Green Synthesis of ZnO and SiO_2 Nanoparticles and its potential application on Pineapple Fabric to impart multifunctional properties

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Research Article

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Abstract

The present study aims to synthesize ZnO and SiO_2 nanoparticles by using the green technique and applied to pineapple fabric to impart multifunctional properties. ZnO nanoparticles were synthesized by using Zinc Acetate where the mixture of Acalypha Indica and Citrus aurantifolia peel powder was used as the catalyst. Also, SiO_2 nanoparticles were synthesized from sugarcane bagasse which was the primary source of silica. Final SiO_2 nanoparticles were obtained by the addition of NaOH and HNO_3. Afterward, the synthesized particles were coated on the pineapple fabric by using the dip-pad-dry-cure method along with the addition of 2 wt% acrylic binder. The structural properties as well as the microstructure and chemistry of these coated samples were characterized by Fourier Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscopy (SEM) analysis. Also, the UV resistance, resistance against microorganisms, and super-hydrophobicity properties were evaluated by UV/VIS Spectrophotometer analysis, antimicrobial test, and spray rating test accordingly where the nanoparticle coated sample showed a very good absorbency in UV rays, superhydrophobic properties, and a very good resistance against microorganism maintaining optimum tensile.

1.0 Introduction

The synthesis of nanoparticles through eco-friendly and sustainable methods has gained significant attention in recent years [1]. This green synthesis approach offers several advantages, including non-toxicity, cost-effectiveness, and environmental friendliness. Natural sources are commonly utilized for nanoparticle synthesis, utilizing substances such as plant extracts, cyclodextrin, and chitosan. Metal oxide nanoparticles, including ZnO and SiO_2, are extensively used in textile applications due to their multifunctional properties [2]. Coating fabrics with these nanoparticles create nanostructures on the fiber surface, imparting properties such as UV protection, antibacterial activity, superhydrophobicity, and photocatalytic self-cleaning capabilities [3]. Various coating methods, including pad dry, sol-gel, plasma polymerization, and layer-by-layer techniques, are employed to apply nanoparticles to textile materials [4]. The coating process aims for even distribution and penetration of nanoparticles into the fabric structure to ensure effectiveness [5].

UV protection is a crucial requirement for textiles due to the harmful effects of UVA and UVB radiation, which can lead to skin cancer, premature aging, and other health issues [6]. TiO_2 is commonly used for photocatalytic degradation, but zinc oxide (ZnO) and zinc-based nanoparticles have also gained attention as effective alternatives due to their high UV resistance [7]. These nanoparticles can be coated on textiles to provide UV-shielding properties under UV light irradiation [8].

Textile materials treated with nanoparticles not only offer UV protection but also exhibit antimicrobial properties [9]. Nanoparticles such as Ag, ZnO, SiO_2, and TiO_2 are utilized for their sterilizing effects mediated by the production of reactive oxygen species (ROS) [10]. Care must be taken to avoid potential decolorization of dyes and damage to fibers caused by the high redox potential of these nanoparticles [11].

The development of superhydrophobic surfaces on textiles has gained significant interest, particularly for outdoor applications. Hierarchical nano-roughness, inspired by lotus leaves, can be achieved by depositing low-energy chemicals onto the textile surface [12]. Nanoparticles like ZnO, TiO_2, and SiO_2 are commonly employed to create multiscale surface roughness, enabling superhydrophobicity [13]. However, the photooxidative ability of ZnO under UV light can lead to a loss of superhydrophobicity, necessitating careful consideration in practical applications [14].

Pineapple fabric, derived from pineapple leaf fibers, offers sustainability and comfort but faces limitations such as durability, microbial growth, and inadequate UV resistance [15]. Incorporating green-synthesized ZnO and SiO2 nanoparticles onto pineapple fabric presents an opportunity to enhance its properties. The project aims to explore environmentally friendly synthesis methods, applying the nanoparticles to the fabric through coating or impregnation techniques. The desired outcomes include improved durability, antimicrobial activity, enhanced UV protection, and soil resistance while maintaining the inherent qualities of the fabric.

This research aims to the development of sustainable and functional textiles by integrating green synthesis, innovative material engineering, and eco-friendly production methods. The findings can drive advancements in the textile industry, offering a viable alternative to conventional fabrics. By promoting the use of eco-friendly materials and technologies, this project aligns with environmental sustainability goals and supports the transition toward a more sustainable and technologically advanced textile industry.

2.0 Materials and Methodology

2.1 Materials

The synthesis of ZnO and SiO nanoparticles involved the utilization of Acalypha Indica Leaf and Citrus Aurantifolia (lemon) Peel sourced from the Bagerhat district, Bangladesh, while Sugarcane Bagasse was collected from the Khulna Newmarket area, Bangladesh.

To ensure an environmentally friendly approach, we employed a minimal amount of chemicals and auxiliaries throughout the process. All the necessary Zinc Acetate, NaOH, HCl, HNO3, Ethanol Solution, and Acrylic Binder were acquired from the Khulna City Scientific Store, Bangladesh. To coat the nanoparticles, we utilized pineapple fabric, which is described in detail in Table 1.

