



## Nanotechnology and Application of Nano Titanium Dioxide, Nano Zinc Oxide, and Nano Copper Oxide on Textile for High Performance

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### Abstract

Nanotechnology is recognized as one of the most promising 21<sup>st</sup> century technologies. Nanotechnology is the study and use of very small things, and it may be applied in all other science subjects, including physics, chemistry, biology, materials science, and engineering. Nanomaterials may be created using a number of techniques, including physical, chemical, and biological ones. The application of nanotechnology allows textiles to become multifunctional and develop materials with unique properties such as antimicrobial, UV protection, easy-cleaning, water- and stain resistant, and anti- odour. The use of nano titanium dioxide, nano zinc oxide, and nano copper oxide on cotton, wool, and cotton/polyester textiles have been addressed.

**Keywords:** Nanotechnology, Nano titanium dioxide, Nano zinc oxide, Nano copper oxide

### 1. Introduction

Nanotechnology is a new interdisciplinary technology that has exploded in popularity in a variety of fields in the previous decade, including materials science, mechanics, electronics, optics, medicine, plastics, energy, and aerospace. Its massive societal influence has been credited with providing the impetus for a second industrial revolution. [1]

The principles of nanotechnology are based on the notion that when a substance's size is decreased to the nanoscale range, its properties radically alter. When a large material is broken down into particles of one or more dimensions, individual particles with dimensions (length, breadth, or thickness) in the nanometre range or even smaller display unexpected properties that differ from those of the bulk material. It is well known that atoms and molecules behave very differently than bulk materials. [1-3]

Nanomaterials are materials with extremely small particle sizes, at least one of which is in the 1–100 nm range. When materials are made at the nanoscale, they have unique electrical, magnetic, catalytic, and optical properties that can be used in a wide range of applications. [4]

Nanoscience technology has emerged as a cutting-edge technology and science in recent years. In the industry, nanoscience and technology have had a considerable impact. It is concerned with the extremely small structure of materials, which

demonstrates unmistakable innovation and improves biological, chemical, and physical properties.

Nanotechnology is well-known and widely employed in a variety of fields to solve problems or improve desired properties. Nanotechnology has been employed in the textile industry to improve the performance of numerous processes such as dyeing, printing, and finishing. Many studies have been conducted on the usage of nanoparticles on fabrics in order to develop finely finished textiles with a variety of valuable functional qualities. Nano magnesium, for example, is employed to obtain antibacterial properties, Nano titanium dioxide is used for UV protection and self-cleaning qualities, while Nano zinc oxide is recognised for its antimicrobial effects and UV protection.

When textiles are treated with Nano-materials, they exhibit different characteristics such as tensile strength, flexibility, wear durability, water and oil repellence, light fastness, and thermal consistency. [5] When these nanomaterials with unique characteristics and features are brought to the domain of functional finishing of textile fabrics, multifunctional properties and added value can be developed. [6]

Depending on the type and nature of the nanomaterials, multiple processes are used to create them. [7-9] The two most common approaches are "top-down" and "bottom-up." Nanoparticles (NPs) provide a small size a location that is required for a

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consistent material reaction. NPs can be generated from hydrous solutions utilising a variety of processes, including sedimentation, micro-emulsion, and hydrothermal procedures. Apart from gas-phase synthesis, wetted chemical synthesis of nanoparticles is a useful alternative to the standard technique. Apart from gas-phase synthesis utilising standard commercial implementations, wetted chemical synthesis of nanoparticles is a useful alternative to the classic method. The NPs have a larger surface area and are more capable of exhibiting unique biological, optical, physical, and chemical properties. Organic and non-organic NPs are the two types of NPs.

Organic NPs, in general, are unable to withstand extreme circumstances such as high pressure and temperature. Inorganic NPs, on the other hand, such as copper oxide, zinc oxide, and titanium dioxide, have a high stability in harsh environments; thus, adopting inorganic NPs has a big impact in many disciplines, particularly microbial resistance. (3) The methods of synthesize nanomaterials are (Conventional Sol-Gel, Microwave Radiation, Gas Phase, Combustion, Templating, Chemical Vapor Deposition, Solvothermal and Hydrothermal. [10]

In this paper we compile and summarize methods of synthesize nanomaterials and application of nano titanium dioxide, nano zinc oxide and nano copper oxide on cotton, wool and cotton/polyester fabrics.

## 2. Methods to Synthesize Nanomaterials

### 2.1. Top-Down Method

Solid and state processing of materials are commonly utilized in this method, which entails breaking down bulk materials into tiny particles through physical processes including crushing,

milling, and grinding. In general, this process is ineffective for producing uniformly shaped nanomaterials, and it is extremely difficult to obtain very small nanoparticles, even when using a lot of energy. The lack of surface structure, which has a substantial impact on physical characteristics and surface chemistry of nanomaterials, is the method's main challenge. Furthermore, the treated forms suffer significant crystallographic loss as a result of this method. [10]

### 2.2. Bottom-Up Method

Manual grinding and the use of ball milling equipment were used in the Bottom-Up technique for the synthesis of noble metal NPs from their salt predecessors. Ball milling's use to the controlled synthesis of NPs has grown as a result of this methodology's success. (Bottom region) depicts this approach, in which NPs are generated by reducing precursor chemicals that form growth species (metal atoms). To produce the final NPs, these atoms go through various nucleation and growth processes (e.g., atomic addition, coalescence, Ostwald ripening, directed attachment). In contrast to what was described in the top-down technique, an increase in milling time leads to an increase in the size of the NPs as a result of growth in the bottom-up strategy.

Due to the coalescence of smaller particles and welding, even in the presence of capping agents, long milling times can further reduce the overall surface area. Bottom-up mechanochemical techniques, in theory, provide a lot of potential for controlling the physicochemical properties of NPs by regulating the nucleation and growth stages during synthesis. [11]

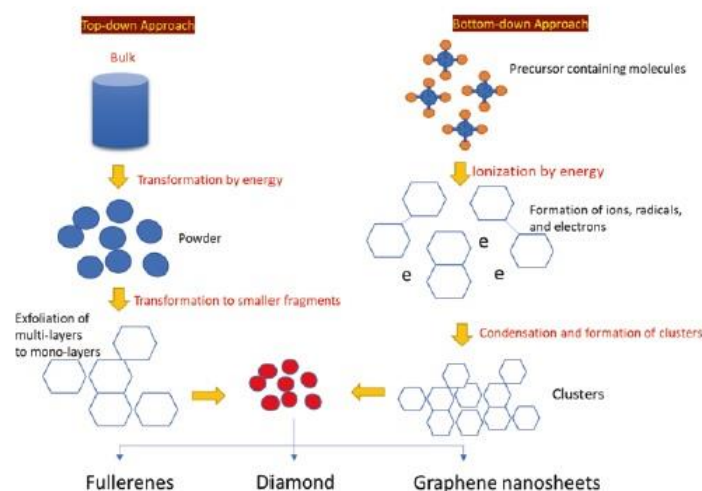


Fig. 1: A diagrammatic illustration of the top-down and bottom-up approaches to nanomaterial synthesis

