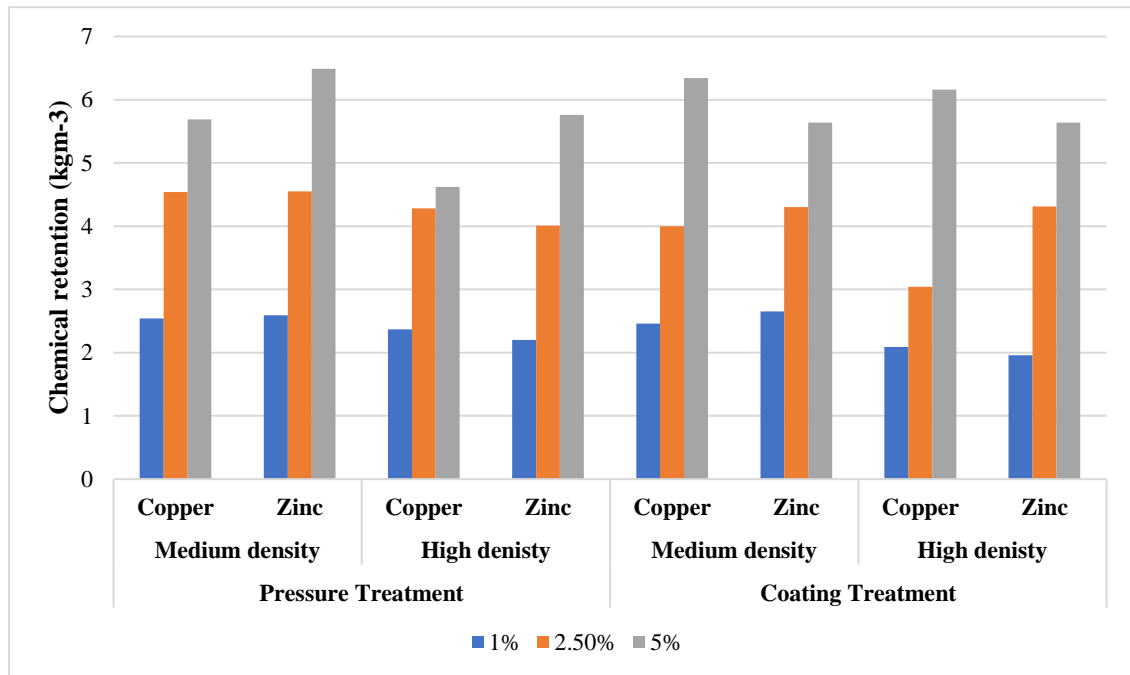


Fig 10. Chemical retention value (kgm⁻³) of various density of coconut wood block treated with nanoparticle concentrations by various methods

Chemical retention refers to the ability of chemical to retain in wood following treatment. In order to enhance its durability, resistant, and protection against wood deteriorating and degradation factors or both abiotic and biotic factors of wood degradation certain level of chemical should be retained. Factors influencing level of chemical retention in wood are the anatomical structure of wood, pre-treatment conditions like seasoning, cleaning or incising and method of preservative application (Kollman and Cote, 1968).

In our study, between density, the average chemical retention of coconut wood using pressure treated to medium density (4.4 kgm⁻³) was more than the high density (3.87 kgm⁻³). Similar result is seen when nanoparticle used as coating, were medium density had chemical retention of 4.32 kgm⁻³ while high density had 3.78 kgm⁻³ (Figure 10). To support this, Németh *et al.* (2013) reported the chemical retention of nano-zinc (0.22%) pressure treated to beech wood and pine wood had 0.177 kgm⁻³ and 0.284 kgm⁻³ respectively. Pine wood being lower denser than beech wood showed more chemical retention than latter. The result was line with the study reported by Bak and Németh

(2018). The reason for the difference among the density is due to difference in the anatomical structure between medium and high density. Wood composed of juvenile with low density and mature wood with high density. Medium density has more juvenile portion i.e., low density than high density. As a result of these, more absorption of nanoparticles happens (Thompson, 1991; Barnett and Jeronimidis, 2003). And medium density contains more pores region than high density (Kollman and Cote, 1968). Also, coconut wood was cleaned with acetone to remove any blockage present and increase the pore space for preservative penetration (section 3.2.1.). As a result of increase in pores space which leads to increase in chemical retention.

Anusha *et al.* (2023) reported that due to increase in solute concentration (i.e., number of nanoparticles in the solution) leads to an increase in the penetration of this solute into the wood (Tamblyn, 1985). As a result of this concentration of the treating solution increases the chemical retention in the wood increases. Thus, the study showed a significant variation in chemical retention of both nanoparticles used at different concentration when treated using pressure and coating. As the concentration of the nanoparticle increases from 1% to 5% the retention of both zinc (2.59 to 6.49 kgm⁻³ for medium density and 2.2 to 5.76 kgm⁻³ for high density) and copper (2.54 to 5.69 kgm⁻³ for medium density and 2.37 to 4.62 kgm⁻³ for high density) increases (Figure 10). Similar results have been seen in the study reported by Pařil *et al.* (2017) and Bak and Nemeth (2018). Bak and Nemeth (2018) reported that when nano zinc treated to Beech wood at concentration from 1% to 5% had a chemical retention from 4.21-22.00 kgm⁻³. Similar result was reported in this paper when treated with nano-copper.

Among nanoparticles used not much difference in the retention levels of zinc and copper in the wood. Kartal (2009); Bak and Nemeth (2018) and Shiny and Sundararaj (2021) all reported same findings. The reason may be due to the uniform penetration of nanoparticles into the wood. But the retention of zinc is more than copper. The reason may be due to smaller size of zinc nanoparticles when compare to copper nanoparticles. It can be confirm from SEM-EDX mapping of nanoparticles distribution in the internal structure of wood shows low EDX spectrum value of copper than zinc (section 4.3.1).

4.4.2. Chemical Leaching Test

The percentage of leaching was determined by comparing the average leach rate of five samples for each treatment to the portion of treated, un-leached samples that were tested for zinc and copper based on initial chemical retention (Table 11).

The detailed leaching value in ppm with respect to 0.25, 1, 2, 4, 6, 8, 10, 12, and 14 days for all treatment applied using pressure and coating is shown in the Table 12. Significant difference was recorded in leaching value with respect to time among density, type of nanoparticles and their concentrations irrespective of method of application.

As the time increases, leaching value of medium density was more than high density this, result was similar with coating method also. Between types of nanoparticles, copper treated sample showed more leaching than zinc treated with both density and method of treatment. Among all treatment, leaching increases from 0.25 days to 1day and recorded the maximum leaching at 1day of leaching. After 1day of leaching, there is a gradual reduction in the leaching value with respect to increase duration of days (Table 12 and 13).

Similar results was recorded in studies reported by Clausen *et al.* (2009), Clausen *et al.* (2010), Clausen *et al.* (2011), Mantanis *et al.* (2014), Temiz *et al.* (2014) and Bak and Németh (2018) but had higher values than reported in our study.

As pressure as a method of treatment, for medium density, maximum leaching was recorded in at 5% copper treated to medium density with 65.28 ppm. Contrastingly, zinc 1% treated to medium density showed low leaching value (2.49 ppm). For high density, the maximum leaching was recorded at 5% copper treated to high density with 49.5 ppm while 1% zinc showed low leaching of 1.79 ppm (Figure 11 and 12).

Similarly, when using coating as a method of treatment, for medium density, maximum leaching was recorded in at 5% copper treated to medium density with 90.31 ppm. Contrastingly, zinc 1% treated to medium density showed low leaching value (3.20 ppm). For high density, maximum leaching was recorded at 5% copper treated to high density with 78.98 ppm while 1% zinc showed low leaching of 2.56 ppm (Figure 13 and 14).

Table 12: Chemical leaching value (ppm) of various density coconut wood pressure treated with different nanoparticle concentration at various time intervals

Treatments	Days									Average leachates (ppm)
	0.25	1	2	4	6	8	10	12	14	
Zinc 1% medium density	0.58	0.65	0.54	0.26	0.21	0.10	0.07	0.04	0.03	2.49
Zinc 2.5% medium density	1.46	1.81	0.76	0.32	0.30	0.13	0.09	0.08	0.05	5.01
Zinc 5% medium density	1.92	2.98	1.16	0.54	0.40	0.21	0.18	0.08	0.07	7.55
Zinc 1% high density	0.41	0.46	0.34	0.22	0.16	0.10	0.03	0.07	0.01	1.79
Zinc 2.5% high density	1.07	1.45	0.78	0.28	0.26	0.20	0.13	0.05	0.02	4.24
Zinc 5% high density	1.48	2.51	1.13	0.49	0.29	0.26	0.17	0.06	0.04	6.43
Copper 1% medium density	5.89	4.84	4.71	3.59	2.49	1.19	0.24	0.14	0.05	23.14
Copper 2.5% medium density	10.96	10.86	9.47	6.09	3.64	0.78	0.58	0.21	0.08	42.67
Copper 5% medium density	13.26	18.99	11.25	8.93	5.46	4.23	1.58	1.27	0.31	65.28
Copper 1% high density	4.88	5.26	4.07	2.81	1.08	0.34	0.30	0.18	0.08	19.01
Copper 2.5% high density	7.79	10.76	6.15	4.49	2.14	1.61	0.48	0.36	0.17	33.95
Copper 5% high density	10.81	15.80	9.07	5.78	3.27	2.20	1.32	0.93	0.33	49.50

Table 13: Chemical leaching value (ppm) of various density coconut wood coated treated with different nanoparticle concentration at various time intervals

Treatments	Day									Average leachates (ppm)
	0.25	1	2	4	6	8	10	12	14	
Coating zinc 1% medium density	0.59	1.31	0.32	0.26	0.20	0.18	0.15	0.13	0.08	3.20
Coating zinc 2.5% medium density	1.12	2.31	0.54	0.67	0.32	0.19	0.13	0.05	0.04	5.37
Coating zinc 5% medium density	2.46	3.11	1.28	0.92	0.56	0.36	0.13	0.10	0.08	9.00
Coating zinc 1% high density	0.55	1.03	0.38	0.18	0.14	0.11	0.07	0.05	0.06	2.56
Coating zinc 2.5% high density	1.60	2.18	0.67	0.41	0.25	0.17	0.07	0.05	0.02	5.42
Coating zinc 5% high density	2.39	2.92	1.12	0.55	0.48	0.26	0.16	0.11	0.07	8.06
Coating copper 1% medium density	4.50	7.52	5.38	4.00	2.98	1.29	0.96	0.64	0.12	27.40
Coating copper 2.5% medium density	11.31	12.84	7.20	6.45	3.84	3.31	2.44	1.73	0.46	49.57
Coating copper 5% medium density	24.83	26.27	12.00	9.50	5.77	4.45	3.94	2.67	0.88	90.31
Coating copper 1% high density	3.97	5.76	3.67	3.56	2.38	1.18	0.98	0.52	0.06	22.08
Coating copper 2.5% high density	10.63	11.14	4.89	3.69	2.79	2.23	1.96	0.91	0.56	38.78
Coating copper 5% high density	22.90	24.82	10.35	6.11	4.58	3.98	2.93	2.46	0.85	78.98