Table 1
Construction of Fabric

Fabric type	**Woven Fabric**
Fabric condition	Pre-treated fabric
GSM	80
EPI	50
PPI	40
Yarn composition	80% cotton + 20% pulp

2.2 Methodology

2.2.1 Preparation of ZnO Nanoparticles

Initially, the Acalypha Indica leaves were carefully washed multiple times to eliminate any impurities they may have carried. Afterward, the leaves were left to dry for a few days. Following the drying process, the leaves were sorted, and only mature leaves were selected for further use. To obtain leaf powder, the chosen leaves were crushed in a blender which is shown in Fig. 1(a).

The Lemon peels collected were chopped and subjected to multiple rinses with clean water to ensure the removal of any contaminants. After chopping, the peels were washed and left to dry at room temperature. Once dried, the peels were ground to produce peel powder, shown in Fig. 1**(b).**

For the extraction process, 3 grams of fine leaf powder and 2 grams of peel powder were dissolved in 100 milliliters of distilled water. The mixture was then stirred for 30 minutes at a constant temperature of 80°C to create the extract. Subsequently, the extract was allowed to cool down to room temperature. Notably, no additional chemicals were added to the extract, which served as a catalyst for the synthesis of ZnO nanoparticles.

To synthesize the nanoparticles, 50 milliliters of the prepared extract were mixed with 2 grams of zinc acetate and stirred for 1 hour at a temperature of 70°C. The resulting precipitate was thoroughly washed multiple times with clean water and then heated in an oven at 80°C for 8 hours. Finally, the calcined ZnO nanopowders were subjected to a temperature of 500°C for 1 hour, resulting in the formation of ZnO nanoparticles.

2.2.2 Preparation of SiO_2 Nanoparticles

The sugarcane bagasse was soaked in distilled water for 8 hours and then thoroughly cleaned with distilled water to remove any dirt or impurities. Subsequently, it was dried in a 90°C oven. To eliminate any metallic contaminants, the sample was treated with 1M HCl. The mixture was immersed in a heated

water bath and then filtered. Multiple washes were performed to eliminate metallic ions, followed by another round of drying in a 90°C oven.

To obtain the sodium silicate solution, the sugarcane bagasse was submerged in a 1M NaOH solution for 30 minutes. The mixture was then heated in a water bath at 90°C for 1 hour. This process resulted in the formation of sodium silicate solution, as described by the Eq. (1)

$SiO_2 + 2NaOH = Na_2SiO_3 + H_2O$ ……..(1)

Next, the sodium silicate solution was rapidly stirred for 30 minutes. During the precipitation procedure, HNO_3 was added in small drops to the solution until the pH reached 8. Following that, 20 mL of ethanol was added. The solution was continuously stirred for 45 minutes and then centrifuged at 4000 rpm to generate the precipitate. The dried precipitate was collected and subjected to calcination in a furnace at 500°C for 1 hour to obtain silica nanoparticles.

2.2.3 Coating with ZnO and SiO2 Nanoparticles

To coat the fabric with ZnO nanoparticles, a precipitation aqueous solution was prepared, incorporating ethanol. The fabric was swiftly submerged in the solution, and the process was carried out for 10 minutes at a temperature of 40°C using an IRE machine. To enhance the adherence of the nanoparticles, 2% of acrylic binder was added to the precipitation solution. This method was chosen as the optimal approach for depositing ZnO nanoparticles. After the submersion, the fabric was padded twice and subsequently dried. It underwent a drying process for 10 minutes at a temperature of 90°C, followed by curing for 15 minutes at 120°C.

For the SiO_2 coating, the fabric previously coated with ZnO nanoparticles was immersed in a solution containing SiO_2 nanoparticles. It was then processed through an IRE machine at a temperature of 40°C for 10 minutes. Following the 10-minute treatment, a 2% acrylic binder was applied to enhance the adhesion of the SiO2 nanoparticles. The fabric was carefully removed from the SiO_2 nanoparticle dispersion and passed between two rolls of a padder to ensure uniform application of the nanoparticles. This procedure was repeated three times to achieve a consistent and even coating of the NPs. Afterward, the fabric was dried at a temperature of 90°C for 10 minutes and subsequently subjected to curing at 120°C for 15 minutes to complete the coating process.

2.3 Characterisation and Measurement

2.3.1 SEM image analysis

The scanning electron microscope (SEM) is a widely utilized tool for in situ micro area examination. It offers numerous advantages, including high resolution, a large depth of field, high magnification capabilities, and powerful stereoscopic vision. SEM photographs provide significantly greater magnification and superior definition compared to photographs obtained using optical microscopes.

The surface morphology of the fabric coated with nanoparticles was examined using a scanning electron microscope (SEM), specifically the Carl Zeiss Sigma 300 model, at a magnification range of 90–100 with a label of 200 micrometers and an accelerating voltage of 5 KV. This analysis was conducted at the Jashore University of Science and Technology in Bangladesh.

2.3.2 Fourier Transform Infrared Spectroscopy

FTIR spectroscopy utilizes infrared light to analyze and identify organic, polymeric, and inorganic materials. It is employed to determine the chemical properties of test materials. The primary applications of FTIR analysis include the identification and characterization of unknown substances (such as films, solids, powders, or liquids), detection of contamination within or on materials (such as particles, fibers, powders, or liquids), identification of additives extracted from polymer matrices, and detection of oxidation, decomposition, or uncured monomers in failure analysis investigations.

