

**COMPARATIVE ANALYSIS OF ANTIMICROBIAL  
ACTIVITY OF VARIOUS NANOPARTICLE-COATED  
FABRIC: A REVIEW**

By

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A thesis submitted to the Department of Mathematics and Natural Sciences in partial  
fulfillment of the requirements for the degree of  
Master of Science in Biotechnology

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## **Declaration**

It is hereby declared that

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2. The thesis does not contain material previously published or written by a third party, except where this is appropriately cited through full and accurate referencing.
3. The thesis does not contain material which has been accepted, or submitted, for any other degree or diploma at a university or other institution.
4. I have acknowledged all main sources of help.
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## Approval

The thesis/project titled “Comparative Analysis of Antimicrobial Activity of Various Nanoparticle-coated Fabric: A Review” submitted by Tahsin Anjum Farah (21376001) of Summer, 2021 has been accepted as satisfactory in partial fulfillment of the requirement for the degree of Master of Science in Biotechnology on 25<sup>th</sup> January, 2023.

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## **Ethics Statement**

This thesis is a review article covering sources from early 2000s to 2022, and there were no ethical conflicts in any of them. Therefore, this thesis also has no ethical conflicts.

## **Abstract**

Biomedicine, pharmaceuticals, electrical appliances, agriculture, food, biosensors, and many other fields and industries have all been transformed by nanoparticles. Additionally, nanoparticles have successfully demonstrated their capacity to coat textiles with antimicrobial properties. This review examines the antimicrobial properties of textiles treated with nanoparticles of metals and metal oxides, specifically silver, copper oxide, zinc oxide, and titanium oxide, using a variety of different synthesis, modification, and coating techniques. Due to its broad-spectrum antibacterial, antiviral, and antifungal activity, copper oxide nanoparticles have the most promising activity as an impregnating material for textiles, according to descriptive comparative analysis of the antimicrobial efficiency, durability, and economic factors between the four nanoparticles. There is still room for research on the effectiveness of copper oxide nanoparticles as antivirals, particularly against coronaviruses or surrogate coronaviruses, especially given the ongoing COVID-19 pandemic.

**Keywords:** Nanoparticles; silver; copper oxide; zinc oxide; titanium oxide; antimicrobial properties

*Dedicated to my lifeline, my mother,*

*Farhana Haq*

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## List of Acronyms

<b>AgNP</b>	Silver nanoparticle
<b>CuONP</b>	Copper oxide nanoparticle
<b>ZnONP</b>	Zinc oxide nanoparticle
<b>TiO<sub>2</sub>NP</b>	Titanium oxide nanoparticle
<b>CMC</b>	Carboxymethyl chitosan
<b>CS</b>	Chitosan
<b>BR</b>	Bacterial reduction rates
<b>FTIR</b>	Fourier transform infrared spectroscopy
<b>EDX</b>	Energy dispersive X-ray spectroscopy
<b>TEM</b>	Transform electron microscopy
<b>SEM</b>	Scanning electron microscopy
<b>HR-TEM</b>	High resolution transform electron microscopy
<b>XRD</b>	X-ray Diffraction
<b>XPS</b>	X-ray photoelectron spectroscopy
<b>TGA</b>	Thermogravimetric analysis
<b>DLS</b>	Dynamic light scattering
<b>AFM</b>	Atomic force microscopy
<b>MAP</b>	Elemental mapping analysis
<b>K-S1 &amp; K-S3</b>	Knit fabric samples 1&3
<b>UPF</b>	Ultraviolet protection factor
<b>BFE</b>	Bacterial filtration efficacy
<b>ΔP</b>	Differential pressure
<b>SDS</b>	Sodium dodecyl sulphate

<b>CTAB</b>	Cetrimonium bromide
<b>SPE</b>	<i>Salvadora persica</i> extract
<b>HAI</b>	Hospital-acquired infections

# Chapter 1

## Introduction

Since the dawn of time, diseases have been a part of human life. The immune system is what defends us against various foreign substances and builds immunity and memory against those foreign antigens. Self-tolerance is the ability of the body to become tolerant to its own antigens, a condition where the immune system attacks its own antigens occasionally and causes autoimmune reactions that result in autoimmune disorders. Aside from these, there are a much larger number of illnesses that can completely subdue us, such as cancer, cardiovascular conditions, diabetes, neurological issues, and many others. The majority of these are brought on by or made worse by pathogenic microorganisms, such as bacteria, viruses, fungi, etc. The World Health Organization reports that as of 2019 cardiovascular disease was the leading cause of death globally, with cancer coming in second (Cardiovascular Diseases, n.d.).

Infections brought on by pathogenic microorganisms, as opposed to these common diseases, have drawn the attention of scientists, researchers, and of course the general public worldwide. Hospital-acquired infections (HAIs), also known as nosocomial infections, are the most severe and common of these infections. Nosocomial infections, also known as HAIs, are those that develop after a patient is admitted to the hospital but before 48 hours have passed or 30 days have passed since discharge (Revelas, 2012). HAIs are one of the top 10 causes of death in the USA, occurring in about 4% of all hospitalized patients (Health Care-Associated Infections | Agency for Healthcare Research and Quality, n.d.). A wide variety of bacteria, viruses, and fungi can spread them. Numerous of these organisms are a part of the commensal flora that coexists in harmony with the human body naturally (Singh et al., 2012). In a hospital setting, they spread primarily through the patient-used textiles, such as bedsheets, towels, scrubs, and even the curtains. Because of this, it's crucial that these specific fabrics have some antimicrobial

activity in order to fight off these dangerous microorganisms and safeguard patients with immunodeficiencies, transplants, and other conditions (Tinker, 2010).

Numerous microorganisms are quickly developing a resistance to antibiotics and other antimicrobial agents (Perelshtein et al., 2016). In a hospital setting, surfaces like tables, counters, bedrails, the hands of nurses and doctors, etc. are frequently disinfected with alcohol-based cleaners. As a result, the hospital's textiles act as a breeding ground for these pathogenic microorganisms (Holt et al., 2018). A lot of work has gone into developing textiles with antimicrobial properties with the growing concerns about health and hygiene in mind. El-Nahhal et al. reported that new methods are being considered where metals or metal oxides are used to give the textiles desired antimicrobial properties. (El-Nahhal et al., 2020). In addition to protecting the wearer from pathogenic microbes, antimicrobial textiles also work to stop the spread of pathogenic microorganisms, the formation of odors and discolorations, and the degradation of textiles caused by the growth of mold, mildew, and other microorganisms (Ferrero & Periolatto, 2012; Holt et al., 2018). Due to their many benefits, including their biodegradability, moisture absorption, breathability, comfort for the skin, and sweat absorption, cotton-based textiles are the most commonly used worldwide, particularly in the medical and healthcare sector (Shaheen & Abd El Aty, 2018). According to Marković et al. (2018), it is also frequently used as gauge materials for dressing wounds. A natural fiber made of cellulose with 1,4-Dglucopyranose as its repeating unit is cotton (Shahidi, 2016). However, cotton fiber on its own has some drawbacks, including a lack of strength, flammability, low UV protection, and a high degree of hydrophilicity (Dhineshabu & Rajendran, 2016). Due to its large surface area and capacity to hold moisture, cotton is also a result of these qualities and is a very good surface for the growth of pathogenic microorganisms (El-Nahhal et al., 2017). Therefore, over the past decade and more, nanotechnology has garnered a lot of attention and demonstrated some impressive results in order to counteract these negative aspects. To give cotton fabrics

antimicrobial activity, inorganic metal and metal oxide nanoparticles have been used to coat the fabric's surface. Inorganic metals and metal oxides are frequently used due to their selective toxicity towards microbes, high stability, and large surface area to volume ratio that increases their efficacy (Rajendran et al., 2013). This review's later sections will go into more detail about the nanoparticles' specific microorganism-targeting mechanisms.

The purpose of this review is to compare the various types of modification processes that are used to create nanoparticles of metals and metal oxides, specifically silver, copper oxide, zinc oxide, and titanium oxide, in terms of their functionalization on cotton fabrics, the effectiveness and durability of their antimicrobial properties, as well as keeping in mind their environmental impacts and economic viability.

## **Chapter 2**

### **Nanobiotechnology and nanoparticles in textile industry**

Nanobiotechnology, as defined by the peer-reviewed journal Nature Research, is "a discipline in which tools from nanotechnology are developed and applied to study biological phenomena, primarily through the use of functional nanoparticles for our own desired purpose.". Over the past ten years and more, the textile industry has used nanobiotechnology extensively to create nanoparticles with antimicrobial properties that can be coated onto various textiles. To do this, a variety of methods including physical modification, chemical modification, and biological synthesis are employed. According to Rajendran et al., this technology can produce modified fabrics with high durability and stability (Rajendran et al., 2013).

Due in large part to the fascinating characteristics of electrical conductivity, magnetism, catalysis, and other properties of nanoparticles, research into this area has exploded in recent years. (Maghimaa & Alharbi, 2020). Inorganic metal oxides, which are nanoparticles, have drawn a lot of attention due to their antimicrobial activity, selective toxicity, high surface area



to volume ratio, tolerance to heat, and capacity to withstand rigorous processing procedures without being harmed or losing integrity (Fu et al., 2005). Biomedicine, agriculture, food production, cosmetics, and textile manufacturing are just a few of the industries that use nanoparticles (Sathiyavimal et al., 2018), as well as in the photocatalytic industry, the pharmaceutical industry as drug delivery and drug vehicles, and biosensors, etc. In (Vasantharaj et al., 2019). Metal or metal oxide nanoparticles are more active and efficient than non-metal ones, according to reports, though both metal and non-metal nanoparticles are used. (Vasantharaj et al., 2019).