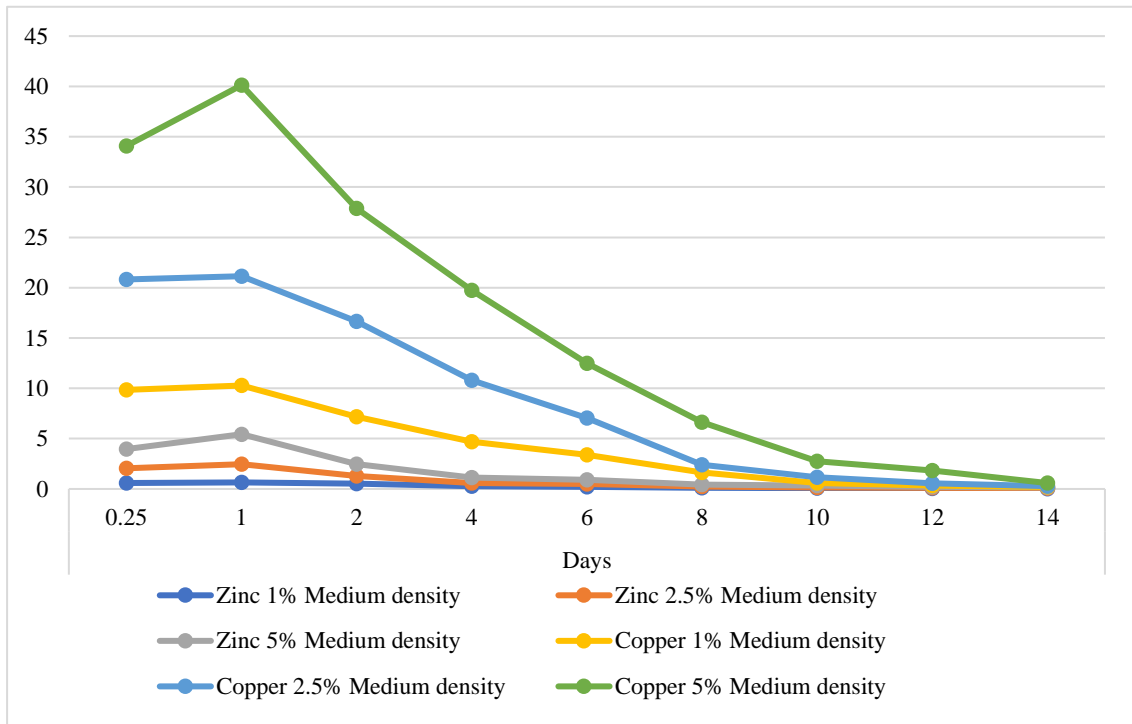


Fig 11: Chemical leaching of pressure treated medium density coconut wood at different time intervals

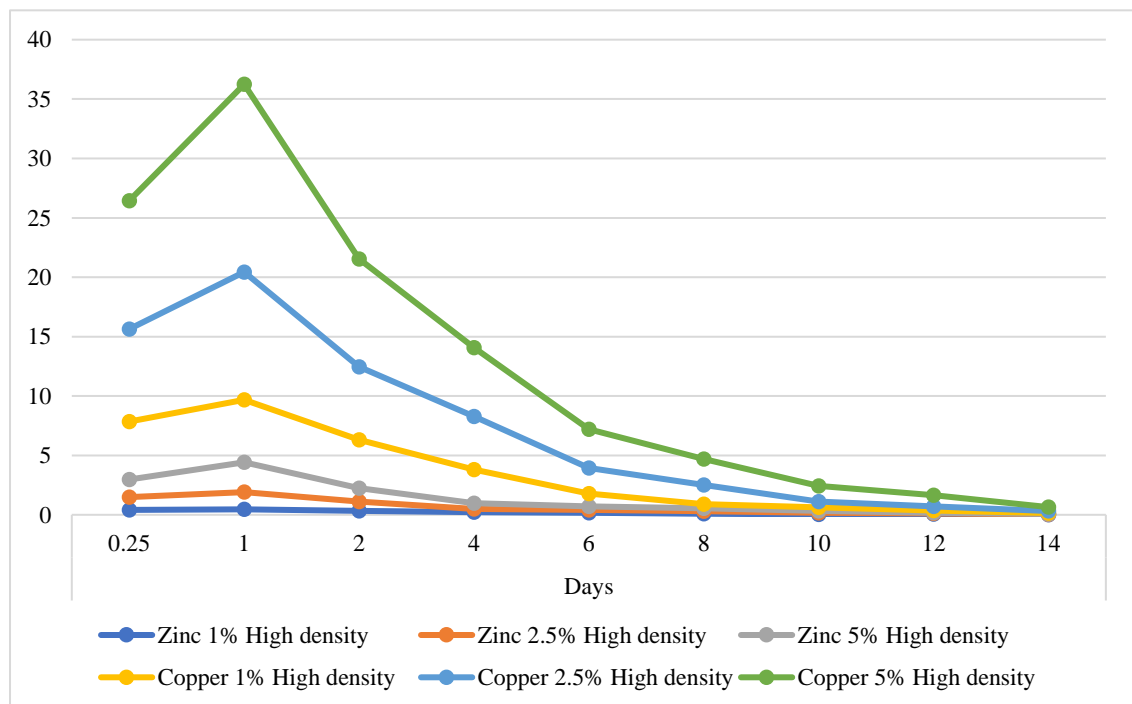


Fig 12: Chemical leaching of pressure treated high density coconut wood at different time intervals

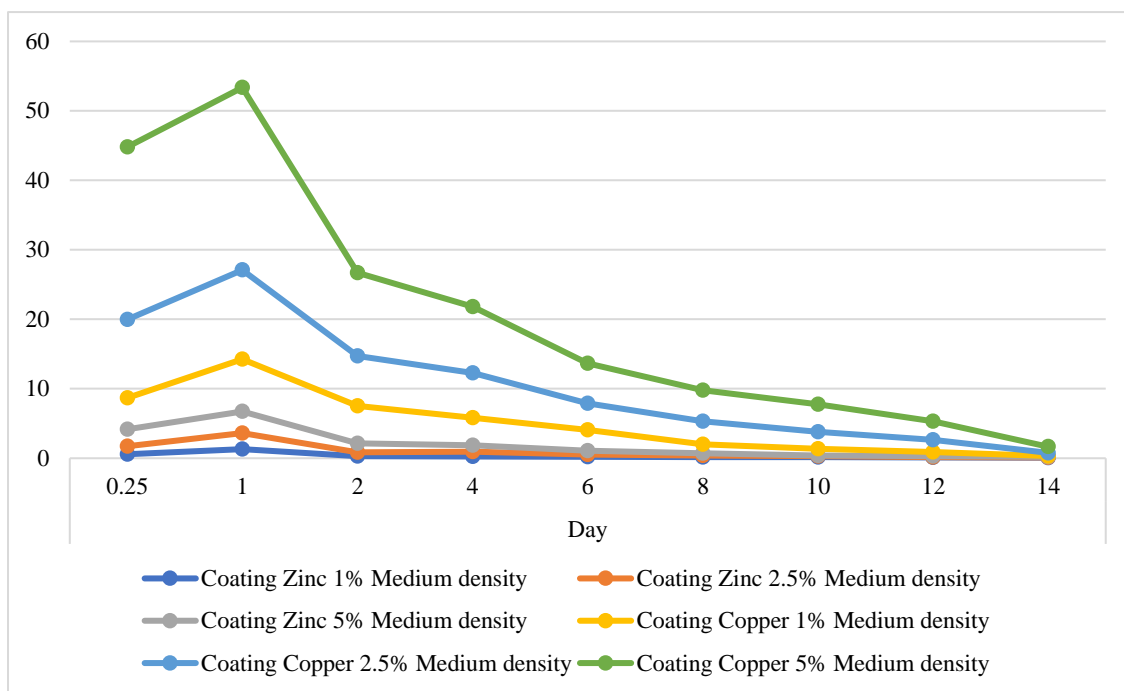


Fig 13: Chemical leaching of coating treated medium density coconut wood at different time intervals

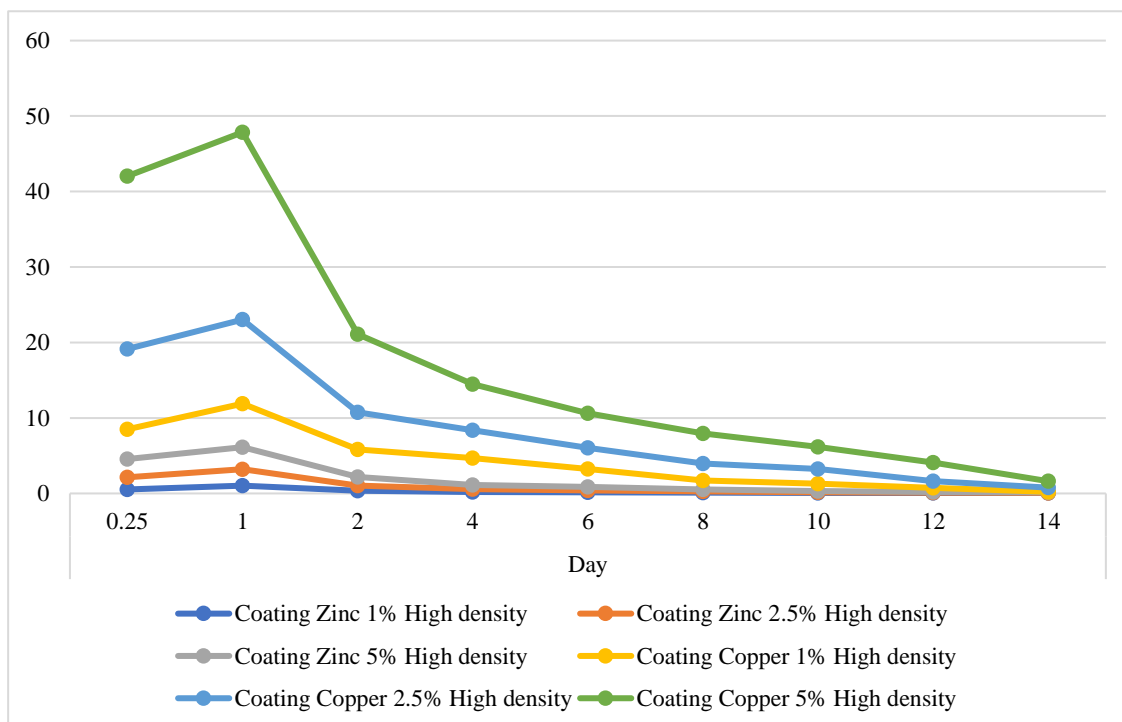


Fig 14: Chemical leaching of coating treated high density coconut wood at different time intervals

There were notable variations in the chemical leaching with respect to various densities, type of nanoparticles, and their concentrations when it was treated to coconut wood using different application techniques (Table 14). High density has shown the minimum leaching of 19.2 ppm when pressure was applied, compared with medium density (24.4 ppm) among various densities used. Among various nanoparticles, zinc treated to high and medium density had shown low leaching (5.76 ppm and 6.62 ppm, respectively). While both medium and high-density treated with copper had shown high leaching of 43.69 ppm and 35.75 ppm, respectively. Generally leaching value was observed to be increasing with advancing concentrations of nanoparticles. A significant difference had been recorded among various nanoparticle concentrations when applied using pressure, with 1% zinc treated to both high and medium-density showing low leaching of (1.79 ppm and 2.49 ppm) which was par with other concentrations of zinc 2% and 5% treated to both densities. While medium density treated with 5% copper showed the maximum leaching of 62.28 ppm, followed by 5% copper high density (49.5 ppm).