2.3.3 Tensile Strength Test

Tensile testing is a widely used method for evaluating materials, offering quick and reliable assessment globally. Compared to other time-consuming and specialized testing methods, tensile testing provides greater reliability in many cases. Testometric tensile testing devices offer efficient and precise data, ensuring excellent reproducibility and accuracy. For the tensile strength evaluation, Universal Testing Machine was used to apply controlled forces and measure the resulting mechanical response of the samples.

2.3.4 Spray Rating Test

The spray rating is a technique employed to evaluate the water resistance of fabrics, regardless of whether they have been treated with a water-repellent finish. However, it does not indicate the fabric's waterproofness or its ability to prevent water penetration. Water repellency is typically achieved through the application of substances such as paraffin, silicone, acid-based melamine, and fluorocarbons. Film coating or lamination techniques can also be employed to impart water-repellent properties to fabrics. The main objective of water repellency is to prevent textile materials from becoming wet when exposed to water.

2.3.5 UV Spectroscopy Test

UV-Vis spectroscopy is an analytical technique that examines the absorption or transmission of specific wavelengths of UV or visible light by a sample, in comparison to a reference or blank sample. It is a form of absorption spectroscopy that focuses on the ultraviolet (UV) range of light (200–400 nm), where molecules absorb energy and undergo electronic transitions from their ground state to higher energy states. The composition of the sample affects this characteristic, providing valuable information about the components present in the sample and their concentrations.

2.3.6 Antimicrobial Test

The disc diffusion or Kirby-Bauer test is a widely used and cost-effective method in microbiology for assessing antimicrobial resistance. It involves placing antibiotic discs onto an agar plate containing bacteria and allowing it to incubate. The presence of a clear area around the disc, known as a zone of inhibition, indicates that the bacteria are either inhibited or killed by the antibiotic.

To determine the antimicrobial properties of fabric samples, the **AATCC-147** standard was employed. The samples were tested against both gram-positive (Staphylococcus aureus) and gram-negative (Escherichia coli) bacteria, both of which are pathogenic. The bacteria were subcultured by incubating them at 37℃ overnight. Nutrient broth and agar media were prepared accordingly for the culture and sub-culture of the bacteria. Nutrient broth media was prepared in five conical flasks (100 ml each), while agar media was prepared in three separate conical flasks (two 500 ml and one 250 ml).

These preparations were carried out for a thesis project, where the focus was on testing the fabric samples for their antimicrobial effectiveness using the described methods.

3.0 Result and Discussion

3.1 Scanning Electron Microscopy Investigation

The SEM was employed to investigate the morphological structure of the nanoparticles-coated pineapple fabric. It was observed that the surface morphology of the raw fabric's cellulose, which is an amorphous and branched polymer, underwent significant changes after the coating process with nanoparticles. The presence of the nanoparticles resulted in a reduction of cellulose content in the processed fiber and an enhancement of the crystallinity of the coated fiber.

Upon examining the uncoated fabric sample, it was evident that the compactness between adjacent fibers of the uncoated pineapple fabric was lower, indicating the presence of a significant amorphous region. In contrast, the coated fabric sample exhibited improved compactness between the fibers, enhancing the tendency of the crystalline portion of the coated pineapple fabric. The coated sample demonstrated higher crystallinity compared to the uncoated sample, as revealed by the morphological structure analysis.

Furthermore, the morphological structure analysis indicated that the uncoated fabric had a higher proportion of amorphous region, while the coated sample showed a reduction in this amorphous region, leading to an improvement in crystallinity. The enhanced fiber compactness of the coated sample ultimately resulted in increased tensile strength.

In Fig. 5, the SEM images of the uncoated and coated samples were displayed, providing visual evidence of the differences in their morphological structures. It was crucial to verify the fibrous structure and physicochemical properties of the nanoparticle-coated fabric, along with the incorporation of the newly added multifunctional characteristics.

3.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FT-IR spectroscopy is an important analytical tool used to identify the composition of a product. Figure 6 displays the KBr-FTIR spectra of ZnO and SiO_2 nanoparticles coated on cotton-pulp fiber, covering a wavelength range of 4000 cm^{-1} to 500 cm^{-1}.

In the spectra, a prominent peak at 3474 cm^{-1} can be observed, which is characteristic of the hydroxyl (OH) groups present in cellulose. Another peak at 2908 cm^{-1} corresponds to the stretching vibration of the C-H bonds in cellulose. Notably, the absence of a peak at 1652 cm^{-1} suggests the absence of carboxyl groups typically found in hemicellulose.

For the ZnO nanoparticles, the absorption band at approximately 610 cm^{-1} is visible in the spectra, indicating the presence of Zn-O bonds formed during the synthesis process. Additionally, an absorption peak at 575.9 cm^{-1} represents the metal-oxygen vibration mode. The peak at 1094 cm^{-1} is attributed to the stretching vibration of the C-O bond in ethanol, which is likely present in the sample.

Regarding the cotton fabric coated with SiO_2 nanoparticles, peaks appear at 1054 cm^{-1}, which correspond to the vibrations of C-O-C and C-O bonds unique to the fabric. These peaks coincide with the amplitudes within the spectra. The Si-O-Si bending vibration generates sidebands at 784 and 526 cm^{-1}.