Nanoparticle use offers a variety of advantages. Nanoparticles exhibit increased activity as a result of their high surface energy and high surface area to volume ratio (Rajendran et al., 2013; Singh et al., 2012), as well as their adaptability to physical, chemical, or biological manipulation. Silver nanoparticles (AgNPs), copper/copper oxide nanoparticles (Cu/CuO NPs), zinc oxide nanoparticles (ZnO NPs), and titanium oxide nanoparticles (TiO<sub>2</sub> NPs) are the most frequently used nanoparticles with the aim of introducing antimicrobial activity into textiles. Each of these has undergone extensive research to improve, among other things, the antimicrobial effects and durability of cotton textiles in particular.

## Chapter 3

### Silver Nanoparticles (AgNPs)

According to Dastjerdi and Montazer (2010), silver has been the subject of the most in-depth research since the dawn of time. Due to its electrical and thermal conductivity, silver is used in a wide range of industries, including the production of coins and in the fields of medicine, electronics, household products, toiletries, jewelry, and electrical appliances (Salleh et al., 2020; Windler et al., 2013). Despite all of its uses, silver's antimicrobial properties are perhaps its most well-known. Due to this important property, silver can be transformed into silver nanoparticles for use in a variety of biomedical and healthcare fields, including the production of patient scrubs, privacy drapes, doctor and nurse scrubs for use in hospitals, protective clothing, and personal clothing. Although studies have shown that silver's ionic form, Ag, is what gives it its antimicrobial properties, the precise mechanism of silver's action is still unknown. (Salleh et al., 2020; Windler et al., 2013).

AgNPs' size, shape, morphology, chemical makeup, particle distribution, and reducing agents used in its synthesis all have a significant impact on how biologically active they are. In the study published by Salleh et al. (2020), AgNPs perform better than their bulk equivalents because their surface area is increased by their smaller size, which makes it easier for the particles to interact with microbial membranes and harm them. AgNPs function by a variety of bacteriocidal mechanisms, some of which involve attachment to cell wall and cell membrane lipids and proteins and disruption of the integrity of the cell wall, resulting in loss of cell contents and ultimately cell lysis (El-Rafie et al., 2014). Additionally, it works by generating reactive oxygen species (ROS), such as superoxide ( $SO_2$ ), hydrogen peroxide ( $H_2O_2$ ), and others. These ROS can interfere with the pathways for making DNA and proteins, as well as with cell metabolism, all of which lead to the microorganism's eventual demise (Ibrahim & Hassan, 2016). AgNPs are effective against both Gram positive and Gram negative bacteria, as

well as some fungi and viruses, according to the literature review that was done. *Staphylococcus aureus* is the most frequently tested bacteria for Gram positive bacteria, and *E. coli* is the standard organism for Gram negative bacteria. In nearly all studies, modified AgNPs demonstrated above 90% antibacterial efficacy for both types of bacteria. (Ibrahim & Hassan, 2016; Qasim et al., 2018; Salleh et al., 2020; Xu et al., 2018, 2019; D. Zhang et al., 2013). Other than the aforementioned two, experiments have been carried out in which AgNPs demonstrated excellent antibacterial activity against *P. aeruginosa*, *C. albicans* and *A. niger* are two common fungi that are susceptible to AgNPs' antifungal effect (Maghimaa & Alharbi, 2020; Shaheen & Abd El Aty, 2018). Last but not least, there has been little research done on the antiviral effect of AgNPs (Salleh et al., 2020), but the scant research to date has demonstrated that AgNPs have antiviral effects against rotavirus. In addition, some theories contend that AgNPs may be helpful in the management of COVID19 because they can bind the viral spikes and obstruct the virus' attachment to and entry into host cells (Salleh et al., 2020)

Although studies have shown that AgNPs are not toxic to human skin, they do have a high level of selective toxicity towards microorganisms. (Chatha et al., 2019). According to a literature review, silver has antimicrobial activity against over 650 microorganisms even at low concentrations, but not on human skin (Dastjerdi & Montazer, 2010; El-Rafie et al., 2014). These molecules can range from biopolymer-based substances like carboxymethyl chitosan to a wide variety of other molecules (Xu et al., 2018, 2019), to a few artificial polymers (Qasim et al., 2018; D. Zhang et al., 2013).

### **3.1 Synthesis and modification processes**

Silver-containing compounds can be converted into silver nanoparticles (AgNPs) using a number of modification techniques. Generally speaking, these include sol-gel coating,

sonochemical method, photocatalytic reduction, chemical reduction, and pad-dry-cure method (Dastjerdi & Montazer, 2010; Maghimaa & Alharbi, 2020). According to Salleh et al., (2020) these techniques include physical, chemical, and biological synthesis. Methods for physical synthesis include laser ablation and vapor condensation. In most cases, reducing agents and other reagents are used in chemical modification processes to reduce silver-based compounds (Song et al., 2009; Yu, 2007) However, there are some drawbacks and issues with these physical and chemical methods, such as the weak coating of the AgNPs, which reduces durability and results in the release of hazardous chemicals into the environment. Additionally, these techniques require a lot of energy. With all of these considerations in mind, biological synthesis of AgNPs has been rapidly gaining the confidence and attention of researchers across the globe due to their low energy consumption, biodegradability, biocompatibility, decreased use of hazardous chemicals, and cost-effectiveness (Salleh et al., 2020). *Curcuma longa* leaf extracts, carboxymethyl chitosan, and other materials are used in biological synthesis. ((Maghimaa & Alharbi, 2020; Xu et al., 2018, 2019). This review will provide a summary of the modification techniques currently in use and attempt to compare them in order to identify the techniques that are most suitable in terms of effectiveness, dependability, environmental friendliness, and cost-effectiveness. The biological synthesis and modification of AgNPs, which are then functionalized onto textile fabrics to give them antimicrobial properties, are the main topics of this section.

### **3.1.1 *Biological synthesis/Green synthesis of AgNPs***

Because it offers a more environmentally friendly approach by substituting the use of toxic and hazardous chemicals with biological extracts from plants, fungi, etc., biological synthesis of silver nanoparticles has been more quickly adopted by researchers worldwide. (Maghimaa & Alharbi, 2020; Rajaboopathi & Thambidurai, 2018; Salleh et al., 2020; Shaheen & Abd El Aty, 2018). This particular method has acquired the moniker "green synthesis" due to this.

As active phyto-compounds can serve as both the reducing agent and the capping agent in the synthesis of nanoparticles using plant extracts or fungal extracts, green synthesis has gained attention on a global scale over time (Maghimaa & Alharbi, 2020). This section will discuss a few of the green synthesis methods, their outcomes, and the antimicrobial potency of each of the resulting AgNPs.

### *3.1.1.1 Myco-synthesis of AgNPs*

The sub-title itself makes it clear that the topic of this section will be the synthesis and modification of AgNPs using fungi and fungal filtrates. This particular method has a number of benefits, such as the fact that it is clean and environmentally friendly, that it does not call for the use of additional reducers and stabilisers, and that it also has the added benefit of allowing the experiment to be carried out at room temperature (Ibrahim & Hassan, 2016). Fungi are a great option for this method because they can produce a lot of the desired product, including a lot of enzymes and proteins, and they are also tolerant of harsh growth conditions (Shaheen & Abd El Aty, 2018). The use of fungal filtrates for AgNP synthesis has been the subject of numerous studies. Three of those studies will be discussed in brief here.

- (a) **Using fungus *Alternaria alternata*:** In this study, *Ibrahim & Hassan (2016)* characterized and showed how to synthesize modified AgNPs with improved antimicrobial activity and durability. This approach shows to be very safe, secure, and cost-effective. In this study, silver nitrate was first prepared, and then it was mixed with the prepared fungal filtrate to produce AgNPs by reducing the silver nitrate to silver atoms. SEM, TEM, DLS, and energy dispersive X-ray spectroscopy (EDX) were used to characterize the synthesized AgNPs. The combined results of all these analyses revealed the morphology of the synthesized AgNPs, which were then coated onto cotton fabrics and identified using thermogravimetric analysis (TGA) and scanning electron

microscopy (SEM). These findings also demonstrated that the cotton fabrics had been successfully coated with AgNPs. Next, both qualitative and quantitative tests were performed to determine the treated cotton fabrics' antimicrobial activity. According to these findings, treated fabrics exhibited a 99.9% inhibition of *E. coli* and *S. aureus* growth at a concentration of 1 mM AgNP, and at 5 mM AgNP, bacterial growth was completely stopped. To see if this AgNP coating could be used to shield the coated cotton fabric from microbial deterioration, soil burial tests were also conducted. Of course, the coated fabric's color remained the same while the uncoated fabric developed a brown hue. Gamma radiation and thermal curing were the two curing techniques used in the durability tests. The thermally-cured fabric still had 99 % and 98 % antimicrobial activity after 20 washes, whereas the gamma-cured fabric had 99 % and 98 % antimicrobial activity against *E. coli* and *S. aureus*, respectively. Thereby, the results of study conducted by Ibrahim & Hassan (2016) show that myco-synthesis has a positive influence over the antimicrobial activity and durability of the synthesized AgNPs.