### 2.3. Hydrothermal Method

The hydrothermal process is typically carried out in a pressurised container known as a "Autoclave," which allows temperature and pressure

to be controlled and maintained. The temperature at the boiling point of water can be raised during nanomaterial manufacturing, allowing the vapour to become saturated. This method has been widely

employed in the manufacture of many nanoparticles. The advantage of this method are that it can be used to regulate reaction temperature, pressure, solvent properties, solution composition, and additives to control material size, particle morphology, crystalline phase, and surface chemistry. [10]

#### **2.4. Solvothermal Method**

The solvothermal method is similar to the hydrothermal process, with the exception that it does not employ water as a solvent. Surprisingly, when organic solvents or compounds with high boiling temperatures are used, this approach is more effective in the synthesis of nanomaterials with good distribution. Furthermore, compared to the hydrothermal approach, this process allows for more precise control of material size and shape. With or without the inclusion of surfactants, this process produces nanomaterials or nanorods. [10]

#### **2.5. Chemical Vapor Deposition Method**

Conditions are established in the vapor-phase synthesis of NPs where the vapour phase combination is thermodynamically unstable in comparison to the generation of the solid substance to be synthesised in nanoparticle form. This approach is extremely versatile in terms of generating a wide range of materials, and it can make use of the vast database of precursor chemistries produced for CVD processes. At ambient temperatures, the precursors can be solid, liquid, or gas, but they are transported to the reactor as a vapour. [12]

#### **2.6. Method of Laser ablation (LA) and pulse laser deposition (PLD)**

A high-powered laser beam is used to vaporise particles from a solid source in the laser ablation process. The laser in a typical LA process might be either continuous or pulsed. By scanning the polymer surface to obtain a lens shape with the correct focal distance and diameter, LA offers a versatile technique in the manufacture of micro- and nanostructures of polymeric materials. [13]

PLD is another vacuum-based PVD method that removes material from the target using laser radiation. The high-powered laser pulses impact the target's surfaces, causing melting, evaporation, and ionisation. Finally, the components that have been ablated settle onto the substrate. For material ablation, PLD employs a pulsed laser beam laser. PLD is used to make a wide range of materials, including polymers, oxides, metallic systems, fullerenes, carbides, and nitrides, among others. [13]

#### **2.7. Soft and hard Templating Method**

To make nanoparticles materials, soft and hard template methods are widely employed. The soft template method is a traditional approach for creating nanostructured materials that is simple to use.

Because of its simple implementation, relatively mild experimental conditions, and the production of materials with a variety of morphologies, the soft template approach has been deemed useful. Soft templates, such as block copolymers, flexible organic molecules, and anionic, cationic, and non-ionic surfactants, are used to make nanoporous materials in the soft templating methods. Hydrogen bonding, van der Waals forces, and electrostatic forces are the most prevalent interactions between the soft templates and the precursors. [14]

Nano-casting is another name for the hard template method. To achieve nanostructures for necessary applications, well-designed solid materials are employed as templates, and the solid template pores are filled with precursor molecules. The hard template must be chosen carefully in order to create well-ordered mesoporous materials. Such hard templates should maintain a mesoporous shape during the precursor conversion process, and they should be easily removed without affecting the nanostructure created. As hard templates, a variety of materials have been employed, including carbon black, silica, carbon nanotubes, particles, colloidal crystals, and wood shells. [14]

#### **2.8. Combustion Method**

Combustion-based procedures, also known as combustion synthesis (CS) or self-propagating high-temperature synthesis (SHS), are an efficient way to make a variety of sophisticated materials while saving energy. Once the initial exothermic reaction mixture is ignited by an external thermal source, a rapid (typically between 0.1 and 10 cm/s) high-temperature (1000–3000 C) reaction wave propagates through the heterogeneous mixture in a self-sustained manner, resulting in the formation of the solid material without requiring any additional energy. Another CS mode, known as volume combustion synthesis (VCS), involves heating the entire sample uniformly until the reaction self-initiates throughout the volume. This less controlled synthesis method is employed for weakly exothermic processes that need to be preheated before ignition. [15]

#### **2.9. Gas-Phase Method**

In the gas-phase synthesis of nanomaterials, so-called precursors degrade into condensable species in a highly supersaturated state, which leads to particle nucleation and growth after being exposed to high temperatures. The ultimate goal of gas-phase nanoparticle synthesis is to have complete control over the materials composition (pure, doped, mixed materials) that can be present as homogeneous materials, nanomaterials of materials with different compositions, or structured materials like core-shell structures or Janus particles that expose two different materials at the surface of a single particle. The degree of oxidation, and hence the complete spectrum

between metallic and totally oxid materials, is particularly significant in the case of flame synthesis of oxide materials, where sub-stoichiometric materials are generally of particular interest due to their changed electrical and catalytic properties. Furthermore, as previously stated, nanoparticle properties are frequently influenced by the primary particle size. As a result, one of the key concerns in nanoparticle synthesis is managing the nanoparticle size. [16]

### 2.10. Microwave Radiation Method

Microwave aided synthesis is widely used in a variety of fields, including biological processes and nanotechnology. Chemical processes are frequently faster and have higher yields with fewer side products than traditional convection heating methods. Microwave reactors have good reaction mixing control, can resist high temperatures and pressures, and are repeatable from reaction to reaction. When a process is started at room temperature, microwave assisted approaches allow better engineering control over the separation of the nucleation and growth stages of nanomaterial creation. Microwave-assisted heating may be able to provide some selectivity in the activation of nanomaterial precursor materials, which is crucial for scalability. Microwave synthesis provides the ability to heat either the solvent or the precursor molecules for the creation of nanomaterials. [17]

### 2.11. Conventional Sol-Gel

Because of its simplicity, reproducibility, consistency, and cost-effectiveness, the sol-gel process is widely used to synthesize materials. The optical characteristics of the sol-gel nanoparticles are excellent. The sol-gel technique may be used to make a number of materials, including nanoparticles, thin-film coatings, and ceramics, with a wide range of uses. An inorganic network is generated in this manner via a chemical reaction that occurs in a solution at a low temperature. The sol-gel process has a number of advantages, including good homogeneity and purity of the synthesized product, as well as being cost-effective and easy. Non-vacuum circumstances are employed to make high-quality homogeneous nanoparticles. In the sol-gel process, an organometallic precursor (such as alkoxides, chloride, beta-diketone, or nitrate) undergoes hydrolysis and condensation reactions in aqueous circumstances to generate a solid substance. To obtain the necessary material, the precursor solution undergoes hydrolysis and condensation reactions, resulting in the development of a gel; this is followed by ageing, solvent extraction, and drying treatment of the synthesized product. [18]