In case of coating treatment, among the densities used, lowest leaching of 25.8 ppm was showed in high density samples, whereas medium density showed highest leaching of 30.8 ppm.

Among other nanoparticles with different densities, zinc treated to the high densities showed lowest leaching (5.02 ppm), which was on par zinc treated medium density samples (5.86 ppm). On the other hand, copper-treated medium-density exhibited the maximum leaching of 55.76 ppm, followed by copper treated high density wood treatment (46.61 ppm). Similar to the pressure treatment, coating treatments also showed similar results with effects of chemical concentrations on leaching. The lowest leaching levels of 2.56 ppm was observed in 1% zinc treated medium density samples, which were comparable to all other concentrations of zinc treated to both medium and high densities. Conversely, the maximum leaching of 90.31 ppm was observed in the case of 5% copper treated medium density, followed by 5% copper high density (78.89 ppm), 2.5% copper treated medium density (49.57 ppm). In both method of application zinc treated sample treated to both densities had less than 10 ppm value.

Table 14: Total quantity of chemical leaching value from the treated coconut wood samples (ppm)

<u>Pressure Treatment</u>					<u>Coating Treatment</u>				
Nanoparticles and its concentrations	Density				Nanoparticles and its concentrations	Density			
	Medium		High			Medium		High	
	Copper	Zinc	Copper	Zinc		Copper	Zinc	Copper	Zinc
1%	23.14	2.49	19.01	1.79	1%	27.4	3.2	22.08	2.56
2.50%	42.67	5.01	33.95	4.24	2.50%	49.57	5.37	38.78	4.97
5%	62.28	7.55	49.5	6.43	5%	90.31	9.0	78.89	7.52
Average of nanoparticle	43.69	6.62	35.75	5.76	Average of nanoparticle	55.76	5.86	46.61	5.02
Average of density	24.4		19.2		Average of density	30.8		25.8	
Average of density-Significant at 0.001%					Average of density-Significant at 0.001%				
Average of nanoparticle-Significant at 0.001%					Average of nanoparticle-Significant at 0.001%				
Average of concentration-Significant at 0.001%					Average of concentration-Significant at 0.001%				

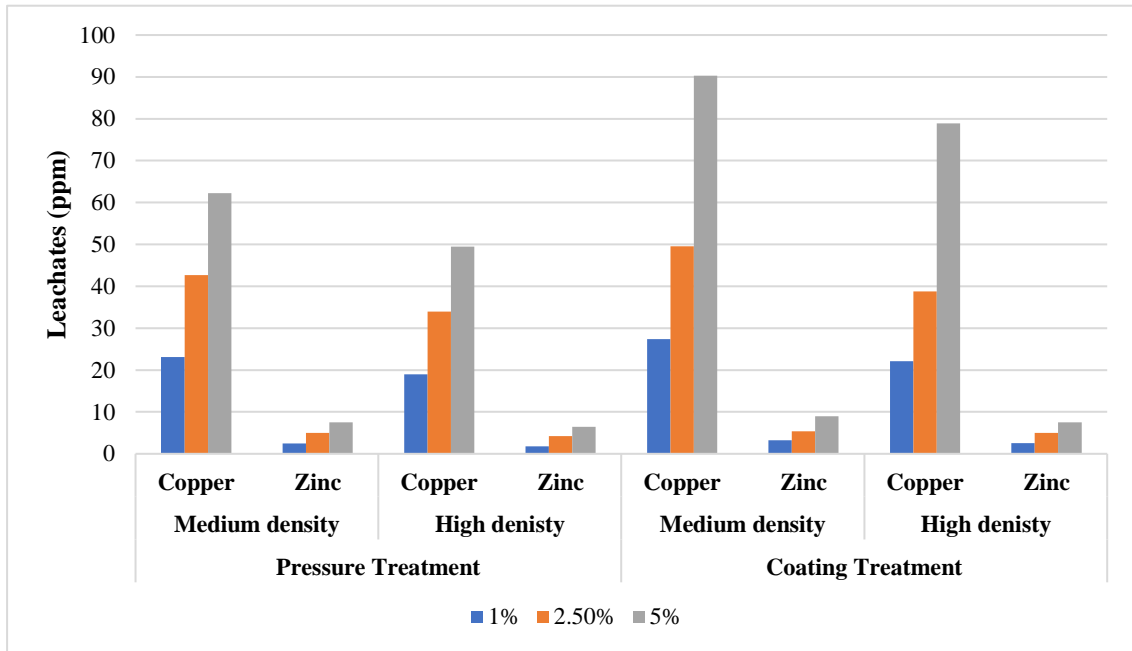


Fig 15: Total quantity of chemical leaching value from the treated coconut wood samples (ppm)

Chemical leaching in the context of wood preservation refers to the release or migration of chemicals from treated wood into the surrounding environment. Factors influencing chemical leaching include product factors (exposure time, surface area, and wood properties), preservative components, and exposure factors (water conditions and substrate composition) (Lebow, 1996).

The study showed that medium-density is susceptible for greater leaching (24.4 ppm-pressure treatment; 30.8 ppm-coating treatment) than high-density wood (19.2 ppm-pressure treatment; 25.8 ppm-coating treatment) (Figure 15). The probable reason for this could be the presence of more pores in the lower density woods than the denser wood (Barnett and Jeronimidis, 2003). As a result of more pores, it could lead to an increase in surface area (Tian *et al.*, 2016). Thus, if surface area is more which leads to increase in leaching (Lebow, 1996). The present study results are supported by the Bak and Nemeth (2018) study on temperate woods. Where they concluded that more leaching from pine wood than beech wood due to difference in the pore size and density.

The present study shown a contradictor result for the effect of different nanoparticles on leaching. In the current study, leaching of nano-zinc was significantly lower than nano-copper (Figure 15). Contrastingly, a study of leaching behaviour on

southern yellow pine treated with zinc and copper nanoparticle carried out by Kartal *et al.*, (2009) showed lower leaching of nano-copper (<1%) while nano-zinc attribute to moderate leaching (31%). The similar results was also reported by Copper (2008) and Mantanis *et al.* (2014). More leaching of copper than the zinc might be due to more nano particles size of copper than zinc. As a result, not much deeper penetration might have occurred (section 4.2.1). The other reason may be due to lower retention bonding with the wood.

In both pressure and coating applications irrespective of the density of wood and nanoparticles, the general trend was observed among the different nanoparticle concentrations i.e., leaching of nanoparticles increased with increasing concentrations. This finding is in accordance with Mantanis *et al.* (2014), Temiz *et al.* (2014) and Bek and Németh (2018).

A similar trend has been seen in our study when nano-copper and nano-zinc treated to medium and high density wood using pressure and coating as a method of application (Figure 15).

4.2.3. Subterranean Termite test

Subterranean termite test was conducted to know the efficiency of nanoparticle treated wood against termite attack. A no-choice test was conducted to know the effect of termite on each treatments. Results of the termite attack shows that, a significant difference among various density, and type of nanoparticles used while no significant difference was recorded between various nanoparticle concentrations irrespective of method of applications (Table 15).

With pressure treatment, between densities, medium density had shown more susceptible to termite attacks with 10.85% mass loss than high density wood (4.46%).

Between different nanoparticles using pressure, copper treated to high density had shown least mass loss of 0.49%. It was statistically similar with copper treated to medium density (0.99%), zinc treated to medium (1.96%) and high density (0.86%). While control with no treatment of medium and high density had shown greater mass loss percentage of 29.61% and 12.02% respectively.

No significant difference was recorded, among various nanoparticle concentrations. Within copper no much variation between concentrations with 5% copper treated to high density shows least mass loss percentage of 0.42%. Similar results was recorded with zinc and its various concentration.

With an 11.3% mass loss during the coating treatment, medium density wood was found to be more vulnerable to termite attacks than high density wood (4.65%).

When coating was applied to type of nanoparticles, copper treated to a high density showed the least amount of mass loss (0.52%). With copper treated to medium density (1.02%), zinc treated to medium density (2.78%), and high density (1.13%), the statistical results were comparable. The control group, which did not receive any treatment for medium or high density, exhibited a higher percentage of mass loss (30.37% and 12.30%, respectively). The lowest mass loss percentage is shown by 5% copper treated to high density among the different concentrations used.

There was no discernible variation found between the different nanoparticle concentrations. There is little difference in copper concentrations; 5% zinc treated at high density exhibits the lowest mass loss percentage of 0.43%. Comparable outcomes were not shown for copper at different concentrations.

4.2.3.1. Visual Examination

The visual scoring of termite attack to coconut wood treated with different treatment using pressure and coating as a method of application is shown in Table 16 and 17 respectively.

Among both treatment 2.5% and 5% copper nanoparticle treated to both density wood showed no damage even after 45 days of incubation in the termite mound thus has been rate as 10 (Plate 14-17). While nano-zinc treated wood sample shown same results has of copper except for 2.5% zinc medium density treated using coating had showed slight degradation to termite attack rated as 9.8 for pressure treated and 8.6 for coating method. For all 1% nanoparticle treated to both densities had shown degradation against termite attack with zinc more susceptible than copper. Overall control sample of both density had shown most susceptible to termite. Within this, medium density had showed

Table 15: Mass loss of treated coconut wood samples after subterranean termite test (%)

<u>Pressure Treatment</u>							<u>Coating Treatment</u>						
Nanoparticles and its concentration	Density						Nanoparticles and its concentration	Density					
	Medium			High				Medium			High		
	Control	Copper	Zinc	Control	Copper	Zinc		Control	Copper	Zinc	Control	Copper	Zinc
1%	29.61	1.25	3.96	12.02	0.58	1.70	1%	30.37	1.25	5.27	12.30	0.60	2.14
2.50%		0.98	1.10		0.46	0.46	2.50%		0.96	2.04		0.51	0.81
5%		0.72	0.81		0.42	0.43	5%		0.83	1.05		0.45	0.43
Average of nanoparticle	29.61	0.99	1.96	12.02	0.49	0.86	Average of nanoparticle	30.37	1.02	2.78	12.30	0.52	1.13
Average of density	10.85			4.46			Average of density	11.3			4.65		
Average of density-Significant at 0.001%							Average of density-Significant at 0.05%						
Average of nanoparticle-Significant at 0.001%							Average of nanoparticle-Significant at 0.001%						
Average of concentration-Not significant							Average of concentration-Not significant						

more damage to termite with more than 50% of cross-section area affected making it as failure (Plate. 14 & 16)

Table 16: Visual scoring of termite attack on pressure treated coconut wood samples

Treatments	Visual Score
Control medium density	3
Control high density	6.2
1% zinc medium density	8.2
1% zinc high density	8.8
1% copper medium density	9.8
1% copper high density	10
2.5% zinc medium density	9.8
2.5% zinc high density	10
2.5% copper medium density	10
2.5% copper high density	10
5% zinc medium density	10
5% zinc high density	10
5% copper medium density	10
5% copper high density	10

Table 17: Visual scoring of termite attack on coating treated coconut wood samples

Treatments	Visual Score
Control medium density	3
Control high density	5.8
1% zinc medium density	6.8
1% zinc high density	8.2
1% copper medium density	9.8
1% copper high density	9.9
2.5% zinc medium density	8.6
2.5% zinc high density	10
2.5% copper medium density	10
2.5% copper high density	10
5% zinc medium density	10
5% zinc high density	10
5% copper medium density	10
5% copper high density	10



Plate 14: Effect of termite on pressure treated with nanoparticles to medium density coconut wood after incubation for subterranean termite test

A) Control, B) Zinc 1%, C) Copper 1%, D) Zinc 2.5%, E) Copper 2.5%, F) Zinc 5%, and G) Copper 5%.