(b) **In-situ myco-synthesis of AgNPs:** According to the literature review, this study was the first to choose to create AgNPs in-situ from fungal filtrates because of their advantages in terms of stability and toughness. Although ex-situ synthesis is frequently used, it has the potential to lead to AgNP aggregation (Shaheen & Abd El Aty, 2018). The silver ions were converted into AgNPs in this study using five different fungal strains that were extracted from medicinal plants. These fungi were *Alternaria arborescens* (1), *Alternaria alternate* (2), *Alternaria brassicae* (3), *Nigrospora oryzae* (4), and *Penicillium crustosum* (5). The silver nitrate solution containing biomass was inoculated onto the cotton fabrics. A color change into brown signaled the deposition of AgNPs. FTIR and EDX were used to characterize this incorporation, and the results

showed that the AgNPs had been successfully deposited. Using the organisms *E. coli*, *C. albicans*, and *A. niger*, the antimicrobial activity of this modified fabric was examined using the agar diffusion method. The AgNPs produced using microbial strains 1 demonstrated the highest activity. This might be explained by the fact that the strains had various release rates, resulting in various zones of inhibition with various sizes. Due to the in-situ AgNPs' improved interaction and attachment with the cotton fibers, durability tests have revealed that they outlasted ex-situ synthesized AgNPs.

### *3.1.1.2 Using seaweed extract*

The three types of seaweeds—green, red, and brown algae—are distinguished by the amount of alginic acid they contain, which has powerful stabilizing and reducing effects. According to Rajaboopathi & Thambidurai (2018), this can be used to convert silver nitrate into AgNPs, which are then coated onto cotton fabrics using the pad-dry-cure method to improve their antimicrobial properties. As a crosslinking agent, citric acid was utilized. FTIR, SEM, TEM, EDX, and atomic force microscopy (AFM) were used to characterize the successful synthesis and coating. Collectively, these demonstrated the successful incorporation of AgNPs onto cotton fabrics, which were subsequently put through antimicrobial and durability tests. The AgNP-coated fabric demonstrated superior UV protection factor (UPF) to the uncoated one as well as superior antibacterial activity against *S. aureus* in comparison to *E. coli*. Even though the fabrics showed slight morphological changes, durability tests after 10 washings somehow revealed better UPF and antibacterial activity than before washing. This study has once more demonstrated the advantages of producing AgNPs in a green manner.

### *3.1.1.3. Using Curcuma longa leaf extracts*

Since ancient times, medicinal plants and leaf extracts have been used extensively for therapeutic purposes. Turmeric sp. has been widely utilized for their pharmaceutical and

medicinal benefits, as well as for the treatment of respiratory illnesses, digestive disorders, and a variety of other diseases (Maghimaa & Alharbi, 2020). Therefore, it was proposed that leaf extracts could also be used to synthesize and functionalize AgNPs onto textiles, enhancing their antimicrobial action. Maghimaa & Alharbi, (2020) carried out this investigation. The synthesised AgNPs were characterized using HR-TEM and FTIR, then coated onto the fabric, which was examined and verified using SEM and EDX, just like any other process. Human pathogenic organisms like *S. aureus*, *P. aeruginosa*, *S. pyogenes*, and *C. albicans* were used to test the coated fabric's antimicrobial activity. The effectiveness of the coated fabric's ability to heal wounds was also evaluated using fibroblast L929 cells. Antimicrobial tests using various doses of the extract revealed high activity against all of the tested microorganisms, with *P. aeruginosa* displaying the highest activity in comparison to the others. The created AgNPs also demonstrated low to no cytotoxicity toward the L929 cells, demonstrating their safety for use while also possessing potential for both antimicrobial and wound-healing properties.

### **3.2 Summarising AgNPs**

When taken as a whole, the various AgNP synthesis techniques, their characterization, and antimicrobial activity and durability efficacy can be time-consuming and complicated. When compared to physical methods, chemical synthesis has some benefits, including a higher yield of product in less time and lower production costs (Salleh et al., 2020). Despite the favorable outcomes, this method is less eco-friendly due to the extensive use of chemicals that can be toxic and dangerous for both humans and animals (Gasaymeh et al., 2010). Genotoxicity, carcinogenicity, cytotoxicity, and general toxicity are additional risks (Ibrahim & Hassan, 2016). Due to chemical overexposure, chemical synthesis can also result in contamination of the synthesized nanoparticles. In the end, when all factors are taken into account, it has repeatedly been shown that biological nanoparticle synthesis offers far more benefits than any



alternative technique. This is due to the fact that this method avoids the use of potentially toxic chemicals by using natural extracts from sources like plants and fungi. The textiles treated using this method also have improved biodegradability, biocompatibility, stability, and antimicrobial properties, in addition to receiving any additional benefits like UV protection. Because of this, the following **Table 1** will assist in giving a quick overview of each of these components.

Synthesis method		Antimicrobial activity		Durability tests	Additional properties	References
		<i>E.coli</i> (%)	<i>S.aureus</i> (%)			
<b>Biological</b>	Using <i>Alternaria alternata</i>	99.9 (1Mm) 100 (5Mm)	99.9 (1Mm) 100 (5Mm)	20w; >98%	(N/A)	(Ibrahim & Hassan, 2016)
	In-situ myco-synthesis	(good)	(good)	In-situ>ex-situ	(N/A)	(Shaheen & Abd El Aty, 2018)
	Seaweed extract	(relatively less)	(more)	10w; activity increased	UV protection	(Rajaboopathi & Thambidurai, 2018)
	<i>C.longa</i> leaf extracts	Relatively less	(relatively less)	(N/A)	Wound-healing	(Maghimaa & Alharbi, 2020)

**Table 4 Various modification processes of AgNPs, along with their antimicrobial efficiency and durability.**

(N/A indicates no usable data recorded). Abbreviations used: CMC; Carboxymethyl chitosan; HBP-NH<sub>2</sub>; Amino-terminated hyperbranched polymer; p-NIPAM; Poly-N-Isopropylacrylamide; *C.longa*; *Curcuma longa* (highest activity against *Pseudomonas aeruginosa*); 50w; 50 weeks.

## Chapter 4

### Copper or copper oxide nanoparticles

Copper and copper oxide are used as nanoparticles at least as frequently as silver. Multiple studies have already established that metals and metal oxides are more potent, stable, and useful as nanoparticles than non-metals (Hameed et al., 2016). Silver is one of the metals that is used as nanoparticles the most frequently, but there are drawbacks to using silver. These include the primary fact that silver is quite brittle in the air, so risks of the silver oxidizing and turning brown persist if not properly protected or covered, such as with encapsulation. According to Hamdan et al., prolonged exposure to the silver can also result in skin discoloration and the resurgence of microorganisms that are resistant to it. (Hamdan et al., 2017). Due to their widespread and wide applicability in a number of interdisciplinary fields, copper is the material of choice for the majority of researchers. Copper is less expensive, more miscible with any polymer, and more stable than silver (Sathiyavimal et al., 2018).

Due to its many advantageous qualities, copper is one of the most commonly used nanoparticles (Almasi et al., 2018; Borkow & Gabbay, 2004, 2005; Sathiyavimal et al., 2018). Since ancient times, copper has been used as an antimicrobial agent due to its well-known broad-spectrum antimicrobial activity (Borkow & Gabbay, 2004). As a disinfectant, bactericide, fungicide, molluscicide, nematocide, anti-fouling agent, and water-purifier, it also serves an antimicrobial purpose (Borkow & Gabbay, 2005). Because it has long been used as a contraceptive in intrauterine devices (IUDs), dentistry, and the prevention of foodborne illnesses, copper has also proven to be beneficial for humans (Borkow & Gabbay, 2005; Lazary et al., 2014).

In addition to being plentiful and inexpensive, copper-based nanoparticles have superior antimicrobial activity to silver (Marković et al., 2018). The antiviral and antifungal effects of this substance are also widespread. Copper kills microorganisms through a variety of different,

variable mechanisms. As bacteria cannot develop a resistance to copper, this very property makes copper even more desirable. Although antibiotic resistance can develop in microbes very quickly, copper resistance in microbes is a very uncommon occurrence. Antibiotic- and antiviral-resistant microorganisms have been successfully combated by copper (Lazary et al., 2014). According to Humphreys (2014), copper's antimicrobial activity increases with copper concentration. According to Lazary et al. (2014), copper oxide nanoparticles have a higher durability and, even if they do wash off into water, they won't harm the water because they are insoluble. The proposed mechanism of action of copper killing microbes includes inhibition of DNA and protein synthesis in microbes, along with disruption of cell wall and cell membrane components, leading to leakage of cell contents and cell lysis. According to Araújo et al. (2018), copper can render bacteria inactive, especially *L. monocytogenes*, *S. typhimurium*, *S. enterica*, and *C. jejuni*. Viruses such as the avian influenza virus (H9N2), the human influenza virus (H1N1), the poliovirus, the bronchitis virus, the HSV, and HIV (Borkow et al., 2010). *Tinea pedis*, a foot fungal infection that is common in diabetic patients, is also treated with it (Borkow & Gabbay, 2005).