### 2.12. Natural (Green) method

In the functionalization process of textiles with NPs, reducing agents, different chemical compounds that act as stabilizing agents, dispersing agents, and binders are commonly used. Because these chemicals can be harmful to human health and the environment, researchers are working to replace them with more environmentally friendly compounds like microbes or phytochemicals. Photosynthesis is a process of nanoparticle creation in which phytochemicals found in plant extracts are employed as reducing, capping, and stabilizing agents. Although the exact mechanism is unknown, it is thought that primary and secondary metabolites are responsible for metal nanoparticle production and act as reducing and stabilizing agents. While phenolic chemicals have a strong antioxidant capacity and are excellent metal ion reducers. [19]

## 3. Application of nanotechnology in textile

Nanotechnology's usage in the textile industry has grown fast as a result of its unique and desirable features. Nanotechnology has a lot of potential for commercial applications in the cotton and textile sectors. Its use can increase the characteristics and value of textile processing and products at a lower cost. Nanotechnology allows textiles to become multifunctional and develop materials with unique properties such as antimicrobial, UV protection, simple cleaning, water and stain resistance, and anti-odor. The future success of nanotechnology in textile applications will be determined by regions where novel principles will be incorporated into durable, multifunctional textile systems without compromising inherent textile features like as processability, elasticity, and so on. [20]

## 4. Role of nano materials in textiles

### 4.1. Titanium dioxide nanoparticles

Incorporating nanoparticles into textile finishing is a new notion that has emerged in recent years. The most important material that has been extensively used in this industry is  $\text{TiO}_2$ . Because of its strong photo-catalytic activity, non-toxicity, and physicochemical stability, it is becoming increasingly popular. [10] A photocatalyst is titanium dioxide ( $\text{TiO}_2$ ). The photocatalytic activity of fabric treated with nano- $\text{TiO}_2$  was found to provide effective protection against germs and stain discoloration. The electrons in titanium dioxide will hop from the valence band to the conduct band when irradiated by light beam higher than their band gaps, forming electron and electric hole (h) pairs on the photocatalyst's surface. The positive electric holes and water produce hydroxyl radicals when the electrons and oxygen combine to form ( $\text{O}_2$ ). Because organic compounds are unstable, when they fall on the surface of a photocatalyst, they will combine with both and produce carbon dioxide and water. "Oxidation-reduction" is the name given to this

cascade reaction. The photocatalyst can decompose common organic matter in the air, such as odour molecules, bacteria, and viruses, using the reaction. [21]

The properties of textiles could be modified with the use of TiO<sub>2</sub> nanoparticles, such as: hydrophobicity / hydrophilicity, UV protection, antibacterial properties and anti-wrinkle resistance. [22]

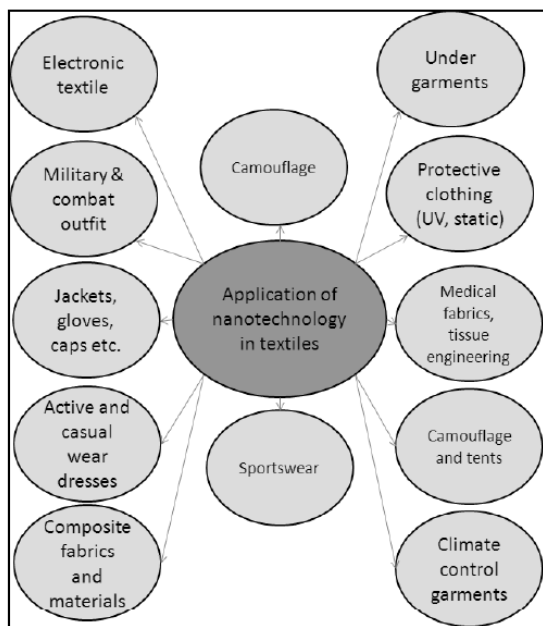


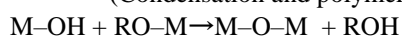
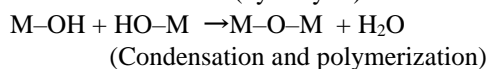
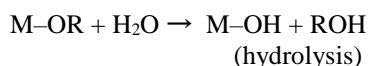
Fig. 2: Some representative applications of nanotechnology in textiles.

#### 4.1.1. Synthesis of TiO<sub>2</sub> nanoparticles

##### 4.1.1.1. So-gel method

The sol-gel synthesis is the most promising method for making TiO<sub>2</sub> nanoparticles because it has several advantages, including low temperatures, versatility in performance, and molecular homogeneity. In the sol-gel process, a titanium precursor, commonly titanium (IV)chloride or titanium tetra isopropoxide (TIP), is hydrolyzed in a solution of water and alcohol under acidic conditions. It entails the hydrolysis of a similar TiO<sub>2</sub> precursor followed by a polymerization reaction (Scheme 1), which results in the creation of the liquid sol phase. The rate of hydrolysis, amount of water, TiO<sub>2</sub>/water ratio, applied temperature, and reaction duration all influence the results of these processes. The development of Ti-O-Ti polymeric chains is favoured in the presence of large amounts of TIP and low hydrolysis rates, whereas the formation of Ti(OH)<sub>4</sub> is favoured in the presence of medium amounts of water and high hydrolysis rates. Because the viscosity of the solution and TiO<sub>2</sub> solubility are temperature dependent, the rate of particle coarsening increases with increasing temperature. The average radius of a TiO<sub>2</sub> nanoparticle increases linearly as reaction time

increases. The creation of the solid gel state is caused by complete polymerization and the loss of solvent.