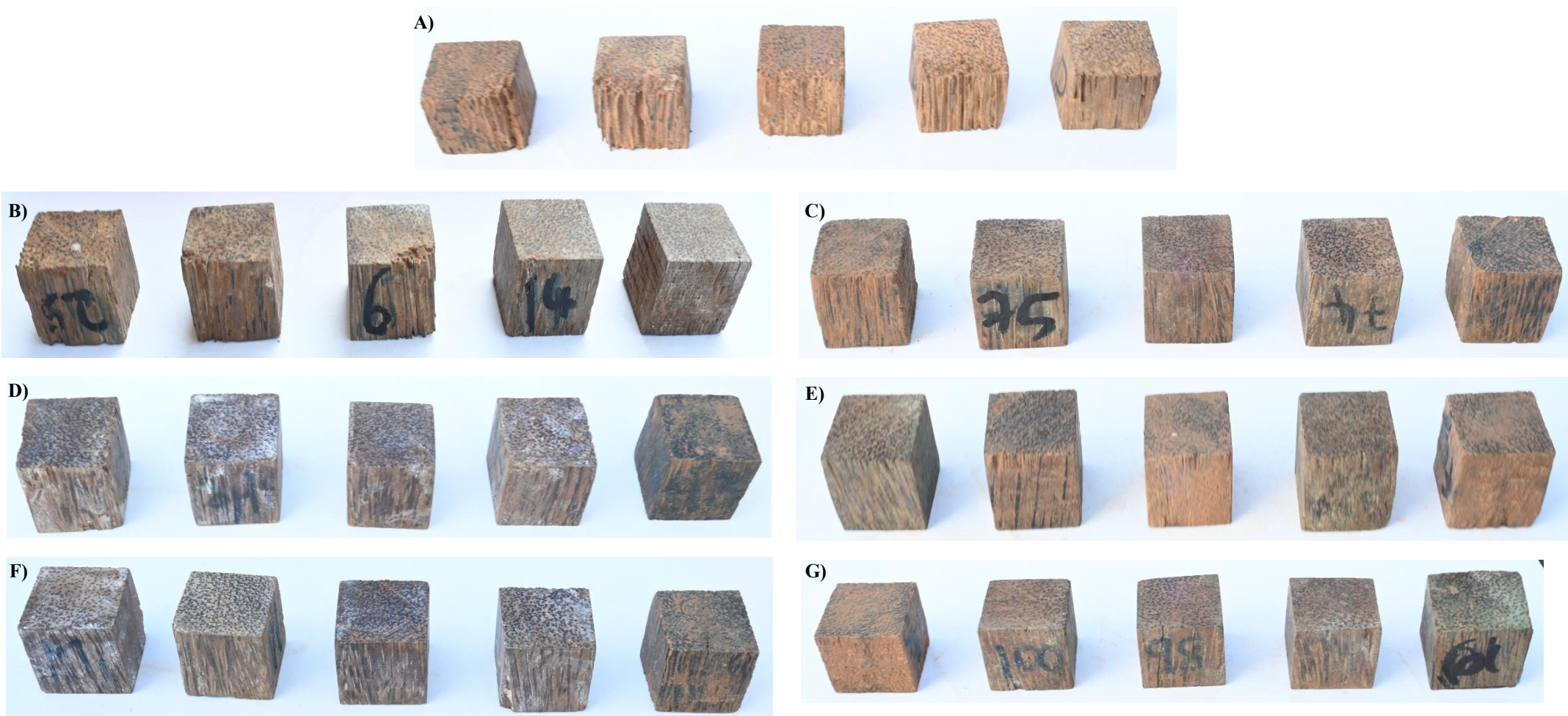


Plate 15: Effect of termite on pressure treated with nanoparticles to high density coconut wood after incubation for subterranean termite test

A) Control, B) Zinc 1%, C) Copper 1%, D) Zinc 2.5%, E) Copper 2.5%, F) Zinc 5%, and G) Copper 5%.

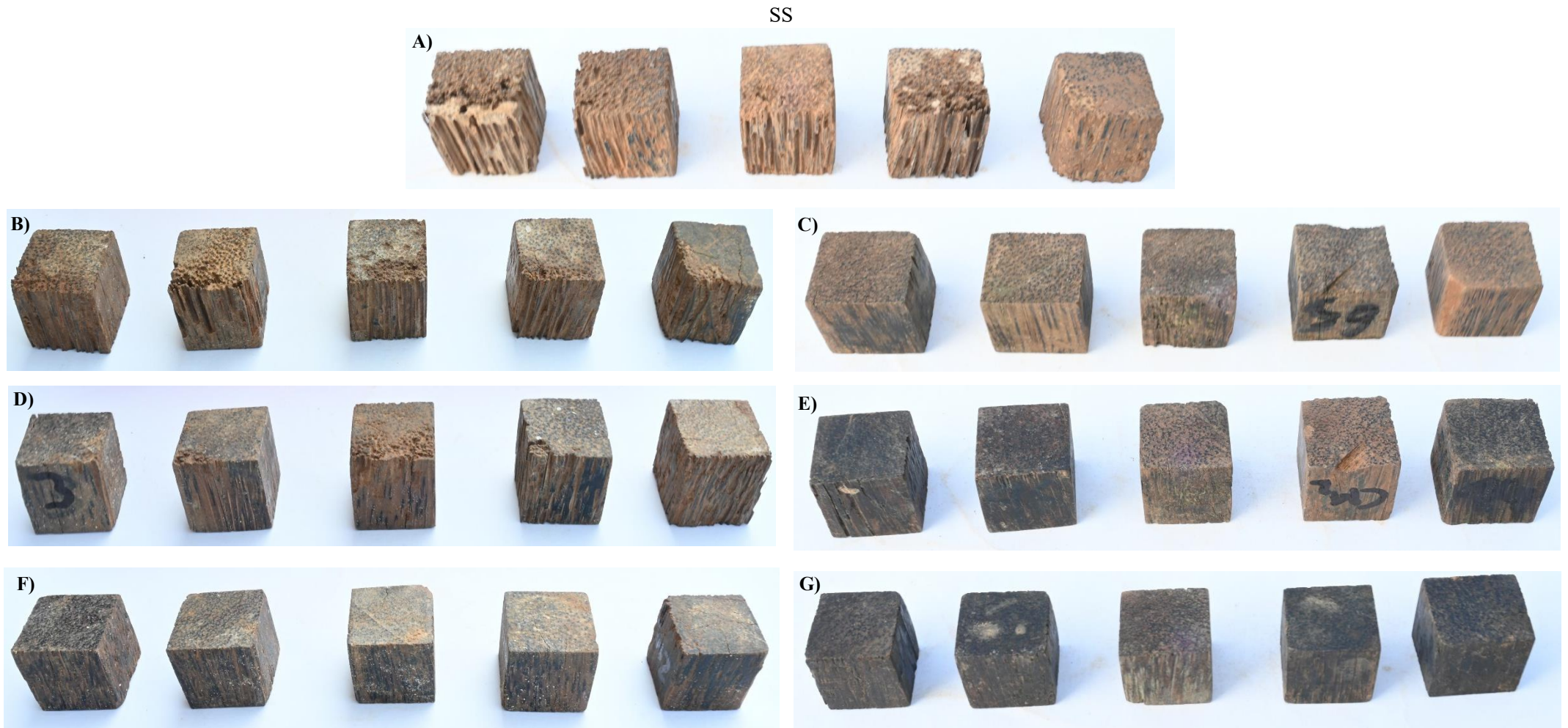


Plate 16: Effect of termite on coated with nanoparticles to medium density coconut wood after incubation for subterranean termite test
A) Control, B) Zinc 1%, C) Copper 1%, D) Zinc 2.5%, E) Copper 2.5%, F) Zinc 5%, and G) Copper 5%.



Plate 17: Effect of termite on pressure treated with nanoparticles to high density coconut wood after incubation for subterranean termite test

A) Control, B) Zinc 1%, C) Copper 1%, D) Zinc 2.5%, E) Copper 2.5%, F) Zinc 5%, and G) Copper 5%.

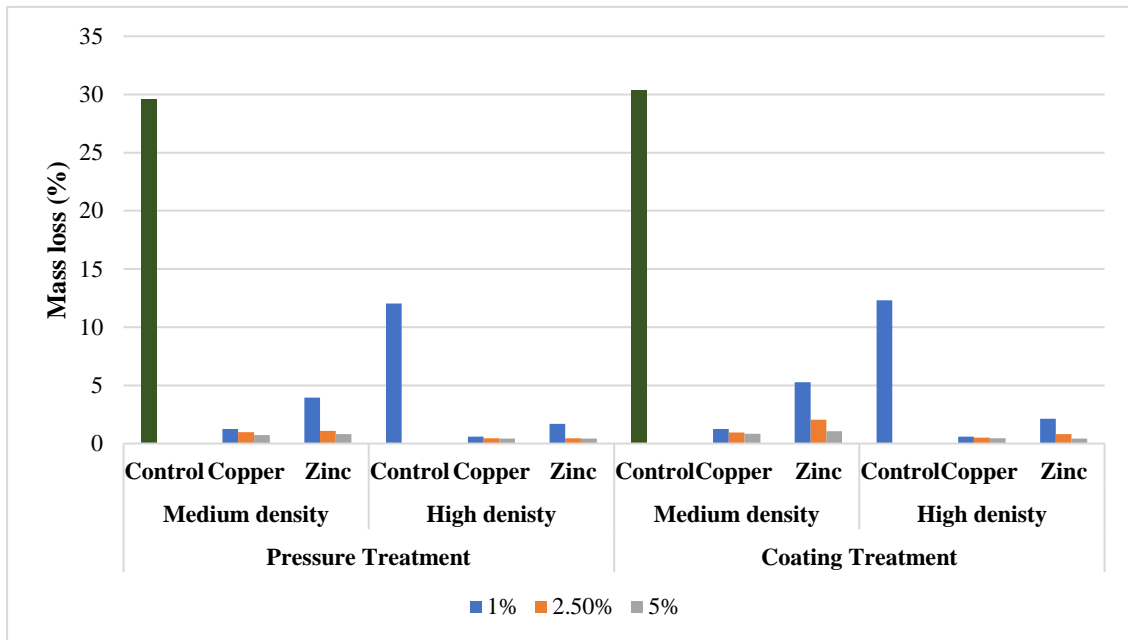


Fig 16: Mass loss of treated coconut wood samples after subterranean termite test (%)

Termites are known for their ability to damage wooden structures by feeding on cellulose, a main component of wood (Ghaly and Edwards, 2011). Researchers have looked into a number of strategies, and one of them uses nanoparticles to keep termites out of wood. Because they are so tiny, nanoparticles can seep into the wood and strengthen its defence against termites.

In both method of application, medium density had showed more susceptible to termite attacks than hardwood density (Figure 16). The reason for the susceptible of the former is due to less resistance to termite attack has it contain high content of cellulose compound and low percentage of chemical or extractive that protect wood against termite attack (Peters *et al.*, 2014; Sujithra *et al.*, 2022).