Copper is extremely toxic to microbes but has little to no effect on human cells, according to reports (Borkow & Gabbay, 2005). The use of hospital sheets impregnated with copper lining for 300 nights, adult diapers lined with copper for six months, and other studies have demonstrated that copper is not sensitive to human skin. None of these demonstrated any allergic reactions in people (Lazary et al., 2014). Additionally, copper has been used to treat allergic rhinitis and mite allergies (Borkow & Gabbay, 2005). Copper nanoparticles (CuNPs), like silver nanoparticles, perform better than their bulk counterparts due to their high surface area, making it easier for them to interact with microbial cell membranes (Araújo et al., 2018).

## **4.1 Synthesis and modification processes**

Araújo et al. (2018) claim that a variety of physical and chemical techniques can be used to create copper nanoparticles. The physical techniques could be high energy mechanical milling, vapor deposition, pulsed laser ablation, etc. Microemulsion, sonochemical/electrochemical reduction, microwave or ultrasound irradiation (for better coating), and hydrothermal synthesis are a few examples of chemical processes. CuNPs' biological synthesis has also been the subject of studies. The work that has been done by scientists around the world to create and impregnate textile materials with CuNPs using a variety of novel processes to improve the antimicrobial action of the modified textiles is examined in the section that follows.

### **4.1.1 Hybrids of CuNP/CuONP and chitosan molecules**

Chitosan (CS) is well known for its biodegradability, biocompatibility, non-toxicity, stability, and most importantly, its inherent antimicrobial properties (Costa et al., 2018). According to Shahid-ul-Islam & Butola (2019), chitosan is a carbohydrate polymer and an N-acetylated form of chitin. The second most prevalent polysaccharide on earth, after cellulose, is chitosan (Sutirman et al., 2019) It is widely used as an antioxidant, an emulsifier, and a thickener in the biotechnology, therapeutics, food, and beverage industries (Rinaudo, 2006).

There has already been a significant amount of research on the use of chitosan and modified forms of chitosan as antimicrobial coatings on cotton, nonwovens, and linen fabrics (Chatha et al., 2019; Klaykruayat et al., 2010). Furthermore, chitosan and CuNPs have been combined to create a synergistic antimicrobial effect. In order to achieve this, a mixture of sonochemically created CuONPs and colloidal chitosan was used to create the hybrid, which was then coated onto cotton fabrics using the sonication method and then dried using the pad-dry-cure method. Then, using SEM, FTIR, and XRD, this was evaluated and verified; each of these demonstrated

successful coating. Using the zone of inhibition method to compare the antimicrobial activity of coated and uncoated fabrics against *S. aureus* and *E. coli*, it was found that the coated fabrics displayed clear zones of inhibition while the uncoated fabrics did not. Conversely, the hybrid CS-CuONP-coated fabrics displayed greater activity than the ones coated solely with chitosan by creating larger clear zones. Reactive oxygen species (ROS), which have negative effects on the test microorganisms, are thought to be produced by the copper, which is why this is. In light of this, the CS-CuONP hybrid's synergistic antimicrobial effect is demonstrated.

Almasi et al. (2018) carried out a related study as well where the antimicrobial effects of CuONPs after being impregnated onto organic-inorganic nanohybrids of bacterial cellulose nanofibers (BCNF) and chitosan nanofibers (CSNF) were evaluated. Araújo et al. (2018) conducted a study in which CuONP was used to impregnate bacterial cellulose. They created the CuONPs hydrothermally, added them to the BC fibers, and evaluated their antimicrobial activity after that. They also showed that the CuONP-BC fabrics had clear zones for every strain of *Enterococcus sp.*, which is a Gram-positive bacterium. and *S.epidermidis* were examined, as well as the yeast *C. albicans*, Gram-negative *E. coli*, *S.enteritidis*, and *P. aeruginosa*.

Almasi et al. (2018) reported this by FTIR, XRD, and SEM analyses of the modified nanofibers showing successful CuONP impregnation. When antimicrobial activity for both was compared, it was discovered that the CuO-BCNF displayed less activity than the combined CuO-CSNF antibacterial effect. The abundant hydroxyl groups on CS's backbone allowed the CuO to cling to it more tightly, as can be seen from the SEM micrographs, whereas BC is quite hydrophilic and only weakly binds to the CuO, making it easy to release. Thus, the activity is diminished. (Almasi et al., 2018; Dhineshababu & Rajendran, 2016). Although the precise mechanism of CuONP in this instance is unknown, it is believed to involve the production of ROS, interaction with, and disruption of the microbial cell wall and cell membrane, which results in cell lysis.

Due to the thick cell walls of Gram negative bacteria, which give them greater resistance to the copper attack, they have also shown that Gram positive *L. monocytogenes* are more susceptible to CuONPs than Gram negative bacteria.

#### **4.1.2 Biological synthesis of CuONPs**

Plant extracts from *Sida acuta* have been used to successfully create copper nanoparticles. (Sathiyavimal et al., 2018). For its use as an antiplasmodial, antimicrobial, and antioxidant agent, *Sida acuta* is well-known. Flavonoids, phenolics, steroids, alkaloids, and other potent active molecules are among those found in it. which can be utilized for better coating cotton fabrics with synthesized CuONPs. By using UV-vis, SEM, TEM, and FTIR, the successful coating was identified and verified. Disk diffusion was used to test the CuONPs' antimicrobial activity against *E. coli* and *S. aureus*, and the results revealed that the CuONPs were more active against *E. coli* than *S. aureus*. Cell wall disintegration and the inhibition of membrane-bound cytochromes are believed to be the mode of action in this case as well. In a related study by Vasantharaj et al. (2019), CuONPs were produced biologically. The synthesized CuONPs demonstrated a greater bactericidal effect against *S. aureus*, followed by *E. coli* and *K. pneumoniae*. Since there isn't a single set mechanism for copper's bactericidal activity, this variation in activity may be the result of different unidentified antimicrobial mechanisms.

Sathiyavimal et al. (2018) also showed that while Cu-coated nylon exhibited bactericidal activity, Cu-coated polyester exhibited a combination of antibacterial, antifungal, and antiviral activity. Depending on their size, shape, and synthesis methods, CuONPs can also exhibit photocatalytic activity. According to this study, the photocatalytic activity of the biologically produced CuONPs against the dyes crystal violet and methylene red was about 93% and 87%, respectively. The CuONPs produced Vasantharaj et al. (2019). also demonstrated comparable photocatalytic activity, where the nanoparticles demonstrated the gradual

decomposition of the crystal violet dye, which was supported by the disappearance of a particular peak at 586 nm after prolonged exposure to sunlight.

#### **4.1.3. Role of CuO-impregnated linens in reducing HAIs**

It was investigated whether switching to copper oxide-impregnated hospital bedding could aid in lowering HAIs while keeping in mind the beneficial qualities of copper. Lazary et al. (2014) studied for a year, which is a lengthy period of time. In this regard, results from 2014 were encouraging. The study was conducted in a long-term care unit for brain injuries. After just 6-7 hours, results showed a significant 50 % and 43 % reduction in both the Gram positive and Gram negative bacterial loads, respectively. Additionally, the duration of antibiotic use, the frequency of gastrointestinal and eye infections, and the number of days that patients had fever have all decreased thanks to the use of CuO-impregnated linens. Therefore, there is still some hope that using CuONPs or linens impregnated with CuO will help to at least try and control HAIs.

#### **4.2 Antiviral activity of copper**

Because it is effective against a wide range of both enveloped and non-enveloped viruses, copper is particularly well known for its antiviral activity. Numerous studies highlighting copper's antiviral properties have already been conducted (Borkow et al., 2010; Borkow & Gabbay, 2004, 2005). The respiratory openings act as the primary portals of entry into the human body for a variety of diseases caused by airborne bacteria and viruses. This information was used to create respiratory masks and nonwoven surgical gowns that were impregnated with copper or copper oxide. Additionally, they are very helpful in attempting to stop the spread of nosocomial infections (Borkow et al., 2010; Borkow & Gabbay, 2004, 2005; Lazary et al., 2014).

Borkow & Gabbay (2004) demonstrated that hospital textiles impregnated with copper can assist in limiting nosocomial infections. Athletes' foot caused by the fungus *Tinea pedis*, which is common in diabetic patients, can be treated and prevented with copper-impregnated mattresses and bedding. Copper can also be used in socks to prevent and treat athlete's foot and shield the toes from further bacterial invasion. The production of ROS by the Cu and Cu<sup>2</sup> ions, which interfere with viral activity and cause viral death, is a major factor in the inactivation of viruses. Because they are primarily host-dependent and lack defense mechanisms like DNA repairing, protective barriers, etc., viruses are particularly more sensitive to copper. like other microorganisms and bacteria.

Without affecting the masks' ability to filter out particles, a novel anti-influenza mask was created that was lined with copper oxide in several layers (Borkow et al., 2010). In comparison to the control masks, which produced less effective results, their findings demonstrated excellent virus inactivation by the CuO-impregnated masks within 30 minutes. This was caused by the H1N1 human influenza A virus and the H9N2 avian influenza virus, both of which were tested, being inactivated by direct contact. The CuO-impregnated masks had successfully passed all of the required tests before being approved for use, including the bacterial filtration efficacy (BFE), differential pressure (P), latex particle challenge, and resistance to synthetic blood tests. The last one is essential because patients, nurses, and medical personnel can all be protected from blood spills by wearing these masks. Even after a continuous 30 minutes of exposure, the masks still demonstrated complete resistance to blood penetration. Along with the fact that increasing the copper concentration in the masks up to 6 times still did not show any effects on human skin sensitization, as confirmed by animal skin test results, electron microscopy had also revealed morphological abnormalities in the test viruses. .