The FTIR analysis provides valuable information about the composition of the ZnO and SiO_2 nanoparticles coated on the cotton-pulp fiber. The peaks observed at specific wavenumbers indicate the presence of different functional groups and bonds in the synthesized materials, aiding in their characterization and identification.

3.3 Evaluation of Tensile Strength

Figure 7 illustrates the coating effect of nanoparticles on the tensile strength of the fabric. The figure demonstrates that the tensile strength of the nanoparticle-coated fabric is relatively higher when compared to the uncoated fabric sample. Specifically, the fabric coated solely with ZnO nanoparticles exhibited an approximate increase in tensile strength of 8.92% compared to the uncoated pineapple fabric. Similarly, the fabric coated solely with SiO2 nanoparticles showed a 5.96% increase in tensile strength compared to the uncoated fabric. However, the fabric coated with both ZnO and SiO2 nanoparticles displayed a significantly higher tensile strength of approximately 31.61% compared to the uncoated fabric.

In this study, it is hypothesized that the presence of ZnO and SiO_2 nanoparticles surrounding the fabric cellulose may form hydrogen bonds with the functional groups of cellulose, leading to an increase in tensile strength. Moreover, the coating process incorporated higher amounts of Zn and Si nanoparticles, which further contributed to the enhanced tensile strength. Importantly, the coating of ZnO and SiO_2 nanoparticles did not cause significant damage to the cotton fabric, thereby explaining the observed increase in tensile strength.

Based on these findings, it can be concluded that the coating of ZnO and Si nanoparticles, through potential hydrogen bonding with the cellulose, positively influences the tensile strength of the fabric. The presence of higher amounts of ZnO and SiO_2 nanoparticles in the coating process further enhances the tensile strength without causing detrimental effects to the cotton fabric.

3.4 Spray Rating Test (ISO Spray Tester)

The purpose of this method is to assess a fabric's ability to resist surface water, specifically its resistance to water droplets without penetration. In this test, a fabric specimen is positioned at a 45° angle beneath a spray nozzle, with a distance of 150 mm between the center of the nozzle and the center of the fabric specimen, as depicted in Fig. 8.

The test begins by pouring 250 mL of deionized water into a funnel at a controlled rate, ensuring a continuous flow of water from the spray nozzle onto the fabric specimen for 25–30 seconds. Following the water spray, the fabric and holder are removed from the test apparatus and tapped twice against a solid object to remove any excess water droplets. Subsequently, the fabric is visually evaluated and assigned a rating.

To rate the fabric performance, a comparative photographic scale is employed, as shown in **Fig. 8**. The International Organization for Standardization (ISO) standards define different numeric scales, such as ISO 0–5, each consisting of six possible ratings. Comparing the uncoated fabric sample with the numeric scale, it received a rating of 50 (ISO 1), indicating that the fabric sample was completely wet on the entire face of the specimen beyond the spray points. On the other hand, when comparing the coated fabric sample with the numeric scale, it obtained a rating of 90 (ISO 4), signifying slight random wetting of the specimen face.

This test evaluates a fabric's ability to resist surface water by subjecting it to controlled water spray and rating its performance based on a photographic scale. The uncoated fabric demonstrated greater water absorption, leading to a lower rating, while the coated fabric exhibited improved water resistance, resulting in a higher rating on the scale.

From Fig. 9,
100 No sticking or wetting of the specimen
90 Slight random sticking or wetting of the specimen face
80 Wetting of specimen face at spray points
70 Partial wetting of the specimen face beyond the spray points

3.5 UV-vis Spectroscopy

Figure 10**(a)** presents an absorbance vs. wavelength curve of an uncoated nanoparticle sample, while Fig. 10**(b)** displays the absorbance vs. wavelength curve of a sample containing nanoparticles. The main objective of this experiment was to compare the maximum absorbance values between these two samples.

Upon analysis of the graphs, it is evident that the absorption edge of the nanoparticle-coated sample occurred within the range of approximately 240–365 nm. In contrast, the uncoated sample exhibited lower absorbance at around 240 nm, after which the absorbance decreased. This observation leads to the conclusion that the nano-coating provided an improvement in UV protection and imparted excellent UV-blocking properties to the fabric. Conversely, the uncoated fabric samples exhibited inferior UV-blocking characteristics.

The absorbance vs. wavelength curves demonstrated that the fabric samples coated with nanoparticles exhibited a higher absorbance within the UV range, indicating enhanced UV protection. In contrast, the uncoated samples showed lower absorbance and therefore displayed inferior UV-blocking properties.

3.6 Visual Assessment of Antimicrobial Activity

In order to prevent fabric spoilage by micro-organisms and can be used as medical textiles, to prevent dermal infections or microbial growth, the antimicrobial test was done for both nanoparticles coated and uncoated fabric samples. After overnight incubation in the morning, all the media plates containing the test samples were observed. It was found that all the samples against the 2 types of bacteria showed very good anti-microbial resistance creating a huge clear visible zone around the treated fabric samples.