A contradictor results has been observed in our study, where nano-copper treated showed slightly high termite mass loss percentage of 10-21% (Mantanis *et al.*, 2014) while nano-zinc treated wood showed low mass loss percentage of 4-7%. Similar results were recorded in Kartal *et al.* (2009), and Green and Arango (2007). The reason may be due to high leaching of copper nanoparticle which leads to slow release of copper ions in the soil making it toxic to termites as copper is considered as one of the insecticides to control termites (Mwalongo, 1999).

The zinc treated had shown mass loss percentage ranging from 0.81-5.27% among various concentration (Figure 16). Similar results were recorded in Clausen *et al.* (2009), Kartal *et al.* (2009) and Mantanis *et al.* (2014) with slightly higher value than the value reported in our study.

Among various concentration no variation is seen statistically. When compared with control and nanoparticle treated wood, control had shown more susceptible to termite attacks (Figure 16). The untreated control with and without fixative had shown greater mass loss of more than 25% getting a least score.

Thus, we can conclude that a minimum concentration of nano-copper to protect coconut wood against termite attack is 1% irrespective of density, while nano-zinc is 2.5%.

The visual examination studies showed nano-zinc treated wood had shown more score than copper treated wood and control (Clausen *et al.*, 2009; Kartal *et al.*, 2009). While there has been a contradictory finding in our study may be attribute to above mentioned reason.

4.2.4. Weatherability Test

Wood is treated with nanoparticles to increase its resistance to weathering damage, discoloration, and UV deterioration. Based on mass loss and greying, the UV weathering process aids in assessing the functionality and robustness of wood treated with nanoparticles in outdoor circumstances.

When coconut wood coated with nanoparticles was subjected to a natural weathering test, different densities and nanoparticles and their various nanoparticle concentrations in the process differed significantly among various application methods (Table 18).

When pressure used as a method of application among density, high density showed the minimum reduction in mass loss percentage of 3.31% when compared to medium density (6.99%). Among various nanoparticles used, zinc treated to high density showed low reduction in mass loss (0.68%) which was par with copper high density (0.80%). While control medium density and high recorded the maximum mass loss percentage of 15.79% and 6.33% respectively.

Different concentrations of nanoparticle applied to various densities treated under pressure showed a statistical difference. 5% zinc treated to high density showed the lowest mass loss percentage of 0.31% which was slightly parring with 2.5% zinc high density (0.61), 5% copper high density (0.68%), 5% zinc medium density (0.75%), 2.5% zinc of medium density (0.79%), 1% zinc high density (1.11%), and, 5% copper medium density (1.39%). While control treated to medium and high density showed the maximum mass loss percentage of 15.79 and 6.33% when pressure was used as a method of application.

High density demonstrated the minimum reduction in mass loss percentage of 2.36% when coating was used as a method of application among densities, in contrast to medium density (5.63%). Among the different nanoparticles utilised, zinc treated to a high density demonstrated a low mass loss reduction (0.75%), comparable to that of copper treated to a high density (0.88%). The maximum mass loss percentages for the control medium density and high were 11.43% and 5.44%, respectively.

Table 18: Mass loss of treated coconut wood samples after weatherability test (%)

<u>Pressure Treatment</u>							<u>Coating Treatment</u>						
Nanoparticles and its concentrations	Density						Nanoparticles and its concentrations	Density					
	Medium			High				Medium			High		
	Control	Copper	Zinc	Control	Copper	Zinc		Control	Copper	Zinc	Control	Copper	Zinc
1%	15.79	4.43	3.46	6.33	0.94	1.11	1%	11.43	5.05	3.78	5.39	1.11	1.20
2.50%		3.40	2.14		0.79	0.61	2.50%		2.97	2.16		0.86	0.67
5%		1.39	0.75		0.68	0.31	5%		1.59	0.88		0.68	0.38
Average of nanoparticle	15.79	3.071	2.11	6.33	0.803	0.676	Average of nanoparticle	11.43	3.2	2.27	5.44	0.88	0.75
Average of density	6.99			3.13			Average of density	5.63			2.36		
Average of density-Significant at 0.001%							Average of density-Significant at 0.001%						
Average of nanoparticle-Significant at 0.001%							Average of nanoparticle-Significant at 0.001%						
Average of concentration-Significant at 0.01%							Average of concentration-Significant at 0.001%						










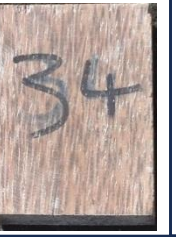
































	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
Control						
Zinc 1%						
Zinc 2.5%						
Zinc 5%						
Copper 1%						
Copper 2.5%						
Copper 5%						

Plate 18: Greying effect on pressure treated with nanoparticles to medium density coconut wood during weathering test






























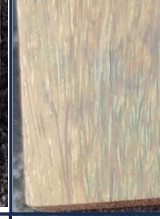
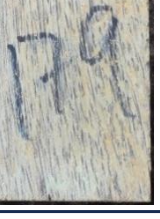





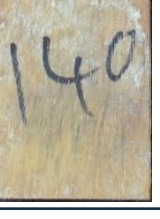





	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
Control						
Zinc 1%						
Zinc 2.5%						
Zinc 5%						
Copper 1%						
Copper 2.5%						
Copper 5%						

Plate 19: Greying effect on pressure treated with nanoparticles to high density coconut wood during weathering test















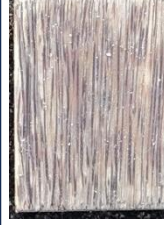

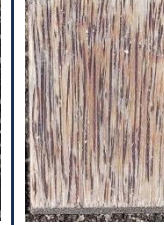
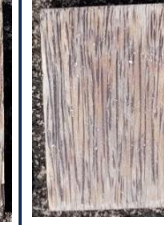





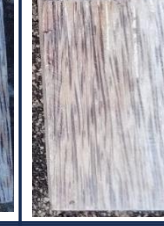














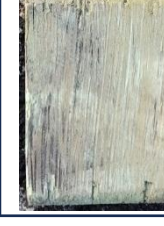



	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
Control						
Zinc 1%						
Zinc 2.5%						
Zinc 5%						
Copper 1%						
Copper 2.5%						
Copper 5%						

Plate 20: Greying effect on coated treated with nanoparticles to medium density coconut wood during weathering test











































	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
Control						
Zinc 1%						
Zinc 2.5%						
Zinc 5%						
Copper 1%						
Copper 2.5%						
Copper 5%						

Plate 21: Greying effect on coated treated with nanoparticles to high density coconut wood during weathering test

A statistical difference was observed between different nanoparticle concentrations applied to different densities treated under coating. The mass loss percentage of 0.38% for 5% zinc treated to high density was the lowest, matching that of 2.5% zinc treated to high density (0.67) and 5% copper treated to high density (0.68%). When pressure was applied, the control group subjected to medium and high densities displayed the maximum mass loss percentages of 11.43% and 5.44%, respectively. Next in order of treatment are 1% copper (5.05%) and 1% zinc (3.78%) to medium density.

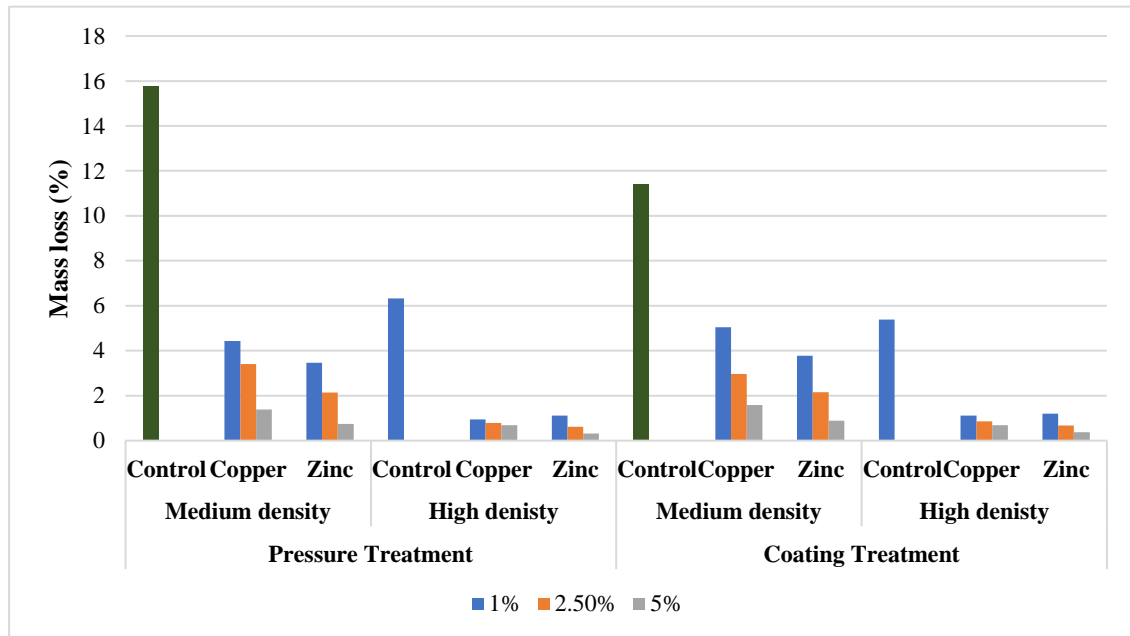


Fig 17: Mass loss after Weatherability test (%)

Use of wood and its modification for cladding and other building functions has been extensively used from the dawn of human civilization. It not only provides aesthetic value but also helps in protection the interior parts against harsh environmental conditions (Hill *et al.*, 2022). To evaluate the property of wood to withstand greying (colour change), mass loss, cracks, splits, abrasion, flaking and other defects weathering test was conducted. Factors influencing outdoor weathering includes chemical, mechanical, light energy factors and wood properties and its modification (Feist, 1990).

Figure 17 shows that lower density wood weathered faster than higher density wood (Hill *et al.*, 2022). Thus, among density, higher density showed lower weathering mass loss percentage than medium density wood when used treated with different nanoparticles and their concentration irrespective of method of application. The reason

for this can be attribute to inherent properties of wood includes, cell wall and its properties, moisture, and extractive (Feist, 1990).

A significant difference was recorded with respect to nanoparticles used when treated to both density of wood using pressure and coating as a method of application (Figure 17). In sunscreen products, Zinc oxide is one of the UV-shielding materials. As a result of this property of Zinc oxides, leads to low weathering loss or mass loss (Clausen *et al.*, 2009; Green and Kartal, 2010). Similarly, micro and nano form of copper has the ability to block the outdoor weathering (Garrido *et al.*, 2016) but not as effective as zinc. Thus, results shows that lower mass loss of copper treated wood when compared with controls.

As the concentration of the nanoparticles used increases especially nano-zinc oxide there is an effective reduction in mass loss percentage. Thus 5% zinc treated to both density showed least mass loss percentage followed by 2.5% zinc. While 5% copper treated to both densities showed slightly higher mass percentage value than zinc both significantly lower value than the control (Figure 17). Similar results were recorded in Clausen *et al.* (2009), Weichelt *et al.* (2011), Terzi *et al.* (2016) in case of zinc and Garrido *et al.* (2016), Lankone *et al.*(2019) in case of copper.

4.2.4.1. Greying

When wood is exposed to the elements especially moisture and sunlight, it naturally begins to turn grey. UV radiation or heat waves may have induced lignin degradation and the loss of natural wood colours are the main causes of this phenomena (Reinprecht *et al.*, 2018).