Imai et al. (2012) had also incorporated the use of copper-zeolite textile in the defense against H5N1 and H5N3 avian influenza viruses. Given that zeolite has a negative surface charge, Cu<sup>2</sup>



ions were used in this instance to modify cotton textiles. According to Imai et al. (2012), zeolite is a "microporous, aluminosilicate mineral with adsorption capabilities.". However, earlier research using Cu<sup>2+</sup> from CuCl<sub>2</sub> showed no effect upon these H5N1 viruses even after 6 hours of exposure. The results indicated that the two different strains of H5N1 tested responded differently to the Cu-zeolite coated textiles. Therefore, the Cu<sup>2+</sup> ions held by zeolite and those in CuCl<sub>2</sub> exhibit various antiviral activities; however, the same concentration of CuCl<sub>2</sub> had completely inactivated the H5N3 viruses. As a result, copper has been employed widely as a powerful antiviral agent. More research is being done to determine the effects of copper as a nematocide, molluscicide, fungicide, algacide, and more. (Borkow & Gabbay, 2005; Imai et al., 2012). In another study conducted by Jampa S. et al. in 2022, copper oxide was incorporated with zeolite nanoparticles to make Cu-Z. These fusion nanoparticle-coated fabrics showed better thermal stability compared to CuO NPs. These modified fabrics also showed higher antiviral activity as well as could deactivate *E.coli* and *S.aureus* efficiently. Moreover, the antibacterial activity of CuONP-coated textile fabrics was further analysed by Roman et al (2020) and they found promising positive results against several Gram positive and Gram negative bacteria.

The antiviral properties of copper could be very helpful given the current global pandemic caused by SARS-CoV-2. Therefore, more research in this area is required.

### **4.3 Summarising CuONPs**

There is no question, in light of the literature review and the aforementioned details, that copper or copper oxide nanoparticles can be used successfully as broad spectrum antibacterial, antifungal, and antiviral agents to lower the incidence of HAIs, as well as in respiratory masks, gloves, socks, and other medical necessities. **Table 2** provides an overview of all these findings.

Modification process	Antibacterial activity		Antiviral activity	Antifungal activity	Additional properties	References
	Gram +ve	Gram -ve				
Hybrids with chitosan	<i>S.aureus</i>	<i>E.coli</i>	(N/A)	(N/A)	(N/A)	(Dhineshabu & Rajendran, 2016)
CuO-BCNF and CuO-CSNF	<i>S.aureus</i> , <i>Enterococcus</i> sp., <i>S.epidermidis</i>	<i>E.coli</i> , <i>P.aeruginosa</i> , <i>S.enteritidis</i>	(N/A)	<i>C.albicans</i>	(N/A)	(Almasi et al., 2018; Araújo et al., 2018)
CuONP from <i>Sida acuta</i> extract	<i>S.aureus</i>	<i>E.coli</i>	(N/A)	(N/A)	Photocatalytic activity, UV protection, dye degradation (93% for CV; 87% for MR)	(Sathiyavimal et al., 2018)
CuO-impregnated linens, hospital textiles, masks	(N/A)	(N/A)	Human influenza A (H1N1), Avian influenza (H9N2)	<i>Tinea pedis</i>	(N/A)	(Borkow et al., 2010; Borkow & Gabbay, 2004; Lazary et al., 2014)
Cu-Zeolite cotton	(N/A)	(N/A)	Different strains of H5N1, H5N3.	(N/A)	(N/A)	(Imai et al., 2012)

**Table 76 Different modifications and combinations of CuONPs with other compounds and their diverse antimicrobial and additional properties.** (N/A indicates no usable data recorded). Abbreviations used: CuO-BCNF & CuO-CSNF; Copper-oxide impregnated bacterial cellulose nanofibers and chitosan nanofibers; CV; Crystal violet; MR; Methyl red; HAIs; Hospital-acquired infections; GI infection; Gastrointestinal infection; H5N1, H5N3; Two different subtypes of Human influenza A virus. The CuO-impregnated linens also reduced HAIs, fever days, antibiotic consumption, GI infection, eye infection.

## Chapter 5

### Zinc/zinc oxide nanoparticles (ZnONPs)

Nanoparticles made of zinc oxide are just as useful, if not more, than the silver and copper nanoparticles discussed above. Drug delivery, tumor cell disintegration, piezoelectric devices, semiconductors, transparent electronics, personal care items, gas sensors, UV-protective cosmetics, paints, and medical textiles are just a few of the many applications for zinc oxide that have already been developed (Dastjerdi & Montazer, 2010; Fouda et al., 2018; Rajiv et al., 2013; Windler et al., 2013). Holt et al. (2018) have noted that zinc oxide nanoparticles can withstand demanding commercial drying processes. The wide range of properties of zinc oxide, including its piezoelectric, pyroelectric, photocatalytic, semiconducting, durability, stability, and biocompatibility, are responsible for all of these. (Rajiv et al., 2013; Silva et al., 2019).

Previous research has shown zinc oxide to have broad-spectrum antibacterial and antifungal properties. This allowed for the incorporation of zinc oxide nanoparticles into textiles to enhance their antimicrobial properties. The smaller size and consequently higher surface area of zinc oxide nanoparticles, which enable them to interact closely with the microbial structures, give them better antimicrobial activity than their bulk counterparts. (Fouda et al., 2018; Silva et al., 2019; Singh et al., 2012). They also benefit from having photocatalytic and UV-protection abilities, which are advantageous (Fouda et al., 2018). Their primary antimicrobial effects include the generation of ROS, cellular wall lysis, photoconductivity, inhibition of DNA replication by binding to phosphate groups in DNA, and inhibitory effects on proteins and enzymes as a result of binding to the thiol (-SH) groups in their structural makeup (Fouda et al., 2018; Rajiv et al., 2013; Silva et al., 2019). In varying degrees, ZnO is effective against fungi, yeasts, and both Gram positive and Gram negative bacteria. There are numerous studies on the synthesis of zinc oxide nanoparticles (ZnONPs) using physical, chemical, and biological

methods. These techniques include enzymatic and sonochemical ones (El-Nahhal et al., 2020; Petkova et al., 2016; Singh et al., 2012), sol-gel procedures (Hasnidawani et al., 2016), chemical vapor synthesis (Lobiak et al., 2015), thermal decomposition, sonication, UV irradiation and surfactants for better stabilization (El-Nahhal et al., 2017; Y. Y. Zhang et al., 2016). The pros and cons of each method vary, but reports indicate that the biosynthesis methods are more widely accepted. The works done over the years to enhance cotton fabrics' properties by synthesizing and modifying ZnONPs will be the main focus of the section that follows.

## **5.1 Biosynthesis of ZnONPs**

Fouda et al. (2018) used extracts of proteins and enzymes from the fungus *Aspergillus terrus* AF-1 to report the biosynthesis of ZnONPs. The proteins primarily worked as reducing and capping agents for the synthesized ZnONPs. Following that, it was tested for cytotoxicity, antibacterial activity, and UV-protection factor (UPF). ZnONPs that were spherical in shape, evenly dispersed, and polydispersed were formed, according to data from FTIR, UV-vis spectroscopy, TEM, SEM, XRD, and DLS. Gram positive *B. subtilis* and *S. aureus* and Gram negative *E. coli* and *P. aeruginosa* were among the microorganisms whose susceptibility to synthesized ZnONPs was examined. When compared to Gram negative bacteria, the clear zones of inhibition for the ZnONPs were larger for the Gram positive bacteria. Both severe phenotypic changes in the exposed microbial cells and cell death were shown by light microscopy and MTT assay results for cytotoxicity, both of which were dose-dependent on the ZnONPs. These biosynthesized ZnONPs also demonstrated strong UVA (76.3%) and UVB (85.4%) blocking properties. UVA and UVB are two of the most harmful UV rays for humans because they can damage DNA and lead to skin cancer. Therefore, these biosynthesized ZnONPs could be applied to medical textiles to fight bacterial infections.

*Parthenium hysterophorus L.* leaf extracts are used in a different study that involves the biosynthesis of ZnONPs. , which is ranked as one of the ten worst weeds in the world (Rajiv et al., 2013). Although it is poisonous and pernicious, Kumar et al. (2011) and Mew et al. (1982) reported that it has pharmacological properties against a variety of diseases. In this study, various leaf extract concentrations were used as reducing and capping agents for the synthesis of ZnONPs of various sizes, and then the size-dependent antimicrobial efficacy of the synthesized ZnONPs was examined. The antifungal activity of ZnONPs on plant fungal pathogens, specifically *A. flavus* and *A. niger* were the focus of this study, instead of human pathogenic organisms. With the aid of UV-vis spectroscopy, FTIR, SEM, TEM, XRD, and TEM analyses, the formation of biosynthesized ZnONPs was verified. According to the size of the inhibition zones, A exhibited the highest antifungal activity of the synthesized ZnONPs. *flavus*, whose measurements exceeded those of the study's positive control. *F. culmorum* has the lowest level of activity. Additionally, the antibacterial activity of ZnONPs was inversely correlated with their size, supporting the previously established finding that smaller NPs have higher activity (Raghupathi et al., 2011).