From Fig. 11, here all the samples were placed in the petri dish separately against 2 types of bacteria to check the bacterial activity. Nanoparticles coated and uncoated samples were checked whether the bacterial activity was working or not. All the samples have shown excellent antibacterial properties or bacterial resistance activity against those bacteria. And, also created a prominently visible zone of inhibition which is the proof of antibacterial activity of both fabric samples. From the figure, it is seen that no zone was created against bacteria for the uncoated sample. On the other hand, nanoparticle-treated fabric samples showed strong resistance against bacteria. The qualitative analysis concerning the area of the zone of inhibition is given in Fig. 11.

4.0 Conclusion

The objective of this experiment was to synthesize ZnO and SiO2 nanoparticles using green nanotechnology principles and apply them to coat pineapple fabric. The aim was to impart multifunctional properties, including UV blocking, antimicrobial activity, superhydrophobicity, and enhanced tensile strength.

The synthesized nanoparticles were subjected to characterization using scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR) to analyze their structural and chemical properties. The evaluation of the multifunctional properties of the coated fabric samples was conducted using ultraviolet-visible (UV-Vis) spectrophotometry to assess their UV-blocking ability, antimicrobial tests to determine their resistance against microorganisms and spray rating analysis to evaluate their superhydrophobic properties.

Based on the obtained data from these tests, it was observed that both the ZnO and SiO2 nanoparticle-coated samples exhibited remarkable resistance against UV rays, displayed antimicrobial activity, demonstrated superhydrophobicity, and maintained higher tensile strength. Importantly, these coated samples retained their inherent fabric properties while acquiring the desired multifunctional characteristics.

In summary, this experiment successfully synthesized ZnO and SiO2 nanoparticles and applied them to coat pineapple fabric. The coated samples exhibited excellent UV protection, antimicrobial properties, superhydrophobicity, and enhanced tensile strength, without compromising the original properties of the fabric.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Date availability

All the data are embedded in the manuscript.

Competing Interests

The authors declare that they have no conflict of interest.

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Authors' contributions

All authors collaborated in conceiving and designing the study. Md. Moniruzzaman originated the article's idea, conducted the literature review, and supervised the overall progress of the experiment. Syed Zubair Hussain established the experimental arrangement, executed the experiment, gathered and analyzed data, and wrote the complete manuscript. S M Atikur Rahman reviewed the manuscript in part, making necessary corrections, and also revised the figures. The entire author team actively engaged in critical revisions of the work.