Scheme 1. Sol-gel synthesis of TiO<sub>2</sub> nanoparticles

Amorphous nanoparticles are produced by the sol-gel technique, which are then transformed to crystalline TiO<sub>2</sub> by heating at temperatures exceeding 400°C. However, this procedure frequently results in nanoparticle agglomeration, which reduces the number of surface hydroxyl groups and surface area, both of which are critical for photocatalytic activity. Some research groups established methods for preparing TiO<sub>2</sub> nanoparticles at temperatures lower than 100°C without the requirement for further heat treatment, this type of reaction is always carried out in an acidic environment, with acetic, nitric, or perchloric acid present. At temperatures of 60-70°C, Andersson and Bard obtained needle-shaped anatase nanoparticles with an average radius of 8 nm, and at 90°C, they obtained needle-shaped anatase nanoparticles with an average radius of 8 nm. [22]

##### 4.1.1.2. Green Synthesis of TiO<sub>2</sub> Nanoparticles

The Aloe Vera plant extract is used in the green production of TiO<sub>2</sub> nanoparticles. Aloe Vera leaves were taken from the plant and properly cleaned before being chopped into little pieces. Boil 100ml distilled water for 2 hours at 90°C with 25 g of the leaves. What man filter paper was used to filter the extract. The filtrate was kept in order to make nanoparticles. Dissolve 1.0 N Titanium Chloride (TiCl<sub>4</sub>) in 100 mL Millipore water to make TiO<sub>2</sub> nanoparticles. Drop by drop, stirring constantly, the leaves extract was added until the pH of the solution reached 7. Continuous stirring was applied to the mixture for 4 hours. This technique produced nanoparticles, which were then separated using man-made filter paper, and the materials were repeatedly washed with water to remove the by-products. The nanoparticles were dried overnight at 100°C and then calcined for 4 hours at 500°C. [23]

##### 4.1.2. Applied of Nano titanium on cotton fabrics

Cotton materials have gotten a lot of attention in recent decades because of its distinctive benefits, such as comfort, softness, and breathability, which have been applied to a variety of industries, including medicine, military, clothes, and wearable technological devices. Cotton fabrics must improve their qualities and functionality for use in children's clothing and medical bandage cloths. As a result, it is critical to protect such clothing from harmful UV



rays, as well as to reduce the transmission of harmful microorganisms and the spread of secondary infections within a curative setting. High-performance textile materials have been manufactured utilizing nanotechnology in textile finishing in recent years, and new qualities have been imparted as a result, increasing the added value of the completed products when compared to traditional finishing agents. [24-29]

#### 4.1.2.1. Antibacterial property

Cotton cloth is a common natural fabric. Cotton fibres are polymers made up of thousands of glucose residues connected together by glycosidic linkages. Cotton fabric has excellent air permeability and a pleasant softness, making it a requirement in everyday life. Cotton fabric, on the other hand, has hydrophilic groups on its surface, such as hydroxyl groups, which may absorb moisture. When individuals use cotton fabric, it absorbs moisture quickly and breeds bacteria, lowering the fabric's performance. As a result, it is important to apply a long-term antibacterial treatment to cotton fabric, which broadens the application range and extends the fabric's service life. The lack of an antibacterial ingredient on the surface of raw cotton could be the explanation. *S. aureus*, *E. coli*, and *C. albicans* were reduced by 27 percent, 35 percent, and 12 percent, respectively, in the nano-TiO<sub>2</sub>-treated cotton sample. In the presence of natural dye, nano-TiO<sub>2</sub> treated cotton showed better antibacterial activity than nano-TiO<sub>2</sub> untreated cotton. The antibacterial activity of treated cotton garments increased dramatically after walnut shell dye was added to nano titanium dioxide. The observed increase in antibacterial activity of treated samples could be explained by the antimicrobial activity of green walnut shells, which is unique. The presence of phenolic and naphthoquinone chemicals in walnut shells improved antibacterial activity significantly. In the presence of the walnut shell dye, the Nano-TiO<sub>2</sub> treated sample showed a 100 percent reduction in both *S. aureus* and *E. coli*. However, the sample's antifungal efficacy against *Candida albicans* was low. The prevalence of *Candida albicans* was 87 percent. This could be owing to the thickness of different microorganisms' cell walls. [30-33]

#### 4.1.2.2. Self-cleaning property

Titanium dioxide nanoparticles have photo catalytic activity, which allows them to destroy color stains. When a photo catalyst like TiO<sub>2</sub> is irradiated with light that has a higher energy than its band gaps, it forms electron-hole pairs that cause redox reactions at the photocatalyst's surface. Can titanium dioxide nanoparticles, for example, disintegrate common organic materials, color molecules, and bacterial cell membranes. [34]

#### 4.1.2.3. UV protection

For functional textiles, the UV blocking protection factor is an important metric. The UV blocking properties of cotton fabrics treated with nano-TiO<sub>2</sub> coatings were good. [35]

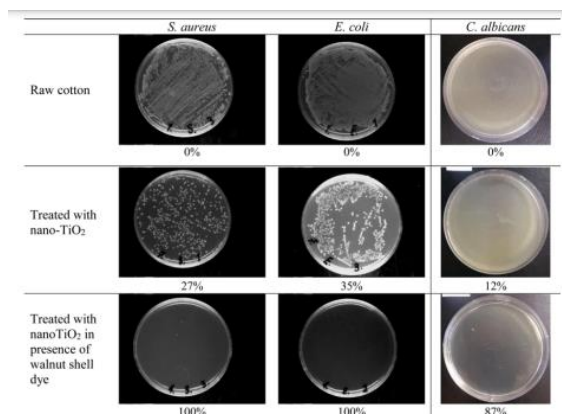


Fig 3: Antimicrobial activity measurement of raw and treated cotton fabrics

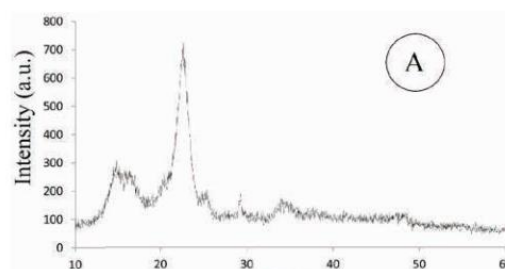


Fig 4: XRD patterns of treated cotton fabric with 5 ml titanium isopropoxide

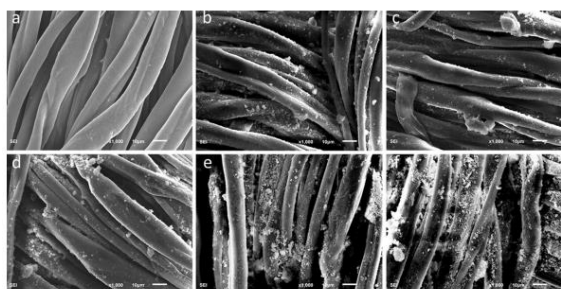


Fig 5. SEM micrographs of the functional cotton fabrics treated with composite coatings of nano-TiO<sub>2</sub> before washing

#### 4.1.3. Applied Nano titanium on wool fabrics

Wool is a textile material known for its durability, warmth, water resistance, and texture. However, this natural fabric made from the protein keratin lacks the stain resistance of synthetic fabrics and is prone to rigorous processing. [36]