## **5.2 Sonochemical and sonoenzymatic synthesis of ZnONPs**

Over time, El-Nahhal et al. (2020), Petkova et al. (2016) and Singh et al. (2012), have used the techniques of sonochemical and sonoenzymatic synthesis of ZnONPs have been used, who then combined them with starch and silver (El-Nahhal et al., 2020) to functionalize ZnONPs on cotton textiles. The ability of ZnONPs to adhere to cotton fibers was said to be enhanced by corn starch, which ultimately increases the ZnONPs' durability by lowering their environmental leaching.

Singh et al. (2012) used one of the first methods of sonochemical coating ZnONPs through UV irradiation, where they synthesized and coated ZnONPs onto fabrics using a single-step

method. This technique resulted in uniform and even coating of the NPs at very high speeds that ensure strong attachment with the fibers, reducing the cost and energy requirements. The antibacterial efficacy of the coated fabrics was then evaluated semi-quantitatively against *S. aureus* and *E. coli* using agar disk diffusion techniques and the shake flask method (using nutrient broth), as well as quantitatively using the absorption method and the shake flask method (again using saline solution). The coated fabrics displayed good antibacterial activity in all 4 types of testing, with *S. aureus* showing stronger antibacterial activity than *E. coli*. It is believed that the ZnONPs work by producing ROS, especially hydrogen peroxide, which interacts with the microbial cell wall and cell membrane and ultimately causes damage that causes the bacteria to die.

Petkova et al. (2016) using simultaneous sonochemical and enzymatic coating of the nanoparticles onto medical textiles, reported a single-step synthesis of ZnONP. In order to ensure better adhesion and consequently stability of the nanoparticles onto the fabrics, they were able to coat the synthesized ZnONPs in multiple layers. The treated fabrics were then tested for their antimicrobial effectiveness against *S. aureus* and *E. coli*, which demonstrated growth inhibition of 67% and 100%, respectively. Although it occasionally results in the production of ROS that can inhibit cellulase activity, ultrasound treatment has been shown to increase cellulase efficiency. When an active enzyme was present, durability tests after 10 washings in the presence of a non-ionic surfactant showed a 67 % NP loss, whereas it revealed a staggering 95 % NP loss when the enzyme was denatured. The enzymatic method improves ZnONP stability, which is further supported by this. A series of 10 washings caused the fabric containing the denatured enzyme to completely lose its antibacterial property, whereas the fabric treated with the active enzyme kept about 50% of its antibacterial activity. Once more, the unwashed fabric treated with both active and denatured enzymes demonstrated an approximate 70% reduction in bacteria.

El-Nahhal et al's (2020) most recent piece of work included the use of sonoenzymatic and UV irradiation processes for coating ZnONPs on cotton fibers. The media contained sodium hydroxide, which was used to reduce Zn(OH)<sub>2</sub> into spherical ZnONPs through the use of ultrasonication. Compared to cotton fibers alone, the starch/ZnONP-coated cotton fabrics demonstrated superior antibacterial activity against *S. aureus* and *E. coli*. Since starch holds more ZnONPs, it has been observed that increasing starch concentration improves antibacterial activity. Another study by the same team found that functionalizing ZnONPs with silver/curcumin had a synergistic antimicrobial effect in comparison to just using Zn-cotton fabrics (El-Nahhal et al. 2020). In order to increase the ZnONPs' ability to adhere to the cotton fibers, various surfactants and UV irradiation were used (El-Nahhal et al. (2017)). Due to their smaller size and larger exposed surface area, these surfactants also contributed to the production of ZnONPs that were more active. The following surfactants (in order of reducing attachment): SDS, followed by HY, CTAB, and then TX-100, provided the best stabilization and consequently the least leaching of the ZnONPs. When tested against *S. aureus* and *E. coli*, these produced NPs of various sizes, leading to varying antibacterial activities, with *S. aureus* showing greater antibacterial activity than *E. coli* when the surfactants SDS and HY were used. Additionally, these ZnONPs demonstrated greater antifungal activity against *C. albicans* than *M. canis*. The synthesized ZnONPs did, nonetheless, exhibit more antibacterial activity overall than antifungal activity.

### **5.3. Antimicrobial activity of aqueous zinc salts**

Researchers have studied the antimicrobial activity of plain aqueous zinc compounds in addition to synthesizing them and examining their ZnONPs' antimicrobial effectiveness. Holt et al. (2018) investigated the presence of residual antimicrobial action in aqueous solutions of zinc salts. They discovered that aqueous solutions of zinc salts, in particular zinc chloride (ZnCl<sub>2</sub>), retained residual antimicrobial activity for up to 30 days and could withstand rigorous

industrial drying processes. ZnO salts did not show the same results as ZnCl<sub>2</sub> because they failed the abrasive testing procedures. This could be explained by ZnCl<sub>2</sub>'s chemical attachment to the fibers of the textiles, whereas ZnO was only physically attached, leading to weaker bonds and easier leaching. Using four salt zinc salt solutions containing ZnCl<sub>2</sub>, ZnSO<sub>4</sub>, ZnNO<sub>3</sub>, and ZnO, the antimicrobial efficacy was evaluated against Gram positive and Gram negative bacteria as well as yeast. Although it performed better than the control, this showed the highest effectiveness against Gram positive bacteria of the three Zn salts, excluding ZnO. Furthermore, treated fabric showed some mass loss during abrasion testing while untreated fabric showed none, which could only be explained by zinc mass loss. Their findings, which show that fabrics treated with ZnCl<sub>2</sub> lost no mass while those treated with ZnO lost mass by 30%, further support the ZnO salts' weak physical bond to the textile fibers.

#### **5.4. Summarizing ZnONPs**

Based on the studies discussed above, it is quite clear how ZnONPs have proven to be very useful in antimicrobial coating of textiles, thereby aiding in increased chances of developing antimicrobial medical textiles. Zinc is more readily accessible, less expensive, and offers UV protection as well as photocatalytic activity. **Table 3** provides a summary of all these findings for easier comprehension.



Synthesis method	Antibacterial activity		Antifungal activity	UV-protection	Durability	References
	Gram positive	Gram negative				
Using <i>Aspergillus terrus AF-1</i>	More effective against <i>B.subtilis</i> , <i>S.aureus</i>	Relatively less effective against <i>E.coli</i> , <i>P.aeruginosa</i>	(N/A)	Blockage of 76.3% UVA; 85.4% UVB	(N/A)	(Fouda et al., 2018)
Using <i>Parthenium hysterophorus L.</i>	(N/A)	(N/A)	More effective against <i>A.flavus</i> than <i>A.niger</i> ; least effective against <i>F.culmorum</i>	(N/A)	(N/A)	(Rajiv et al., 2013)
Sonochemical, enzymatic and UV irradiation coating	67% reduction in <i>S.aureus</i>	100% reduction in <i>E.coli</i>	(N/A)	(N/A)	After 10 washings: 67% NP loss with active enzyme; 95% NP loss with denatured enzyme	(Petkova et al., 2016; Singh et al., 2012)
Sonochemical coating in presence of NaOH as reducing agent	Well effective against <i>S.aureus</i>	Well effective against <i>E.coli</i>	(N/A)	(N/A)	(N/A)	(El-Nahhal et al., 2020)
Aqueous zinc salts	All except ZnO showed highest activity	Relatively less effectivity	Relatively less effectivity	(N/A)	No mass loss(for ZnCl <sub>2</sub> ); 30% mass loss (for ZnO)	(Holt et al., 2018)

**Table 124** Antimicrobial, UPF and durability properties of differently synthesised ZnONPs

(N/A indicates no usable data recorded). Abbreviations used: NaOH; Sodium hydroxide; ZnO; Zinc oxide; ZnCl<sub>2</sub>; Zinc chloride.

## Chapter 6

### Titanium oxide nanoparticles (TiONPs)

Titanium oxide nanoparticles will be the final metal oxide nanoparticle to be discussed in this essay. Known for its stunning silver color, low density, and high strength, titanium is a member of the transition metal family. Titanium is used as a connection between bones and to replace hip joints in the biomedical field, as well as in orthodontic procedures in the field of dentistry (Titanium's Special Properties — Science Learning Hub, n.d.). It is also renowned for having a special ability to be extremely resistant to corrosion caused by chlorine and seawater. Titanium oxide nanoparticles (TiONPs), in addition, have made their way into the field of biotechnology due to their numerous and desirable qualities, including photocatalytic activity, UV protection, high stability, safety, non-toxicity, low cost, high availability, biocompatibility, and of course, antimicrobial, anti-cancer, and anti-inflammatory activities. ((Ahmadi et al., 2019; Dastjerdi & Montazer, 2010; Humphreys, 2014; Parthasarathi & Thilagavathi, 2013; Rao et al., 2019). They are also used in the food, paper, cosmetics, textile, and other industries because of their wide range of properties (Muhd Julkapli et al., 2014). These are additionally employed in the field of pharmacology for the administration of medications as well as for imaging, diagnosing, and treating a variety of illnesses (Ramar et al., 2015). Additionally, because of their protective qualities and ability to lengthen the shelf life of these products, they are used as packaging materials for food and drugs (Ahmadi et al., 2019). Because of their anti-tumor effects, they have demonstrated their value as a potential cancer treatment strategy when combined with other treatment methods (Rao et al., 2019).