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Figures

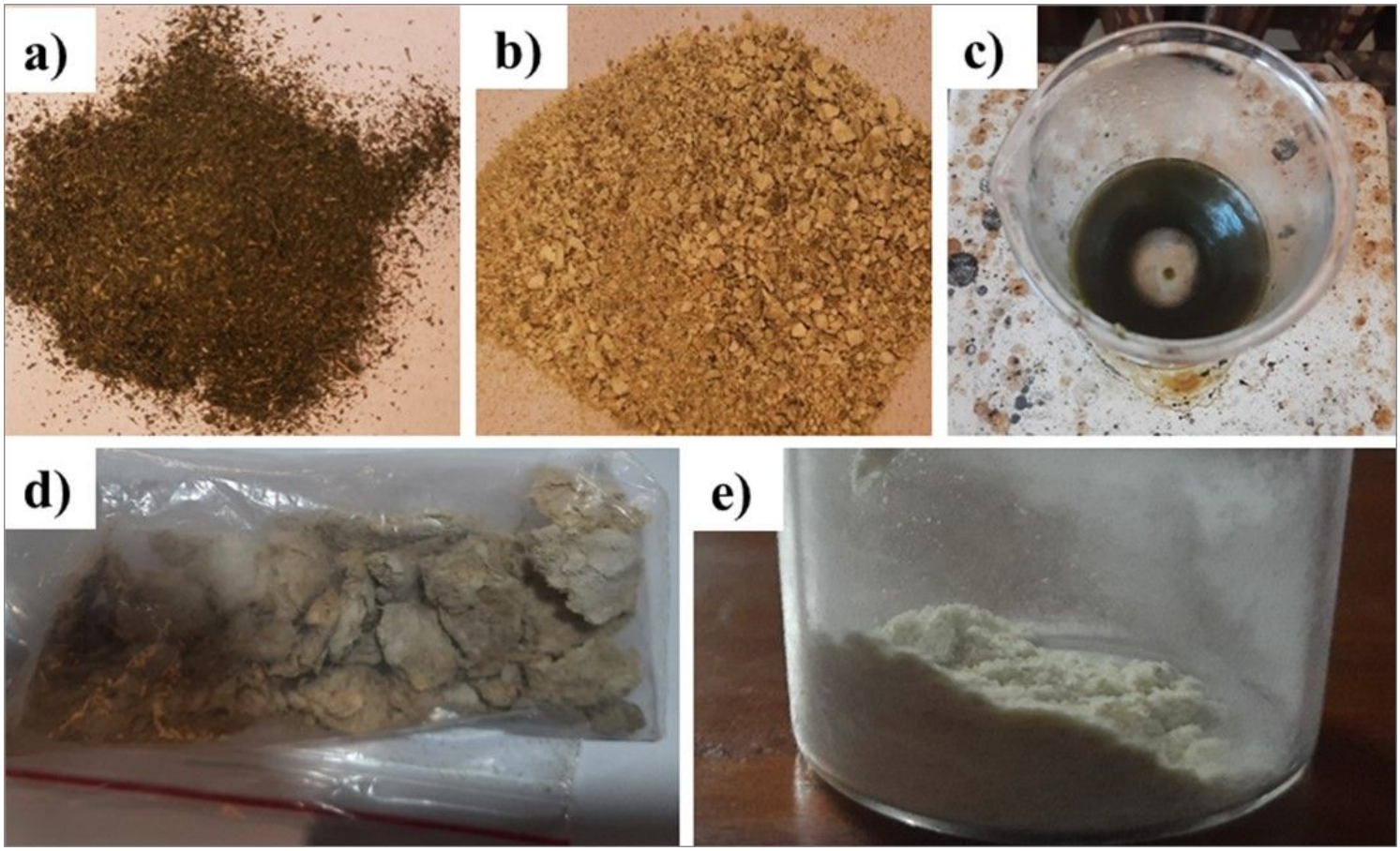

Figure 1

a) *Acalypha Indica* leaf Powder, b) Lemon Peel Powder, c) Mixture Stirring, d) Precipitation, after heating, e) ZnO Nano Particles

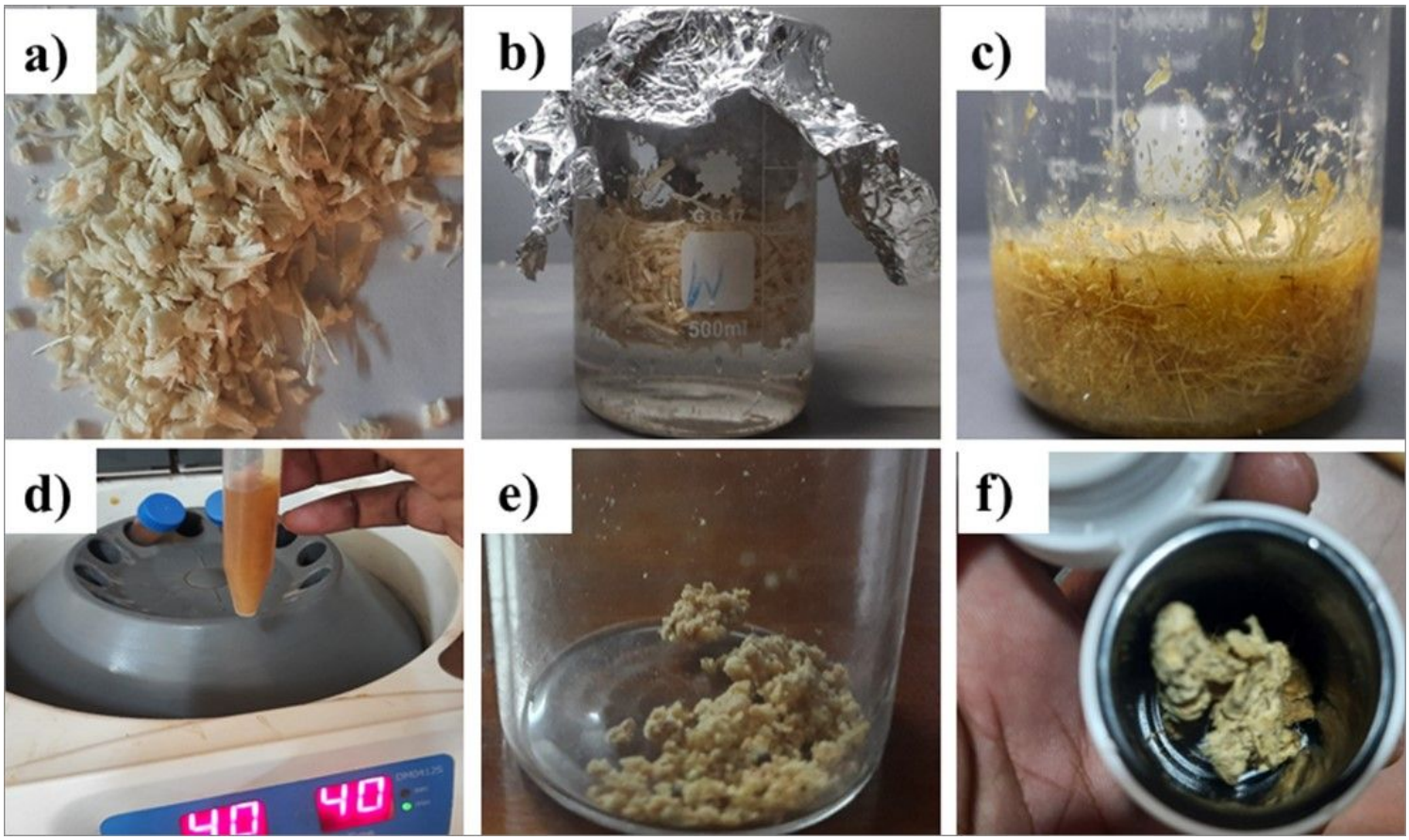

Figure 2

a) Sugarcane Bagasse, b) Bagasse soaked into Distill Water, c)HCl Mixed Sugarcane Bagasse, d) Centrifuging the Precipitate, e) Dried Precipitation, f) SiO2 Nano Particles