##### 4.1.3.1. UV protection

UV rays, which are a component of the sunlight that reaches the earth, have some negative effects on

fabrics. UV radiation has a significantly higher energy per photon than visible light or infrared radiation, despite its lower intensity. UV rays, which have a high destructive intensity, cause a variety of ailments in people, including skin cancer, eye difficulties, erythema, and sunburn, to mention a few. As a result, it appears that UV protective clothing is required. Wool is an example of a natural fabric with a low photostability. Covering the surface of wool with nano TiO<sub>2</sub> particles is a novel effective technology that has only recently been developed. Nano TiO<sub>2</sub> particles have been applied to fabrics using a variety of procedures and chemicals. The deposition of nano TiO<sub>2</sub> particles on the surface of wool keratins using a sol-gel process has proven to be an excellent UV protection method. [10]

#### 4.1.3.2. Wettability property

Wool is hydrophobic, making it one of the most essential animal fibres. It was discovered that increasing the number of TiO<sub>2</sub> nanoparticles on the surface of wool enhanced its hydrophilicity and allowed water droplets to spread more quickly. Electrons and holes play a key part in this process, as they do in photocatalysis, by activating a sequence of reduction and oxidation processes. Negative electrons and positive holes are involved in the reduction of Ti<sup>4+</sup> to Ti<sup>3+</sup> and the production of oxygen anions (O<sub>2</sub>) in this fashion. Super oxide anions are converted to oxygen molecules by a sequence of processes, and the residual oxygen vacancies are expelled, boosting fabric wettability. [10]

#### 4.1.3.3. self-cleaning property

Protein fibres, which can convert light into self-cleaning power to breakdown stains, grime, and hazardous microbes in a photo-catalytic purification process, are promising materials for a variety of applications. Fabrics with self-cleaning properties have a layer of TiO<sub>2</sub> on their surface and, as a result, clean themselves. TiO<sub>2</sub>'s photocatalytic property aids in the decomposition of organic compounds that come into contact with the surface, preventing them from accumulating. [37, 38]



Fig.6: Oxidized samples treated with 0.075 g/l TiO<sub>2</sub> and stained by fruit juice

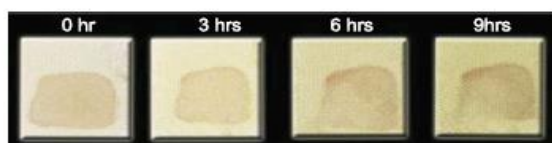


Fig. 7: Untreated raw wool stained by fruit juice.

#### 4.1.4. Applied Nano titanium on cotton/polyester blend fabrics

Because of their low cost, biodegradability, and sustainability, natural fibres are the preferred choice for apparel. Cotton has been a popular natural material since antiquity due to its soft natural feel, high absorbency, and high breathability. Cleaning, drying, and ironing cotton items, on the other hand, takes 72 % more than synthetics. They are also weak, have a short lifespan, are easily flammable, and have a low wrinkling resistance. Chemical treatments to improve cotton fabric qualities result in high processing costs, huge effluent loads, and tensile strength loss. Polyester and other synthetic fibres have superior mechanical, durability, and wrinkle resistance. [39] These synthetic fibres, on the other hand, offer disadvantages like as static charge build-up and reduced breathability. As a result, tremendous efforts have been made to produce novel fibres that can be used in a variety of applications. As a result, a lot of work has gone into developing novel fibres that have the desirable qualities of both natural and synthetic fibres. [40]

##### 4.1.4.1. Antibacterial property

They discovered that pre-treatment of cotton/polyester blend fabric with cold plasma for 6 minutes before use of TiO<sub>2</sub> NPs improved antibacterial activity against *S. aureus* and *E. coli* bacteria, increased antistatic property, and improved NP adherence in the fabric surface after 50 washes. The treated fabric's elongation, tensile strength, and air permeability all decreased slightly. [33, 41]

#### 4.2. Zinc oxide nanoparticles (ZnO-NP)

Because of its outstanding photo-catalytic, electrical, electronics, optical, dermatological, and anti-bacterial capabilities, zinc oxide has been the most popular nano particle among other varieties. It also has three distinct properties: semi conductivity, piezoelectricity, and bio safety compatibility. Because of its outstanding photo-catalytic, electrical, electronics, optical, dermatological, and anti-bacterial capabilities, zinc oxide has been the most popular nano particle among other varieties. [42]

ZnO has been extensively studied in the textile sector because ZnO-NP has good UV blocking, antibacterial, photocatalytic self-cleaning, hydrophobic, and flame-retardant qualities when added to textiles. [7, 19, 28, 33, 38, 43-46]

##### 4.2.1. Synthesis of ZnO nanoparticles

Ex situ and in situ synthesis are the two main ways for chemically producing ZnO-NP and applying it to textiles. The ex situ process is a two-step procedure in which the synthesis of ZnO-NP is carried out as a separate reaction between Zn-precursor and precipitating/reducing agent, followed by calcination, and the dispersion of formed ZnO-NP

is prepared in a separate bath and coated on textiles in the second step. [19]

#### 4.2.2. Green Synthesis of ZnO Nanoparticles

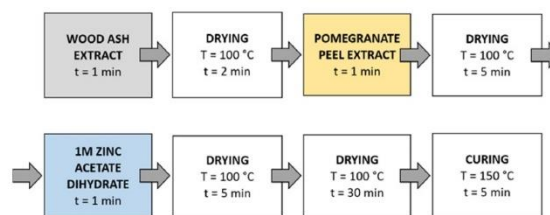
On cotton fabric, green in situ production of zinc oxide nanoparticles (ZnO-NP). For the synthesis of ZnO-NP from zinc acetate, pomegranate peel extract was employed as a reducing agent and wood ash extract as an alkali source. There are four main synthesis processes, each with its own set of advantages and disadvantages, The most acceptable method for achieving outstanding ultraviolet (UV) protection qualities of the functionalized textile was evaluated by comparing the drying times of immersion of fabric in active solutions for synthesis, padding, and ultrasonication. [47]

According to the research, Keliab, which is made up of the ashes of burnt leaves and stems of the *Seidlitzia Rosmarinus* plant and contains a lot of sodium and potassium carbonates, was utilized as an alkali source. Zinc acetate dihydrate solution was used as a zinc precursor in the in situ synthesis of ZnO-NP on raw cotton fabric. For 30 minutes, the cloth was soaked in the precursor solution while being constantly stirred. The Keliab solution was then slowly poured into the synthesis bath, which was then heated to 90°C. For the next 60 minutes, the synthesis process was continued. For 30 minutes, the cloth was soaked in the precursor solution while being constantly stirred. The Keliab solution was then slowly poured into the synthesis bath, which was then heated to 90°C. For the next 60 minutes, the synthesis process was continued. For 30 minutes, the cloth was soaked in the precursor solution while being constantly stirred. The Keliab solution was then slowly poured into the synthesis bath, which was then heated to 90°C. For the next 60 minutes, the synthesis process was continued. The samples were then dried for 30 minutes at 80 degrees Celsius and then cured for 3 minutes at 150 degrees Celsius.