In the literature review that was covered, it was found that TiO<sub>2</sub>NPs were most frequently used for their excellent photocatalytic properties, though researchers were also looking into their antimicrobial and biocompatibility properties. Thus, some of the research on TiO<sub>2</sub>NPs' use in

photocatalysis and antimicrobial (antibacterial, antifungal, and antiviral) activity has been covered in the sections below.

### **6.1. Antiviral activity of TiO<sub>2</sub>NPs in surgical gowns**

In order to prevent contamination of sterile areas and thereby help prevent or reduce nosocomial infections, surgical gowns in hospitals must obviously be made of a material that is resistant to blood, liquids, and abrasion as well as, of course, resistance to microbial colonization. To prevent suffocation, the fabrics must be able to withstand moisture while still allowing air to flow through. Along with the rising concern for health and hygiene, Parthasarathi & Thilagavathi (2013) kept this in mind. They created a nanodispersion of TiO<sub>2</sub> and used the pad-dry-cure method to coat nonwoven fabrics with it. Three-layered nonwoven fabrics made of polypropylene, polytetrafluoroethylene, and polyester were selected as the outer, middle, and inner layers, respectively. A proper coating of the fabrics was evident when the synthesized nanoparticles and coated fabrics were characterized using HR-TEM and SEM. The fabric's microporosity enables good breathability. All viral penetration tests on the fabric treated with nanoparticles were successful, demonstrating the fabric's excellent viral resistance. This indicated that the fabric had been treated to be immune to HBV, HCV, HIV, and Phi X174. According to their research, these single-use, disposable gowns make up only 2% of all hospital waste while preventing the release of toxic chemicals into water, a problem with reusable gowns that arises from the need for frequent washing.

### **6.2. Green synthesis of TiO<sub>2</sub> NPs**

Due to the obvious advantages of environmental friendliness and low cost operations, studies have been conducted focusing on the green synthesis of TiO<sub>2</sub> nanoparticles.

Ahmadi and colleagues conducted a study in 2019 where extracts from *Miswak (Salvadora persica L.)* (SPE) were employed in the production of TiO<sub>2</sub> NPs for use as packaging in the

food and drug industries (Ahmadi et al. 2019). A small shrub tree native to the Middle East called miswak has unique biological traits, including antimicrobial properties. The World Health Organization (WHO) has already approved the use of *Miswak* roots to promote better oral hygiene. In addition to acting as anti-inflammatory, antimicrobial, and antioxidant agents, SPE is non-toxic (H. Balto et al., 2017; H. A. Balto et al., 2014; Chelli-Chentouf et al., 2012; Mohamed & Khan, 2013). These advantageous characteristics are due to the presence of several bioactive substances, with benzyl isothiocyanate (BITC) serving as the primary antimicrobial agent. As a result, Ahmadi et al. (2019) combined all the various characteristics of these materials and created a mixture that was combined with carboxymethyl cellulose (CMC) to produce films using the casting method. The end result of this process was to endow the treated material with antimicrobial, thermophysical, and barrier properties. Physical and mechanical properties, elemental mapping analysis (MAP), XRD, SEM, FTIR, UV-vis, spectroscopy, EDX, and TGA-DTG methods were used to further validate and confirm the results. These all supported the formation of homogenous films, which were then tested for antimicrobial effectiveness against *S. aureus* and *E. coli*. Excellent antibacterial properties were demonstrated against both by pure SPE and the CMC- TiO<sub>2</sub>-SPE, with the latter exhibiting a dose-dependent increase in antibacterial activity.

In another investigation, Rao et al. (2019) utilized aqueous *Acacia nilotica* extracts as the bio-reductant to create silver-doped TiO<sub>2</sub> nanoparticles. Utilizing FTIR, XRD, FESEM, EDS, and TEM, synthetic Ag/TiO<sub>2</sub> was characterized. These were then put through antibacterial and antifungal tests using well diffusion methods, where they demonstrated their activity in the following order: *E. coli*>*C. albicans*>*MRSA*>*P. aeruginosa*. Because of this, these Ag/TiO<sub>2</sub> nanoparticles were effective against yeasts, Gram positive and negative bacteria, and both. This is thought to have an ROS-generating mechanism that causes phospholipid peroxidation, which kills cells, as its end result. Additionally, using the MTT assay in the MCF-7 cells, a breast

cancer cell line, the cytotoxicity and oxidative stress of the synthesized nanoparticles were assessed. This demonstrated that as nanoparticle concentration rises, the interaction between the particles and cells increases, amplifying cytotoxic effects and lowering cell viability. Additionally, they lower the glutathione (GSH) concentrations in tumor cells. GSH contributes to signal transduction pathways and other ways that tumor cells can multiply. All of these thus provide sufficient evidence that Ag/TiO<sub>2</sub> nanoparticles may be used as an effective anticancer therapy for cancer patients.

These advantageous qualities of the biosynthesized nanoparticles from the two studies mentioned above point strongly to their potential as medical textile coatings. In the near future, however, that sector will not be actively pursued.

### **6.3 Fe-N doped TiO<sub>2</sub> nanoparticles**

One of the most thoroughly studied characteristics of TiO<sub>2</sub> is its photocatalytic ability. Stan et al. (2016, 2019) have conducted extensive and in-depth research on the photocatalysis, antimicrobial, and biocompatibility of Fe-N doped TiO<sub>2</sub> nanoparticles, and used their findings to coat cotton fibers with the Fe-N doped TiO<sub>2</sub> nanoparticles to create self-cleaning medical textiles.

Stan along with other people had utilized graphene oxide or Fe-N doped TiO<sub>2</sub> nanoparticles that had been hydrothermally synthesized in 2016 or 2019 as coating materials for cotton textiles. Instead of extracting usable nanoparticles from waste, which significantly raises the cost of experimentation, the team in the earlier study (2016) concentrated particularly on coating with repurposed dispersions of the TiO<sub>2</sub> -1% Fe-N nanoparticles. They discovered that recycling nanoparticle dispersions increased the concentration of nanoparticles in the fabric, resulting in an increase in photocatalytic activity. Trichromatic coordinates method confirmed that visible light was the most effective form of light for photocatalysis, as opposed to UV and

solar light. Similar outcomes for photocatalytic activity were found in the more recent study (Stan et al. 2019).

Similar results for both studies' antimicrobial efficiency tests were obtained, but the effects on the fabrics were different because of the fabric's composition, the microbial strain, and the incubation time. However, the initial investigation revealed antibacterial activity in the following order: *E. coli* > *P. aeruginosa* > *Saureus* > *E. faecalis* Stan et al. (2016). On the other hand, a recent study revealed a 77% and a 33% inhibition of *E. faecalis* growth by knit samples K-S1 and K-S3 samples, respectively, and no discernible effect was seen in *E. coli* after 24 hours of contact with the fabrics, with K-S2 showing marginally inhibitory effects Stan et al. (2019). The production of ROS, which ruptured microbial cell membranes and caused DNA damage, could be the cause of TiO<sub>2</sub>'s antimicrobial activity. The structural and metabolic differences between Gram positive and Gram negative bacteria are thought to be the cause of the difference in this action, with Gram negative bacteria having more treatment resistance due to the outer membrane of lipoproteins in those bacteria.

After a brief exposure of 4 hours of contact, (Stan et al., 2016) found no cytotoxic effects, as well as continuous exposure over a 24-hour period (Stan et al., 2019) was observed when experiments were conducted on human dermal fibroblasts. Absence of any morphological or cell viability changes served as confirmation of this. The biocompatibility of the TiO<sub>2</sub> nanoparticles is thus demonstrated. The presence of graphene in the multiple layers of coating increased the treated fabric's hydrophobicity, which makes them more resistant to microbial colonization and ensures self-cleaning.

#### **6.4. Summarising TiO<sub>2</sub>NPs**

It is clear how beneficial titanium oxide nanoparticles can be to the world based on the works by various research teams mentioned above. TiO<sub>2</sub>NPs are one of the most prized subcategories

of nanoparticles due to their extensive beneficial properties of photocatalysis, UV protection, biocompatibility, high availability, as well as various antibacterial, antifungal, and antiviral effects. Despite all of this, additional research is still required to confirm these beneficial properties, and in the meantime, TiO<sub>2</sub>NPs are being searched for additional novel properties and applications. **Table 4** provides a brief summary of the TiO<sub>2</sub>NPs research mentioned above.