Among pressure treated wood, from week 1 to week 6 there has been a significant greying of wood in control in medium density than hardwood density (Plate 18 & 19). Similarly 1% nano-zinc and nano-copper treated to both showed moderate greying when compared to control, while greying was more effective after 4th week. While 2.5% nano-zinc and copper treated to medium and high density had shown slightly greying after 5th week and 6th week respectively. In contrast, there is no greying effect in 5% nano-zinc and copper treated wood in both density but, copper had shown colour change from ochre to blue-green indicating oxidation of copper (Plate). In all treatment at 2nd-3rd week slight absorption of moisture due to heavy rain.

A significant greying effect has been recorded among density wise when treated using coating with medium density showing more susceptible to greying than high density.

Among coating method of treatment, from week 1 to week 6 there has been a significant greying of wood in control in medium density than hardwood density (Plate 20 & 21). Similarly 1% nano-zinc and nano-copper treated to both showed moderate greying when compared to control, which was more effective after week 2. While 2.5% nano-zinc and copper treated to medium and high density had shown slightly greying after week 5. In contrast, there is no greying effect in 5% nano-zinc and copper treated wood in both density but, copper had shown colour change from ochre to blue-green indicating oxidation of copper (Plate 18-21). In all treatment at 2nd – 3rd week, absorption of moisture due to heavy rain. All fixative got leached after two week, as this fixative has no hydrophobic property (Lee *et al.*, 2020).

4.2.5. Water absorption Test

4.2.5.1. Water absorption percentage

The efficacy of adding nanoparticles to wood to improve its water resistance and durability is determined by the water absorption test, which is an essential assessment technique.

When coconut wood coated with nanoparticles was subjected to a water absorption test, different densities, nanoparticles and their various concentrations in the process differed significantly among various method of application (Table 19).

With pressure as a method of treatment, among density high-density had shown a minimum water absorption percentage of 15.6% than medium-density (27.2%). Among various nanoparticles, zinc treated to high-density had low water absorption (10.1%), followed by copper high-density (14.1%), and zinc medium-density (19.2%). While control medium-density and copper medium-density had shown high water absorption of 39.7% and 24.4%, respectively.

Among various nanoparticle concentrations, 5% zinc treated to high density had shown less absorption of 6.63%, slightly par with 2.5% zinc treated to high density (9.24%) and 5% copper treated to high density (11.94%). While control treated to medium density had shown high water absorption of 39.66% followed by 1% copper medium density (28.44%) when pressure was used as a method of application.

When coconut wood sample coated with different treatments, a significant difference in water absorption among medium and high density of 28.4% and 16.0% respectively. Among various nanoparticles, zinc treated to high density showed low water absorption percentage of 10.7% followed by copper applied to high density (16.0%).

At different nanoparticle concentrations, low absorption of 7.13% was seen in the 5% zinc treated to high density, which was on par with the 2.5% zinc treated to high density (9.74%) and the 5% copper treated to high density (12.85%). In contrast, a control group treated to medium density exhibited a high-water absorption rate of 40.48%. This was followed by a medium density of 1% copper (27.72%) and 1% zinc (26.2%) when coating was used as a method of application.

Table 19: Water Absorption behaviour of treated coconut wood samples (%)

<u>Pressure Treatment</u>							<u>Coating Treatment</u>						
Nanoparticles and its concentrations	Density						Nanoparticles and its concentrations	Density					
	Medium			High				Medium			High		
	Control	Copper	Zinc	Control	Copper	Zinc		Control	Copper	Zinc	Control	Copper	Zinc
1%	39.66	28.44	25.13	22.54	17.28	14.56	1%	40.48	27.72	26.2	21.43	18.24	15.17
2.50%		22.19	17.53		13.2	9.24	2.50%		23.54	18.35		16.8	9.74
5%		18.14	14.83		11.94	6.63	5%		19.92	16.82		12.85	7.13
Average of nanoparticle	39.7	24.4	19.2	22.5	14.1	10.1	Average of nanoparticle	40.5	23.3	20.5	21.4	16.0	10.7
Average of density	27.2			15.6			Average of density	28.4			16.0		
Average of density-Significant at 0.001%							Average of density-Significant at 0.001%						
Average of nanoparticle-Significant at 0.001%							Average of nanoparticle-Significant at 0.001%						
Average of concentration-Significant at 0.001%							Average of concentration-Significant at 0.001%						

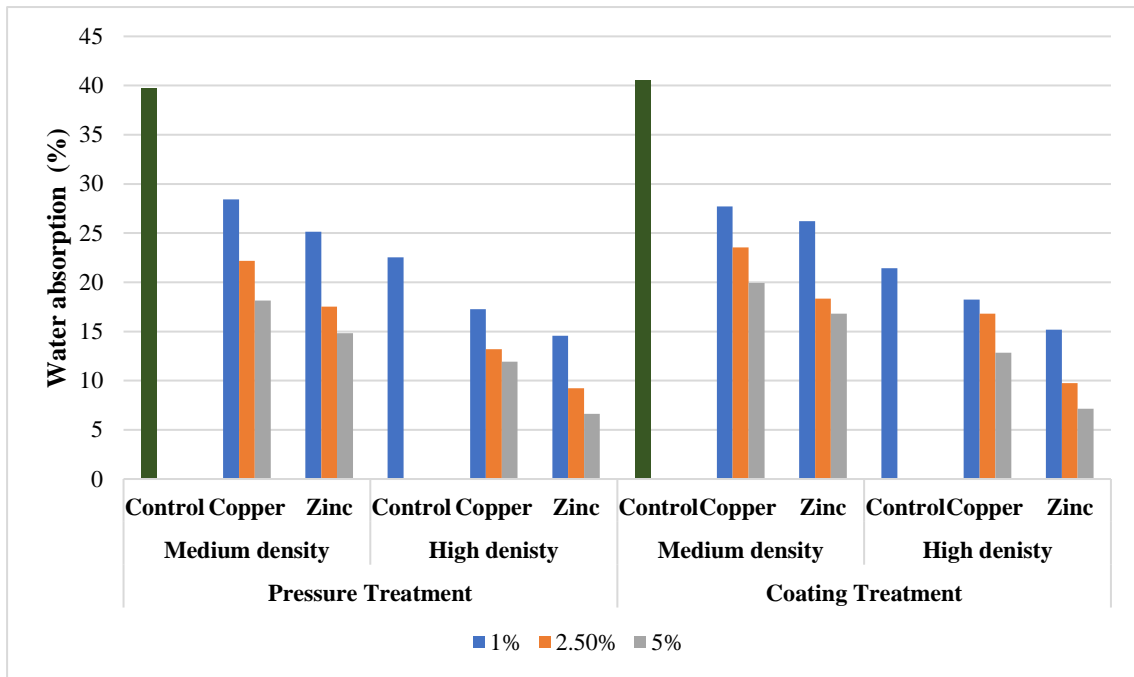


Fig 18: Water Absorption behaviour of treated coconut wood samples (%)

Wood is a hygroscopic material (Fredriksson, 2019). It absorbs moisture through diffusion and capillary flow. This moisture plays a vital role in the susceptibility of wood to, low dimensional stability, durability, moulds and decays (Fengel and Wegener, 2011; Bak *et al.*, 2017). As a result of this it leads to reduction in its utilization. Recent findings showed that using nanomaterials, the hygroscopicity of wood can be reduced as this nanoparticle have water contact angle more than 90° making it hydrophobic in nature and thus improve in the dimension stability (Li *et al.*, 2007 and Wang and Piao, 2011). As a result of these it reduces the occurrence of above-mentioned problems.

Among various density, high density had shown less water absorption percentage than medium density when treated with different nanoparticles used irrespective of treatment method (Figure 18). The reason for the greater absorption of medium density than high density can be attributed to more pores structure of medium density than high density wood which results in more water uptake (Barnett and Jeronimidis, 2003). Hence, medium density wood has strong tendency to water absorption than high density wood.

A significant difference was recorded with respect to nanoparticles used when treated to both densities of wood using pressure and coating as a method of application (Figure 18). Nano-zinc and copper has an ability to improve dimensional stability and hygroscopicity as it as water contact angle more than 90° making it more hydrophobic

nature (Muhcu *et al.*, 2017). But zinc has more effective in reducing hygroscopicity than copper (Taghiyari and Farajpour, 2013). As a result of this, low water absorption percentage was recorded in wood treated to nano-zinc than wood treated to nano-copper and controls. To support this, Terzi *et al.* (2016), who concluded nano-zinc had least water absorption percentage when compared with others. Similar results were recorded by Green and Kartal (2010) and Soltani *et al.* (2013).

As the concentration of the solution increases the water absorption behaviour gradually reduced due to increase in the concentration of the solution used in the treatment when used as a pressure treatment (Green and Kartal, 2010). Hence study showed that 5% zinc treated to high density showed lower water absorption percentage than other treatment used at varying concentration. When coating used as a method of application, study shows same trend. But pressure treated sample showed slightly low water absorption than coated treatment (Figure 18).

4.2.5.2. Volume swelling percentage

The effectiveness of adding nanoparticles to wood reduces swelling and dimensional changes brought on by moisture absorption is fundamentally assessed by the volume swelling test of wood treated with nanoparticles. This test is crucial to comprehending how wood's structural stability and durability may be improved by nanoparticle treatments, particularly in high-humidity or moisture-exposed situations.

When coconut wood treated with nanoparticles was subjected to a volume swelling test, different densities and nanoparticles and their various concentrations in the process differed significantly when pressure was used as a method of application (Table 20).

High density has shown a low volume swelling percentage of 3.11% when pressure was applied, compared with medium density (4.27%) among various densities used.

In comparison with different nanoparticles, zinc and copper applied at a medium density showed a moderate volume swelling percentage of 2.83 and 3.11%, while zinc and copper treated at a high density showed a low value of 1.9% and 2.6%, respectively.

In contrast, the medium-density control group displayed a high a volume swelling percentage of 5.45%.

Among different concentrations of nanoparticle applied to various densities treated under pressure showed a statistical difference. 5% zinc treated to high density showed lowest a volume swelling percentage of 1.47% which was par with 2.5% zinc high density (1.49%), 5% zinc medium density (1.76%), 1% zinc high density (2.16%), 5% copper high density (2.26%) and, 5% copper medium density (2.35%). While control treated to medium density showed maximum a volume swelling percentage of 5.45%.

When coating is used as a method of application, high density outperforms medium density (4.31%) in terms of a volume swelling percentage, with a low of 3.9%.

Zinc and copper did not differ much among the different nanoparticles, but they did significantly differ when compared to the control. When applied to medium density, copper and zinc showed maximum a volume swelling percentages of 3.0% and 3.21%, respectively, while when treated to high density, copper and zinc showed the minimum a volume swelling percentages of 2.92% and 2.94%, respectively. In contrast, the medium-density control group displayed a high a volume swelling percentage of 5.49%.