When compared to untreated samples, the functionalized samples demonstrated improved antibacterial characteristics against *Staphylococcus aureus* and *Escherichia coli*, increased tensile strength, and better crease recovery. Despite the positive results, the synthesis procedure was time and energy intensive, as it was carried out for 90 minutes at high temperatures. Furthermore, the plant utilized to make natural extract is not a waste product and can be used for other things. This approach is not a good illustration of green circular economy since if it were done on a large scale, the demand for growing this plant would skyrocket. Extracts from nature The fruit of the pomegranate fruits were separated from the seeds, then washed, chopped into little pieces, and air dried. Using a household blender, the dried peels were ground into powder. 100 g/L of powder was immersed in bi-distilled water to make the extracts.

The solution's temperature was raised to boiling point, kept for 5 minutes, and then allowed to cool for two hours. The solution was then centrifuged at 4000 rpm for 10 minutes to separate the solid particles from the liquid, which was further filtered using PES mesh to remove any leftover particles, yielding pure liquid extracts. The extracted materials were promptly put to use. We used a natural extract made from wood ash waste to assure an alkaline medium for the in situ synthesis of ZnO. The ash from commercial wood pellets that were burned for the heating system was collected and utilized to make water extract without any further modification or purification. By dissolving 10 g/L of ash powder in bi-distilled water, wood ash extract was created. After 5 minutes, the mixture was vacuum filtered. [11]

The samples were submerged in a wood ash extract for 1 minute before drying for 2 minutes in a continuous dryer. The samples were then submerged for 1 minute in pomegranate peel extract and dried for 5 minutes in a continuous dryer. The samples were then submerged for 1 minute in a 1 M zinc acetate dihydrate solution and dried for 5 minutes in a continuous dryer. The samples were then dried in a laboratory oven at 100 degrees Celsius for 30 minutes and 150 degrees Celsius for 5 minutes.



This method was found to be the most appropriate, as it produced cotton fabric with excellent UV protective properties thanks to the uniformly distributed numerous ZnO wurtzite nanoparticles on its surface, and it has a lot of potential to help advance the development of environmentally friendly functional textiles, because it simultaneously addresses multiple problems such as the use of discarded by-products of food production and pellet heating without requiring expensive specialised equipment, reducing the environmental impact of traditional chemical textile functionalization and enabling the production of protective textiles that could be used in the clothing, medical, or technical textile sectors. [19]

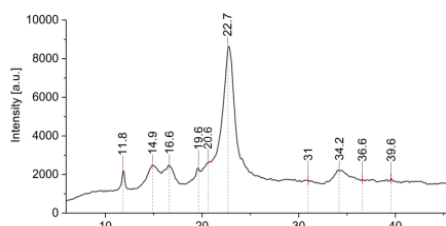


Fig.8: XRD pattern of cotton sample where suit Synthesis of ZnO nanoparticles



### 4.2.3. Applied ZnO (NPS) on cotton fabrics

#### 4.2.3.1. Antibacterial activities

The in-situ approach of synthesising ZnO NPs on cotton fibres was carried out according to a previously published procedure. To ensure the removal of leftover chemicals, the cloth was first washed in warm water using a nonionic detergent. The cloth was washed, rinsed with warm water, and dried in a 75°C oven for 60 minutes. It was then placed in a 100 mL solution of 0.005 M  $Zn(NO_3)_2 \cdot 6H_2O$ . After 15 minutes, the reaction vessel was filled with 0.02 mol  $NH_4Cl$ , 0.01 M urea, and 5 mL ammonia solution. The system was rapidly heated to 90°C (10°C min<sup>-1</sup>) and held under magnetic stirring (300 rpm) for 60 minutes. Following the completion of the reaction, the fabric was rinsed multiple times with distilled water before being washed. The antibacterial activity of in situ produced ZnO NPs on cotton fabrics was tested using the agar diffusion method against both gram-negative (*E. coli*) and gram-positive (*S. aureus*) bacteria. Cotton textiles with a diameter of 20 mm were uniformly pressed on the agar and incubated at 37°C for 24 hours. The antibacterial action of the textiles was assessed after incubation by measuring and recording zones of inhibition produced around the discs in millimetres (mm). [48]

#### 4.2.3.2. Thermal properties

Chemical compounds and polymers that are appropriate for each fibre type, fibre blend, fabric weight, and construction are used in flame-retardant textile treatments. The flammability properties of fibre, cellulosic burns easily with a glow followed by the creation of char, whereas wool only tolerates combustion with difficulty. Synthetic fibres may slowly melt without igniting (nylon 6 and 66), Burns and melts quickly (polyester and acrylic). The thermal processes and combustion of organic polymers, whether in fibre film or other forms, occur in a progressive and definable order, with thermal degradation occurring before the material's ignition and combustion. The polymer may burst into flame, melt, shrink, char, or thermally degrade without flame after combustion. Flame retardants work by preventing combustible products from forming and/or altering the typical distribution of breakdown products emitted by the original material. Textiles have been treated using a variety of finishing processes to make them flame resistant. Coating, graft or homo polymerization, cross-linking, covalent bond formation, and other processes are examples. [48]

cotton samples were treated with Nano ZnO was generated in four distinct concentrations (0.25, 0.5, 1, 2 percent). In a 1:1:0.5 ratio, ZnO and propanol are added to 100 mL deionized water with acrylic binder for each concentration. Strips of samples were dipped and soaked for 10 minutes in the prepared solutions

with a liquor ratio of 1:100 under magnetic stirring, then padded to guarantee complete pick up. The padded samples were dried for 15 minutes at 100°C, then thermofixed for 2 minutes at 150°C. Finally, the samples were washed for 5 minutes with 2 gm/L sodium lauryl sulphate, rinsed in distilled water, and air dried. The cotton samples' crystal properties improved the most at [ZnO] 0.25 percent, followed by a decrease. [49]

### 4.2.4. Applied Nano zinc oxide on wool fabrics

Zinc oxide (ZnO) is a widely used nanoparticle having photocatalytic activity under light irradiation in a variety of sectors, particularly textiles and polymers. The nanoparticles in question are semiconductors. the effect of nanoparticles on fabrics due to the fact that in alkali media, wool has a negative charge while zinc oxide (ZnO) has a positive charge. Zinc oxide (ZnO) nanoparticles are functionalized on wool fabrics to achieve outstanding UV-blocking and photocatalytic properties, as well as making the textile antimicrobial. [50]