Figure 3

Universal Testing Machine

Figure 4

Spray Rating Test

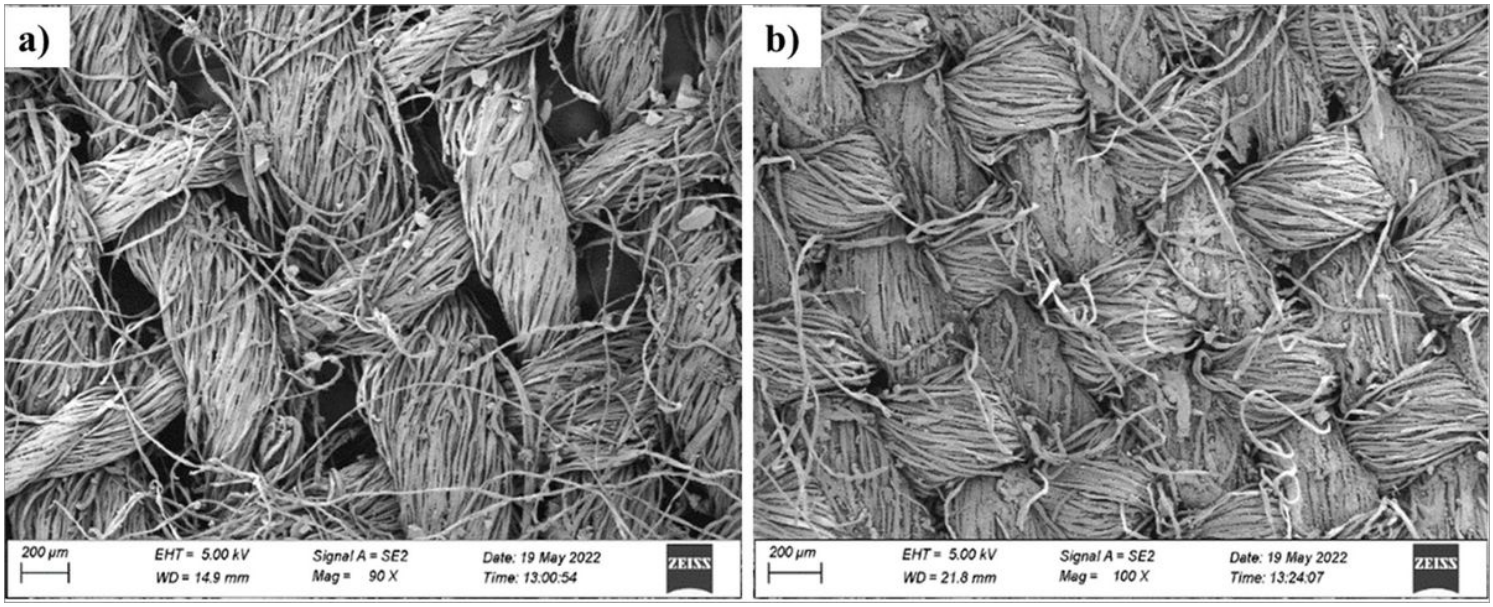

Figure 5

SEM image of (a) Uncoated and (b) Coated Fabric