The results of applying varying nanoparticle concentrations to densities under coating revealed a marginally small statistical difference. Volume swelling percentage of 1.77% for 5% zinc treated to high density was the lowest when compared to 5% copper high density (2.11%), 2.5% copper high density (2.33%), 2.5% zinc high density (2.44%), 5% zinc medium density (2.46%), and 5% copper medium density (2.56 %). The maximum a volume swelling percentage observed in the control group that was subjected to medium density was 5.45%

Table 20: Volume swelling behaviour of treated coconut wood samples (%)

<u>Pressure Treatment</u>							<u>Coating Treatment</u>						
Nanoparticles and its concentrations	Density						Nanoparticles and its concentrations	Density					
	Medium			High				Medium			High		
	Control	Copper	Zinc	Control	Copper	Zinc		Control	Copper	Zinc	Control	Copper	Zinc
1%	5.45	4.07	4.02	3.39	3.12	2.73	1%	5.49	3.96	3.97	3.35	2.69	2.96
2.50%		2.91	2.7		2.43	1.49	2.50%		3.12	2.58		2.33	2.44
5%		2.35	1.76		2.26	1.47	5%		2.56	2.46		2.11	1.77
Average of nanoparticle	5.45	3.11	2.83	3.39	2.6	1.9	Average of nanoparticle	5.49	3.21	3.0	3.35	2.92	2.94
Average of density	4.27			3.11			Average of density	4.31			3.9		
Average of density-Significant at 0.01%							Average of density-Significant at 0.001%						
Average of nanoparticle-Significant at 0.001%							Average of nanoparticle-Significant at 0.001%						
Average of concentration-Significant at 0.05%							Average of concentration-Significant at 0.1%						

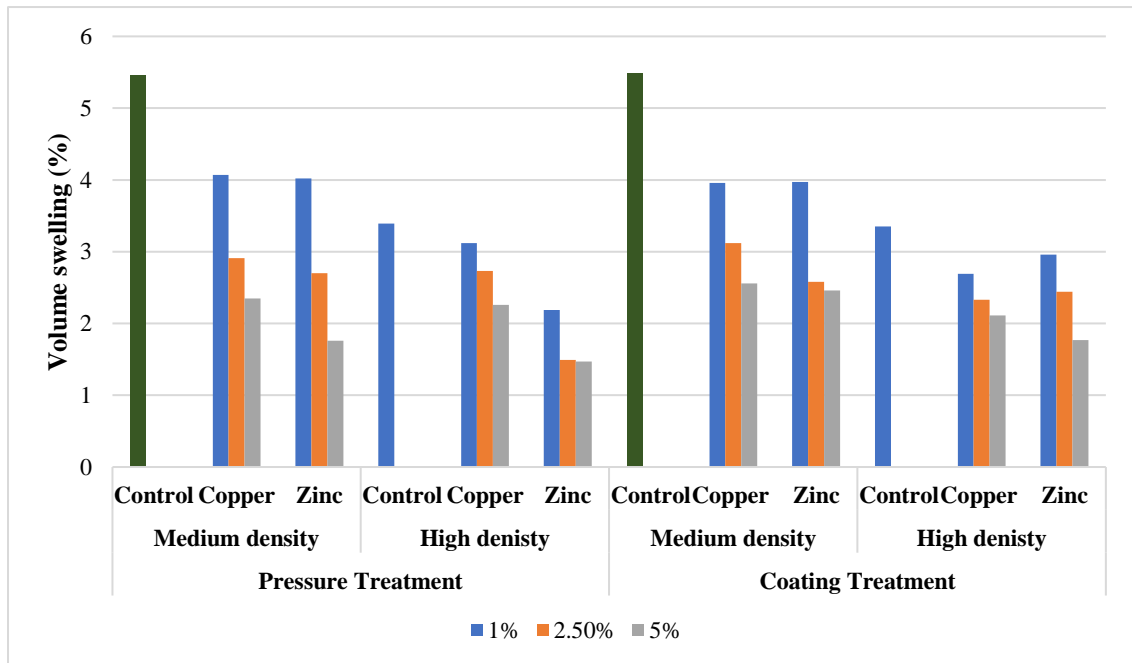


Fig 19: Volume swelling behaviour of treated coconut wood samples (%)

The volume swelling of wood treated with nanoparticles depends on the type of nanoparticles its concentration, and treatment process. Nanoparticles can enhance wood's properties, such as dimensional stability and environmental resistance. Proper application can minimize humidity changes, especially in outdoor applications where wood is exposed to varying conditions (Holy *et al.*, 2020).

Regardless of the method of treatment, high density had less volume swelling percentage than medium density when treated with different nanoparticles (Figure 20). Medium density wood has a strong tendency to volume expand more than high density wood because it has more pores than high density wood, which leads to more water absorption (Mantanis *et al.*, 2014). This causes greater volume swelling of medium density wood over high density wood.

When pressure and coating were used to treat the two densities of wood, a notable difference in the nanoparticles used was observed (Figure 19). According to Muhcu *et al.* (2017), nano-zinc and nano-copper have the potential to enhance dimensional stability and hygroscopicity. However, zinc reduces hygroscopicity more effectively than copper (Taghiyari and Farajpour, 2013). Consequently, compared to wood treated to nano-copper and controls, wood treated to nano-zinc showed a lower volume swelling percentage. This is corroborated by Terzi *et al.* (2016), who found that when compared to other

materials, nano-zinc had the lowest volume swelling percentage. Green and Kartal (2010) and Soltani *et al.* (2013) both reported similar outcomes.

Since the solution used in the pressure treatment gradually increases in concentration as its concentration rises, the water's absorption behaviour gradually decreases as well (Green and Kartal, 2010). As a result, the study demonstrated that 5% zinc treated to a high density had a lower percentage of water absorption than other treatments applied at different concentrations. The study revealed a similar pattern when coating was utilised as an application method. As illustrated in Figure 19, however, pressure-treated samples displayed less volume swelling than coated-treated samples. As pressure treatment is more effective method of treatment than coating (Lebow, 1996)

SUMMARY

5. SUMMARY

The study "Preservation of coconut wood using inorganic nanoparticles" was conducted in 2022–2023 at Kerala Agricultural University's College of Forestry in Vellanikkara, Thrissur. The goal is "to synthesise inorganic nanoparticles, their characterization and to test the physico-chemical and biocidal properties of coconut wood treated with inorganic nanoparticles".

A summary of the research findings are provided below:

5.1. Yield of Nanoparticles

The yield of copper nanoparticles was 1.62 g using copper sulphate pentahydrate (2.49 g) as a precursor salt under a chemical reduction process. Whereas, zinc sulphate heptahydrate (14.4 g) yields 10.1 g of zinc nanoparticles by employing the precipitation method of nanoparticle preparation.

5.2. Characterization of Nanoparticles

1. From SEM analysis, the synthesized copper (Cu) nanoparticles were irregularly cuboidal shaped with a mean size of 173.66 nm. While the synthesized zinc (Zn) nanoparticles are irregularly flake shaped with a mean size of 105.44 nm.
2. From EDX spectrum, the peak at 0.5 keV is associated with the binding energy of oxygen (OK_{α}), while the peaks at 0.94, 8.05, and 8.91 keV are associated with CuL_{α} , CuK_{α} , and CuK_{β} , respectively. While, the peak at 0.5 keV is associated with the binding energy of oxygen (OK_{α}), while the peaks at 1.01, 8.64, and 9.57 keV are associated with ZnL_{α} , ZnK_{α} , and ZnK_{β} , respectively. The weightage percentage of Cu and O in copper nanoparticles is 58.63 % and 41.37 %, respectively. While the Weightage percentages of Zn and O in zinc nanoparticles are 81.88 % and 18.12 %.
3. The FTIR spectrum of copper nanoparticles had peaks at 3442.92 cm^{-1} (Hydroxyl group), 2921.63 cm^{-1} (C-H alkyl group), 2851.72 cm^{-1} (C-H alkyl group), 1628.59 cm^{-1} (to C=O stretching), 1379.82 cm^{-1} (C-O groups), 1157.08 cm^{-1} (C-O groups), 1081.57 cm^{-1} (C-O groups), 1017.25 cm^{-1} (C-O groups), 861.9 cm^{-1} (Cu_2O), 764.59 cm^{-1} (Cu_2O), 624.32 cm^{-1} (Cu_2O), 526.47 cm^{-1}

(CuO), and 434.67 cm^{-1} (CuO). The FTIR spectrum of copper nanoparticles had peaks at 3414.83 cm^{-1} (Hydroxyl group or zinc oxide form) and 2920.66 cm^{-1} (Weak hydroxyl group or zinc oxide form), possibly due to containing zinc oxide or zinc hydroxide or other hydroxyl groups. Peak values show the existence of the in alkane at 2850.76 cm^{-1} (C-H group), 1136.83 cm^{-1} (C-O) groups. The peak at 995.09 cm^{-1} and 619.52 cm^{-1} corresponds to metal-oxygen i.e., zinc hydroxide (Zn-OH) and zinc-oxide (ZnO) stretching vibration mode.

4. XRD peak observed for copper nanoparticle at 2θ values of 29.6° , 36.4° , 42.2° , 43.3° , 50.4° , 61.3° , 73.5° , and 77.4° corresponds to 110, 111, 200, 111, 200, 220, 311, and 222 crystalline structure lattice planes of copper nanoparticles respectively. Peak value 29.5° , 36.4° , 42.2° , 61.3° , and 77.4° are related to Cu_2O while, 43.3° , 50.4° , and 73.5° are related to CuO. XRD peak observed for zinc nanoparticle at 2θ values of 31.8° , 34.4° , 36.2° , 47.5° , 56.6° , 62.9° , 66.4° , 68.0° , 69.1° , 72.6° , and 77.0° corresponds to 100, 002, 101, 102, 110, 103, 200, 112, 201, 004, and 022 crystalline structure lattice planes of zinc-oxide respectively.

5.3. Incubation for different tests

5.3.1. Chemical retention test

1. Pressure treatment
 - a. Medium density noticed a maximum retention of 4.4 kgm^{-3} compared with high density (3.87 kgm^{-3}).
 - b. Among the nanoparticles, zinc and copper treated to medium density showed high retention (4.54 kgm^{-3} and 4.26 kgm^{-3} , respectively), while copper and zinc high density recorded low retention of 3.67 kgm^{-3} and 4.08 kgm^{-3} respectively.
 - c. With different nanoparticle concentration, 5% zinc treated to medium density showing maximum retention of 6.49 kgm^{-3} which was slightly comparable with 5% copper treated to medium density (5.69 kgm^{-3}). While, 1% copper and zinc treated to high density showed minimum retention of 2.37 kgm^{-3} and 2.2 kgm^{-3} .
2. Coating treatment
 - a. Maximum retention was observed at a medium density (4.32 kgm^{-3}), whereas a high density of 3.78 kgm^{-3} was observed.

- b. While the copper treated to high density showed poor retention of 3.59 kgm^{-3} and 3.97 kgm^{-3} , respectively, the zinc treated to medium density showed high retention of 4.43 kgm^{-3} and 4.21 kgm^{-3} .
- c. Among various concentration, highest retention of 6.34 kgm^{-3} was observed for 5% copper treated to medium density, and 6.16 kgm^{-3} for 5% zinc treated to the same medium density. Conversely, minimal retention was shown by 1% zinc and copper treated to high density (1.96 kgm^{-3} and 2.09 kgm^{-3} , respectively).

5.3.2. Chemical leaching test

Among all treatment, leaching increases from 0.25 days to 1day and recorded maximum leaching at 1day of leaching. After 1day of leaching, there is a gradual reduction in the leaching value with respect to increase duration of days.