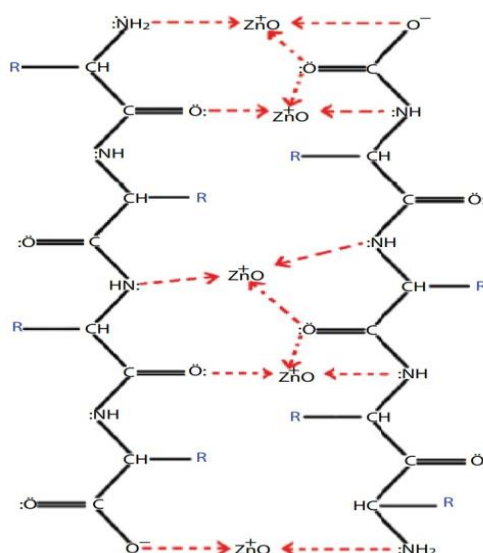


Fig.9: Nano cross-linking of wool protein chains with nano ZnO

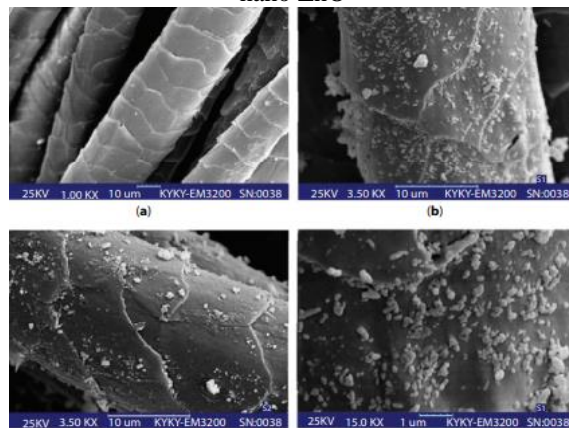


Fig.10: SEM images of (a) untreated wool fabric and (b, c, d) wool fabrics treated with nano ZnO

#### 4.2.5. Applied Nano zinc oxide on cotton/ polyester blend fabrics

Cotton /Polyester blended fabrics have successfully been treated with nano finishes. The aspects to consider in nano finishing synthetic textile materials have been odour elimination, antistatic, and antibacterial properties. Antimicrobial properties of zinc oxide (ZnO) nano particles have been employed. Cotton/polyester samples perform better than 100% cotton samples because polyester mix samples are less sensitive to microbial development than 100% cotton samples. [42]

##### 4.2.5.1. Antibacterial property

The Exhaust –dry –cure procedure was used to finish the cotton/polyester blended fabric samples with Zinc Oxide nano particles of 30 nm and 90 nm. For 15 minutes, samples measuring 30 × 30 cm were cut and submerged in a solution containing 2 percent ZnO of 30 nm size and 1 percent Acrylic binder at an MLR of 1:20. The samples were then taken out and squeezed in two bowl padding mangles at 60% expression to eliminate surplus liquor before being air dried. To remove unfixed particles, the samples were cured at 140°C for 2 minutes and rinsed with 2 g /l Sodium Lauryl Sulphate. The samples were then washed in water multiple times to remove the soap solution. To make a comparison, samples were also finished with 90 nm ZnO particles. Against both Gram-positive and Gram-negative species, samples finished with 30 nm ZnO particles exhibit a higher bacterial reduction % than those finished with 90 nm ZnO particles. This is owing to the fact that nanoparticles with a diameter of 30nm have a larger surface area to volume ratio than those with a diameter of 90nm. This is consistent with previous observations that smaller particles have more surface area and hence more interaction with bacteria, resulting in greater bactericidal and fungicidal efficacy. [42, 49]

#### 4.3. Copper nanoparticles

It has intriguing properties like good electrochemical activity, a large specific surface area, a correct redox potential, and great solution stability. The optical, electrical, magnetic, and mechanical properties of CuO nanoparticles are diverse, controlling their size and morphology can demonstrate this. CuO nanoparticles have drawn more interest from researchers due to their potential utility in a variety of applications, and they have become one of the most often utilized transition metal oxides. This can be demonstrated by manipulating their size and morphology. [36]

##### 4.3.1. Synthesis of copper nanoparticles

Copper nanoparticles contain optical, catalytic, and chemical capabilities that are unique to the nanoscale. Using copper (II) sulphate pentahydrate as a precursor salt and starch as a capping agent, Cu

nanoparticles were created using a chemical reduction method. The procedure begins with vigorous swirling of 0.1 M copper (II) sulphate pentahydrate solution into 120 mL of starch (1.2 percent) solution for 30 minutes. Under continuous quick stirring, 50 mL of 0.2 M ascorbic acid solution is added to the synthesis solution in the second step. After that, 30 mL of 1 M sodium hydroxide solution was progressively added to the produced solution while it was heated at 80 C for 2 hours with constant stirring. Under continuous quick stirring, 50 mL of 0.2 M ascorbic acid solution is added to the synthesis solution in the second step. After that, 30 mL of 1 M sodium hydroxide solution was progressively added to the produced solution while it was heated at 80 C for 2 hours with constant stirring. The solution's colour changed from yellow to ocher. After the reaction was completed, the solution was removed from the heat and allowed to settle overnight before the supernatant solution was carefully discarded. The precipitates were filtered out of the solution and washed three times with deionized water and ethanol to remove the excess starch linked to the nanoparticles. The resulting ocher-colored precipitates are dried at room temperature. Nanoparticles were dried and stored in a glass vial for later investigation. [51, 52]

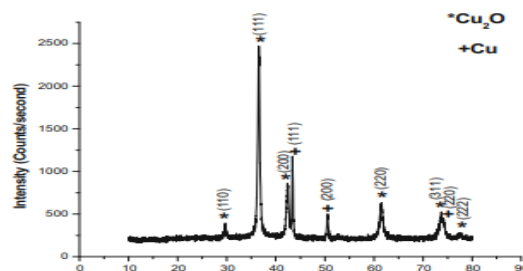


Fig.11: XRD pattern of Cu and Cu<sub>2</sub>O nanoparticles

##### 4.3.2. Green synthesis of copper nanoparticles

According to a previous report, black tea leaf extracts were used to synthesise copper nanoparticles (NPs). Tea leaf extract was made by precisely weighing 10 g of tea leaves and transferring them into a 250 mL conical flask already holding 100 mL of DI-H<sub>2</sub>O. After that, the combinations were heated for 20 minutes at 80°C, cooled, and filtered through Extract was then filtered number. 1 filter paper. Filtrates were kept at 4°C for one week and then used. CuSO<sub>4</sub> solution with the matching tea leaf extract was used to make copper nanoparticles from black tea (g-Cu NPs). CuSO<sub>4</sub><sup>1</sup> (1 mmol/L) and tea leaves extract were combined in a 4:1 volume ratio and stirred continuously for 10 minutes at 80°C. The resulting suspensions were left at room temperature for 24 h to complete reaction and separated using the above-mentioned protocol. [53]