Category	Antibacterial activity		Antifungal activity	Antiviral activity	Photocatalytic activity & Cytotoxicity	References
	Gram +ve	Gram -ve				
TiO <sub>2</sub> in surgical gowns	(N/A)	(N/A)	(N/A)	HBV, HCV, HIV, Phi X174	(N/A)	(Parthasarathi & Thilagavathi, 2013)
Synthesis using SPE	<i>S.aureus</i>	<i>E.coli</i>	(N/A)	(N/A)	(N/A)	(Ahmadi et al., 2019)
Synthesis using aqueous extracts of <i>A.nilotica</i>	<i>MRSA</i>	<i>E.coli</i> , <i>P.aeruginosa</i>	<i>C.albicans</i>	(N/A)	Destroyed MCF-7 cells, displaying anticancer property	(Rao et al., 2019)
Fe-N doped TiO <sub>2</sub> (reused dispersion)	<i>S.aureus</i> , <i>E.faecalis</i>	<i>E.coli</i> , <i>P.aeruginosa</i>	(N/A)	(N/A)	Better activity under visible light; No effect on human dermal fibroblasts	(Stan et al., 2016)
Graphene oxide/Fe-N doped TiO <sub>2</sub>	77% and 33% <i>E.faecalis</i> inhibition by K-S1 &K-S3 respectively.	No significant effect on <i>E.coli</i> after 24hrs	(N/A)	(N/A)	Better activity under visible light; No effect on human dermal fibroblasts	(Stan et al., 2019)

**Table 140 Antimicrobial, photocatalytic and cytotoxicity effects of differently synthesised TiO<sub>2</sub>NPs.** (N/A:

No usable data recorded). Abbreviations used: HBV, HCV; Hepatitis B and Hepatitis C virus (respectively); HIV; Human immunodeficiency virus; SPE; *Salvadora persica* leaf extract; MRSA; Methicillin-resistant *Staphylococcus aureus*; K-S1 & K-S3; Knit fabric samples 1&3



## **Chapter 7**

### **Which nanoparticle is better for textile industry?**

Actually, it is impossible to choose one nanoparticle over another by applying a strict rule. Each of the nanoparticles has unique advantages and disadvantages in terms of their antimicrobial efficacy, the synthesis and modification processes used, cost and energy requirements, as well as their durability of action, as was already covered in detail in the sections above. The textile industry will undoubtedly need such a component that can be produced on a large scale and has the least amount of issues in order to mass-produce textiles with excellent antimicrobial and barrier properties. A broad spectrum of antibacterial, antifungal, and antiviral properties should be present in the chosen nanoparticle. The nanoparticles must be selected with the current coronavirus pandemic in mind in order to make them offer even a small amount of protection to the general populace. The selected nanoparticle should be capable of being used as effective fabric coatings for masks, personal protective equipment (PPE) for frontline workers, patient and nurse scrubs, privacy drapes, as well as made into the typical everyday-wear clothing for the general public.

Therefore, in order to select the most practical metal or metal oxide nanoparticle to be used in the textile industry, the following paragraphs present a comparative analysis, centered on the various modification processes used, as well as their antimicrobial efficacy and durability. Cost-effectiveness and environmental friendliness should also be taken into consideration. This review is a descriptive comparison rather than a statistical one because there aren't enough statistical data to support it.

## **7.1 Comparison based on modification processes**

Each of the metal nanoparticles has a variety of synthesis and modification processes, as was already seen above. These include chemical techniques like sonochemical and enzymatic reactions, UV radiation, the use of various polymers, biopolymers, and chemical molecules as binders and stabilisers, and last but not least, biosynthesis using various plant extracts, fungus filtrates, etc. The following Table 5 provides a brief summary of the antimicrobial effect based on the various processes used for each of the AgNPs, CuONPs, ZnONPs, and TiO<sub>2</sub>NPs.

Modification process	Different nanoparticles and their antimicrobial effect			
	AgNPs	CuONPs	ZnONPs	TiO <sub>2</sub> NPs
<b>Chemical</b>	<b>Antibacterial:</b> 96-100% for <i>E.coli</i> , <i>S.aureus</i>	<b>Antibacterial:</b> <i>S.aureus</i> , <i>Enterococcus</i> <i>sp.</i> and <i>S.epidermidis</i> ; <i>E.coli</i> , <i>S.enteritidis</i> and <i>P.aeruginosa</i>  <b>Antifungal:</b> <i>C.albicans</i>  <b>Antiviral:</b> H1N1, H5N1, H5N3, HIV, HBV, HCV, bronchitis virus, poliovirus	<b>Antibacterial:</b> <i>E.coli</i> , <i>S.aureus</i>  <b>Antifungal:</b> <i>C.albicans</i> , <i>M.canis</i>	<b>Antibacterial:</b> <i>E.coli</i> > <i>P.aeruginosa</i> > <i>S.aureus</i> > <i>E.faecalis</i>  <b>Antiviral:</b> HBV, HCV, HIV, Phi X174
<b>Biosynthesis</b>	<b>Antibacterial:</b> 99-100% for <i>E.coli</i> , <i>S.aureus</i>	<b>Antibacterial:</b> <i>E.coli</i> , <i>S.aureus</i>	<b>Antibacterial:</b> More effective against G+ve <i>S.aureus</i> and <i>B.subtilis</i> than G-ve <i>E.coli</i> and <i>P.aeruginosa</i>  <b>Antifungal:</b> <i>A.flavus</i> , <i>A.niger</i>	<b>Antibacterial:</b> <i>S.aureus</i> , <i>E.coli</i> , <i>MRSA</i> , <i>P.aeruginosa</i>  <b>Antifungal:</b> <i>C.albicans</i>
<b>Additional properties</b>	UV-protection (UPF); Wound-healing; No cytotoxicity	Photocatalysis, UPF; dye degradation; No cytotoxicity; HAIs reduction	UPF; No cytotoxicity	Excellent photocatalysis,; Anti-cancer potential; No cytotoxicity

**Table 141 Comparison between AgNPs, CuONPs, ZnONPs, TiO<sub>2</sub>NPs in terms of synthesis methods, broad-spectrum antimicrobial activity and additional properties.**

## 7.2 Comparison based on durability actions

The ability of the synthesised nanoparticles to maintain their antimicrobial qualities even after repeated washings is crucial. Additionally, it's critical that the nanoparticles adhere firmly to the textile fibers to prevent leaching into the environment, which would primarily harm aquatic life. Taking into account these factors, **Table 6** compares the four nanoparticles mentioned above based on their durability.

<b>Durability actions: Retainability of antimicrobial activity</b>			
<b>AgNPs</b>	<b>CuONPs</b>	<b>ZnONPs</b>	<b>TiO<sub>2</sub>NPs</b>
After 20-50 washings, 88-98% bacteriocidal	(N/A)	After 10 launderings, retains 33% NPs (with active enzyme treatment); No mass loss for ZnCl <sub>2</sub>	(N/A)

**Table 172 Durability-based comparison among AgNPs, CuONPs, ZnONPs, TiO<sub>2</sub>NPs.** (N/A: No usable data recorded).

## 7.3 Economic comparison

There is no shadow of a doubt that using biosynthesis techniques to create any type of nanoparticle is one of the most widely praised strategies, as shown by the literature review and the abundantly clear evidence in front of us. For the same obvious reasons, the physical and chemical methods demand a lot of energy, a hot environment, a lot of chemicals, etc. The biosynthesis methods, on the other hand, make use of all the natural resources present on Earth, including extracts from plants, shrubs, fungi, algae, etc. These reduce additional health risks for the researchers and primarily the aquatic community because they do not necessitate the excessive use of chemicals that are toxic and hazardous for life on Earth.

With the possible exception of titanium, which is quite expensive on its own and is required in large quantities for some methods, the metals themselves are also generally readily available and reasonably priced (Stan et al., 2016).

## Chapter 8

### Discussion

It goes without saying that the application of nanobiotechnology and nanoparticles to health and hygiene has managed to transform the biomedical and textile industries. The purpose of this paper was to provide some insight into the antimicrobial properties of fabrics treated with the four most popular metal and metal oxide nanoparticles, namely AgNPs, CuONPs, ZnONPs, and TiO<sub>2</sub>NPs. It can be inferred that copper and copper oxide nanoparticles have a very high potential as impregnating materials for textiles given their broad-spectrum antimicrobial efficiency, as well as the added benefits of photocatalysis, UPF, dye degradation, non-toxicity to humans but highly toxic to microbes even at low concentrations, and reports of having been successful in reducing HAIs. Copper is inexpensive, readily available, and most importantly, among its many antimicrobial effects, it has been demonstrated in a number of studies just how potent the CuONPs are in terms of their antiviral potentials. Because of the ongoing SARS-CoV-2 pandemic, copper's exceptional antiviral properties are crucial right now. The viricidal effects of CuNPs and CuONPs on coronaviruses or surrogate coronaviruses have been the subject of extensive research, but despite this, there are currently few studies in this area. In addition, a study carried out by Lazary et al. (2014) found that CuO-impregnated hospital textiles and linens have been successful in lowering the frequency of HAIs, the number of fever days, antibiotic use, GI infection, and eye infection. Considering all these positive aspects, it can be hypothesized that copper and copper oxide nanoparticles might represent a very strong candidate for triggering antimicrobial activity in textiles.

## **Chapter 9**

### **Conclusion**

It is crucial to maintain good hygiene and health. Since the COVID-19 pandemic's emergence, it has now drawn the most attention. Antimicrobial fabrics and clothing are crucial in these trying times. In addition to proving their value in coating textiles and introducing antimicrobial properties into them, nanoparticles are making great strides in a wide range of industries. The results of this study all point to the advantages of using nanoparticle-based fabrics, giving rise to some optimism about the prospects for future research into the creation of textiles that can shield people from the ongoing pandemic and other infectious diseases.

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