1. Pressure treatment

- a. When pressure was applied, high density demonstrated a minimum leaching of 19.2 ppm, while medium density (24.4 ppm) showed the highest leaching among the different densities used.
- b. Zinc treated to high and medium density had demonstrated low leaching (5.76 ppm and 6.62 ppm, respectively) using the pressure method in combination with other nanoparticle. However, medium- and high-density copper-treated samples demonstrated significant leaching of 43.69 ppm and 35.75 ppm, respectively.
- c. Low leaching of 1.79 ppm and 2.49 ppm was observed in 1% zinc treated to both high and medium densities; this was comparable to other zinc concentrations of 2% and 5% treated to both densities. A maximum leaching of 62.28 ppm was observed in the case of medium density treated with 5% copper; this was followed by 5% copper high density, 2.5% copper medium and high density, and 1% copper medium and high density.

2. Coating treatment

- a. The leaching that was highest was at medium density with 30.8 ppm, and the lowest was at high density (25.8 ppm).
- b. Zinc treated at medium and high densities demonstrated low leaching (5.86 ppm and 4.02 ppm, respectively), among other nanoparticles. However, samples treated with medium and high densities of copper showed significant leaching of 55.76 ppm and 46.61 ppm, respectively.

- c. Comparable to the other concentrations of zinc, 2% and 5% treated to both densities, low leaching levels of 2.56 ppm and 3.2 ppm were found in the cases of 1% zinc treated to both high and medium densities. On the other hand, in the case of 5% copper medium density, a maximum leaching of 90.31 ppm was noted. This was followed by 5% copper high density, 2.5% copper medium and high density, and 1% copper medium and high density.

5.3.3. Subterranean termite test

The study found that copper nanoparticles, treated with 2.5% and 5%, showed no damage after 45 days in termite mounds, while nano-zinc showed slight degradation, with zinc being more susceptible than copper.

1. Pressure treatment

- a. With 10.85% mass loss, medium density wood was found to be more vulnerable to termite attacks than high density wood (4.46%).
- b. The chemical with the least mass loss, copper treated to high density, was found to be 0.49%. The results showed statistical similarity when zinc was treated to medium density (1.96%), copper to medium density (0.99%), and zinc to high density (0.86%). The mass loss percentages for the control group, which did not receive treatment for medium or high density, were higher at 29.61% and 12.02%, respectively.
- c. There was no discernible variation found between the different concentrations. There is little difference in copper concentrations; 5% copper treated at high density exhibits the lowest mass loss percentage of 0.42%. Comparable outcomes were noted for zinc at different concentrations.

2. Coating treatment

- a. Medium density wood lose 11.3% of its mass during the coating treatment, making it more susceptible to termite attacks than high density wood (4.65%).
- b. Among the different nanoparticle, copper that was subjected to a high density showed the least mass loss (0.52%). The statistical results were comparable for copper treated to medium density (1.02%), zinc treated to medium density (2.78%), and zinc treated to high density (1.13%). The control group showed a higher percentage of mass loss (30.37% and 12.30%, respectively), since they were not treated for medium or high density.
- c. No appreciable difference was observed between the various concentrations. The concentrations of copper show little variation; zinc treated at high density

at 5% shows the lowest mass loss percentage (0.43%). Similar results were observed for copper at various concentrations.

5.3.4. Weatherability test

1. Pressure treatment

- a. 3.31% was the lowest mass loss percentage reduction in high density compared to 6.99% in medium density.
- b. Zinc treated to a high density demonstrated a low mass loss reduction (0.68%), comparable to that of copper treated to a high density (0.80%). The maximum mass loss percentages for the control medium density and high were 15.79% and 6.33%, respectively.
- c. Within concentration the mass loss percentage of 0.31% for 5% zinc treated to high density was the lowest, and it was comparable to that of 2.5% zinc high density (0.61), 5% copper high density (0.68%), 5% zinc medium density (0.75%), 2.5% zinc of medium density (0.79%), 1% zinc high density (1.11%), and 5% copper medium density (1.39%). The control group subjected to medium and high densities displayed a maximum mass loss percentage of 15.79 and 6.33%, respectively.
- d. The study found significant greying of wood in medium density wood treated with nano-zinc and copper from week 1 to 6, with moderate greying more effective after week 4. However, no greying effect was observed in 5% nano-zinc and copper treated wood, with copper showing oxidation.

2. Coating treatment

- a. High density coating showed a minimum reduction in mass loss percentage of 2.36% when applied among densities, compared to medium density (5.63%).
- b. Zinc treated to high density showed low mass loss reduction (0.75%), similar to copper treated to high density (0.88%). Maximum mass loss percentages were 11.43% and 5.44%, respectively.
- c. In comparison to 2.5% zinc treated to high density (0.67) and 5% copper treated to high density (0.68%), the mass loss percentage of 0.38% for 5% zinc treated to high density was the lowest. The control group with medium and high densities showed maximum mass loss percentages of 11.43% and 5.44%, respectively. 1% copper (5.05%) and 1% zinc (3.78%) to medium density are the next in treatment order.

- d. The study found significant greying of wood in medium density wood treated with 1% nano-zinc and nano-copper, with moderate greying more effective after week 2. However, no greying effect was observed in 5% nano-zinc and copper treated wood, but copper showed colour change from ochre to blue-green.

5.3.5. Water absorption test

5.3.5.1. Water absorption percentage

1. Pressure treatment
 - a. Compared to medium-density (27.2%), high-density had the lowest water absorption percentage at 15.6%.
 - b. Zinc treated to high density was the nanoparticle with the lowest water absorption among the others (10.1%), followed by copper treated to high density (14.1%) and zinc treated to medium density (19.2%). While the medium densities of copper and control had high water absorption rates of 24.4% and 39.7%, respectively.
 - c. A comparison of different concentrations revealed that 6% of the zinc treated to high density had less absorption than 2.5% of the zinc treated to high density (9.24%) and 5% of the copper treated to high density (11.94%). In contrast, a medium density control group exhibited a high water absorption rate of 39.66%, with 1% copper medium density coming in second at 28.44 percent.
2. Coating treatment
 - a. There was a notable difference in water absorption between the medium and high density coconut wood samples (28.4% and 16.0%, respectively) when coated with different treatments.
 - b. Among the different nanoparticle, copper applied to high density (16.0%) showed the lowest water absorption percentage, followed by zinc treated to high density (10.7%).
 - c. At varying concentrations, the 5% zinc treated to high density showed low absorption of 7.13%, comparable to the 2.5% zinc treated to high density (9.74%) and the 5% copper treated to high density (12.85%). A control group that was subjected to medium density, on the other hand, showed a high water absorption rate of 40.48%. A medium density of 1% copper (27.72%) and 1% zinc (26.2%) came next.

5.3.5.2. Volume swelling percentage

1. Pressure treatment

- a. Among the different densities, high density demonstrated a lower volume swelling percentage (3.11%) than medium density (4.27%).
- b. The highest volume swelling percentages for copper and zinc were 3.0% and 3.21%, respectively. The lowest volume swelling percentages for copper and zinc were 2.92% and 2.94%, respectively. By comparison, the control group with a medium density showed a high volume swelling percentage of 5.49%.
- c. Applying zinc and copper to a medium density resulted in a moderate volume swelling percentage of 2.83 % and 3.11%, respectively, whereas applying zinc and copper to a high density resulted in a low swelling value of 1.9% and 2.6%. By comparison, the control group with a medium density showed a high volume swelling percentage of 5.45%.

2. Coating treatment

- a. In terms of volume swelling percentage, high density performs better than medium density (4.31%), with a low of 3.9%.
- b. The highest volume swelling percentages for copper and zinc were 3.0% and 3.21%, respectively. The lowest volume swelling percentages for copper and zinc were 2.92% and 2.94%, respectively. By comparison, the control group with a medium density showed a high volume swelling percentage of 5.49%.
- c. In comparison to 5% copper high density (2.11%), 2.5% copper high density (2.33%), 2.5% zinc high density (2.44%), 5% zinc medium density (2.46%), and 5% copper medium density (2.56%), the volume swelling percentage of 1.77% for 5% zinc treated to high density was the lowest. The control group, which was exposed to medium density, showed a maximum volume swelling percentage of 5.45%.

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ABSTRACT

**PRESERVATION OF COCONUT WOOD USING INORGANIC
NANOPARTICLES**

by

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ABSTRACT OF THE THESIS

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ABSTRACT

Coconut is a vital crop of Kerala, potential substitute for conventional timber. However, it is susceptible to termites, red palm weevils, bark weevils, shot hole or bark borers, and other pathogens. Traditional wood preservatives like Chromated Copper Arsenate and Chromated Copper Borate pose environmental risks. To address these challenges, researchers are exploring the concept of nanotechnology as a potential solution for wood protection. Nanoparticle has high efficiency to maintain wood natural color, and controlled release of active ingredients at a low concentration, making them a priority for future wood protection applications. As there is limited research on coconut's durability, degradability, and biocide properties of nanoparticles study has been conducted to understand the same.

The copper and zinc nanoparticle was prepared by chemical reduction and precipitation method respectively. Medium and high density coconut wood was impregnated with this nanoparticles at 1%, 2.5% and 5% concentration. Two method of impregnation was carried out i.e., coating with fixative and pressure without fixative. Later, these samples were undergone chemical retention test, leaching test, subterranean termite test, weatherability test and water absorption test according to various standards of ASTM and AWWA. Nested anova was carried out to know the significant difference among various factors and their levels.

Chemical reduction and precipitation method of copper and zinc nanoparticle preparation yielded 65.06% and 70.15% respectively. The SEM image of copper and zinc nanoparticles confirmed to have irregular cuboidal and flake shape with average size of 173.66 nm and 105.44 nm respectively. The presence of copper and zinc in the nanoparticles was verified by EDX analysis. The presence of the corresponding nanoparticles' metal-hydroxyl and metal-oxide groups was verified by the FTIR spectrum. XRD analysis shows that most of the copper in copper nanoparticles is present as copper oxide and cuprous oxide whereas, zinc nanoparticles as zinc oxide.

The chemical retention of both pressure and coating treatment had significantly varied among various density, and nanoparticle concentrations. Among density, medium density had shown higher retention than high density. Concentration of 5% zinc and 5%

copper treated to medium density had shown greater retention among various nanoparticle concentration. The chemical leaching test showed that high leaching of nanoparticles occurred during initial stage. Pressure treated coconut wood with nanoparticles statistically varied between various density, nanoparticle and its concentration. High density had shown low leaching compared to medium density. Among nanoparticle and its concentration, zinc has shown the least leachability when compared to control. Same trend was seen in coating method.

The subterranean termite test (no-choice) shown significant variation for mass loss and visual appearance among various density, and nanoparticles. While there was no significant difference seen with respect to different nanoparticle concentrations. Among density class medium was susceptible to termite attacks. Copper treated sample shown significant resistant to termite damage when compared to zinc treated sample. Statistically significant variation among density, nanoparticle and its concentration was seen in a weatherability test. High density sample was least susceptible to mass loss and greying. Zinc and copper treated sample shown excellent resistant to mass loss and greying as compared to control. Water absorption test shows a remarkable variation among density, nanoparticle and its concentration in the coconut wood treated by pressure and coating. Medium density shown greater water absorption and volume swelling percentage. Among nanoparticles, control shown more water absorption and volume swelling percentage.

Hence, nanoparticles in wood preservation offer improved durability and performance due to their small size and unique properties. They offer enhanced resistance to leaching, termites, and other environmental factors, leading increased lifespan for wood-based products. However, further research is needed to ensure the safe and responsible application of nanoparticle-based wood preservation technologies, while considering cost and scalability.