### 4.3.3. Applied Nano copper oxide on cotton fabrics

#### 4.3.3.1. Antibacterial property

The use of copper oxide nanoparticles (CuO NPs) on cotton fabric to improve antibacterial and hydrophobic qualities. The fabric's hydrophobicity and mechanical qualities were also assessed using electron microscopy and a universal testing machine after the nanoparticles were dispersed onto it. The fabric's hydrophobicity and mechanical qualities were also assessed using electron microscopy and a universal testing machine after the nanoparticles were dispersed onto it. Treated cotton fabric has a higher tensile strength (32 MPa) than untreated cotton fabric (27 MPa), and copper nanoparticle-coated cotton fabric is relatively hydrophobic. Furthermore, bactericidal efficacy of CuO NPs-treated and untreated cotton fabrics against various gram-negative and gram-positive microorganisms was tested. Finally, the CuO NPs-coated cotton fabric shows stronger antibacterial activity against *E. coli* and superior antimicrobial activity even after 30 cycles of washing, indicating that it has a higher potential to be used as a medical textile to prevent cross-infection in a clinical setting. [53-57]

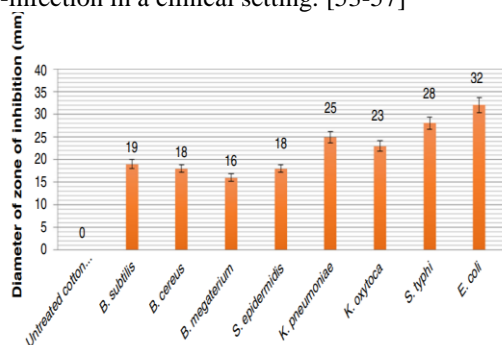


Fig.12: Diameter of the zone of inhibition of cotton fabric treated with CuO NPs

#### 4.3.3.2. Photocatalytic properties

Metal oxide semiconductors are incorporated onto textile substrates to produce textiles with photocatalytic or self-cleaning characteristics. This means that textiles containing semiconductor nanoparticles can oxidize the color of the dye solution or stains when exposed to UV or sunlight. Under day or UV light, copper oxide nanoparticles as a semiconductor have a high potential for degrading various dyes such as Cationic Dyes, Methyl orange, and Methyl red (MR). The photocatalytic characteristics of cotton fabric treated with copper oxide nanoparticles against methylene blue staining were also described. The presence of copper oxide nanoparticles as metal oxide semiconductors on the surface of cotton fabric accounts for the completed cotton sample's good photocatalytic performance. These nanoparticles can be stimulated by exposing them to UV or sunlight, causing the electrons in the valence band to hop to the conduction band, resulting in the formation of positive holes. Hydrogen peroxide

and hydroxyl radicals are formed when holes and electrons combine with water molecules and oxygen. Finally, the presence of hydroxyl radicals in the atmosphere causes methylene blue to degrade via oxidation to harmless materials such as inorganic acids,  $H_2O$ , and  $CO_2$ . [58]

### 4.3.4. Applied Nano copper oxide on wool fabric

#### 4.3.4.1. Antibacterial activities

Wool modification has gotten a lot of attention lately, thanks to its massive output and wide range of uses. Antibacterial characteristics are increasingly important in today's world; as a result, antibacterial wool materials have sparked a surge in popularity in recent years. In situ synthesis and deposition of different nanoparticles on fabrics has recently been introduced as a simple and effective method for obtaining antibacterial textiles, which is now being investigated by numerous researchers. The antibacterial properties of nanocolored wool fabrics containing cupric oxide nanoparticles are outstanding. When pathogen bacteria come into touch with loaded cupric oxide nanoparticles on wool fibers, they can get infected. As a result, during the oxidation of lipids and proteins, copper ions can be liberated from the surface, damaging the cell membrane with reactive hydroxyl radicals. With their nanoscale diameters, they can easily adhere to amine and carboxyl groups of cell bacteria, preventing biochemical processing of bacteria. Finally, they infiltrate the bacterial cell, burst it, and induce cell death. [59, 60]

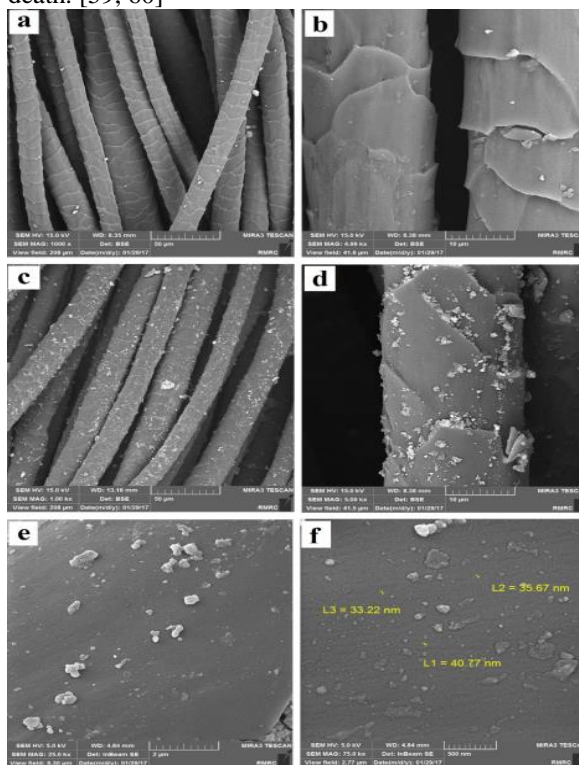


Fig.13: FESEM images of (a, b) untreated and (c-f) treated wool samples with cupric oxide nanoparticles in different magnifications.

#### 4.3.5. Applied Nano copper oxide on cotton/polyester blend fabrics

Copper nanoparticles were applied to polyester-cotton blend fabrics using a pad-dry-cure process. The K/S and color attributes of the nano copper treated woven fabric were improved when it was colored with a natural dye. Even after 30 washing cycles, the cloth coated with copper nanoparticles demonstrated effective antibacterial action against *S. aureus* and *E. coli*. Strong chemical bonds were used to adhere the copper nanoparticles to the cloth, resulting in outstanding endurance. [61]

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