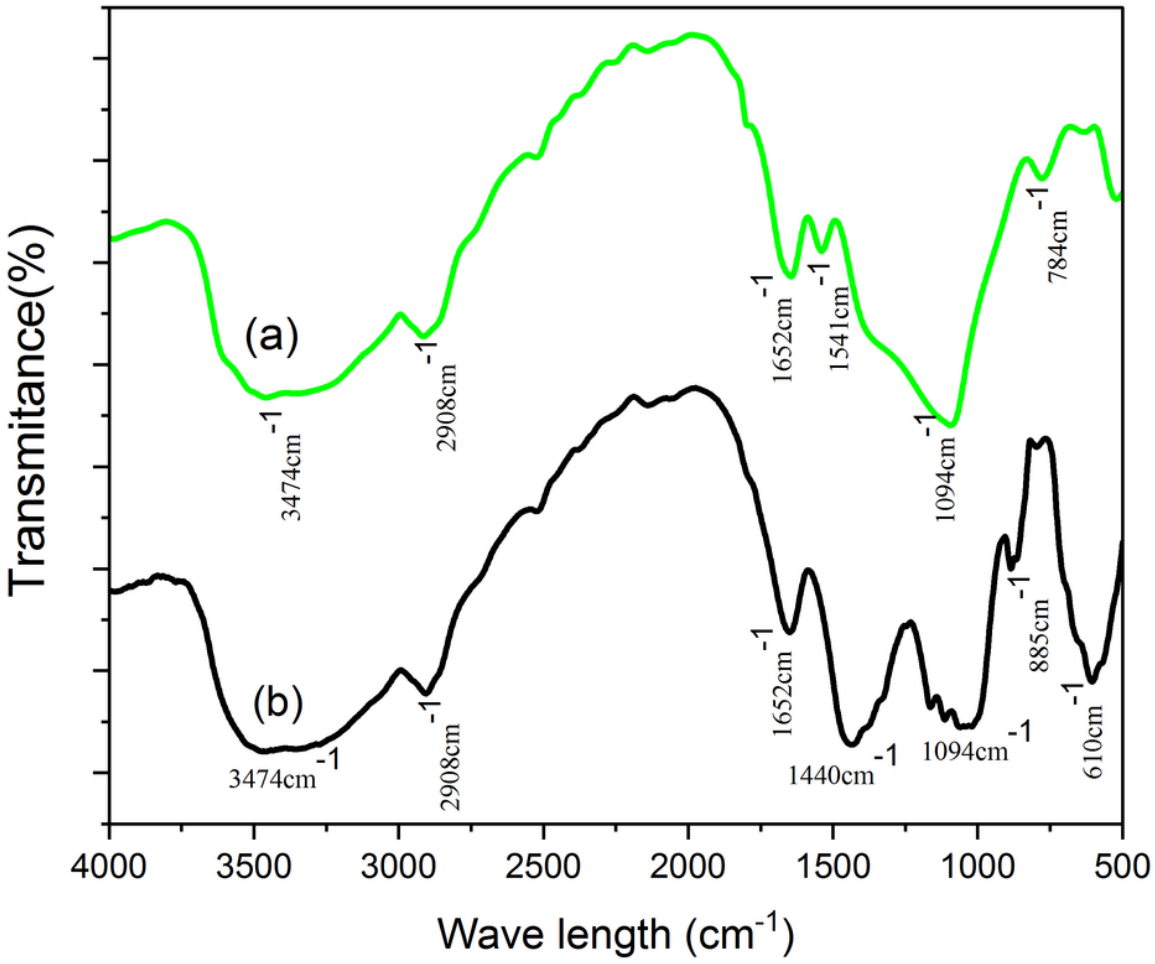

Figure 6

FTIR Spectra of (a) Silica (b) Zinc Coated Fabric

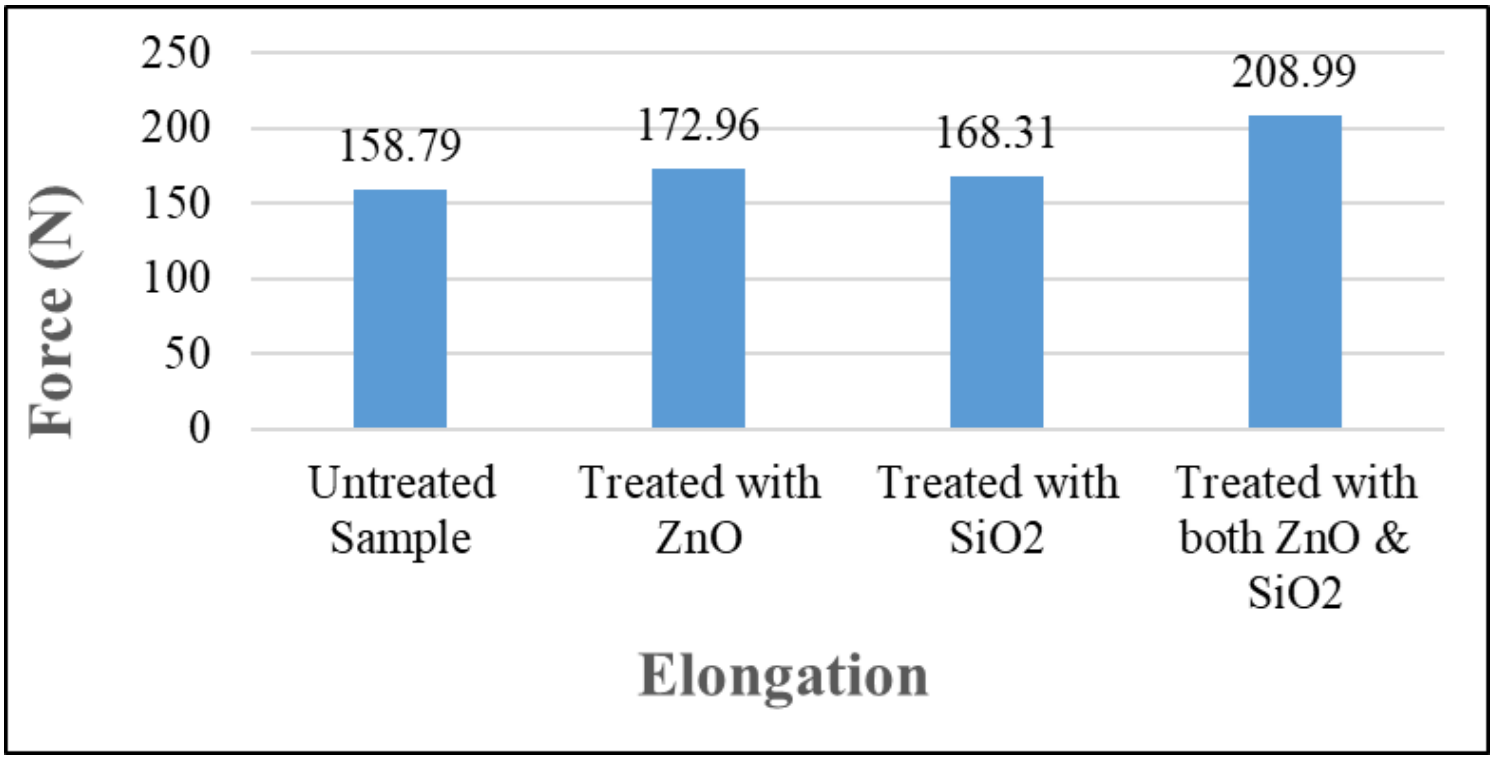

Figure 7

Evaluation of Tensile Strength

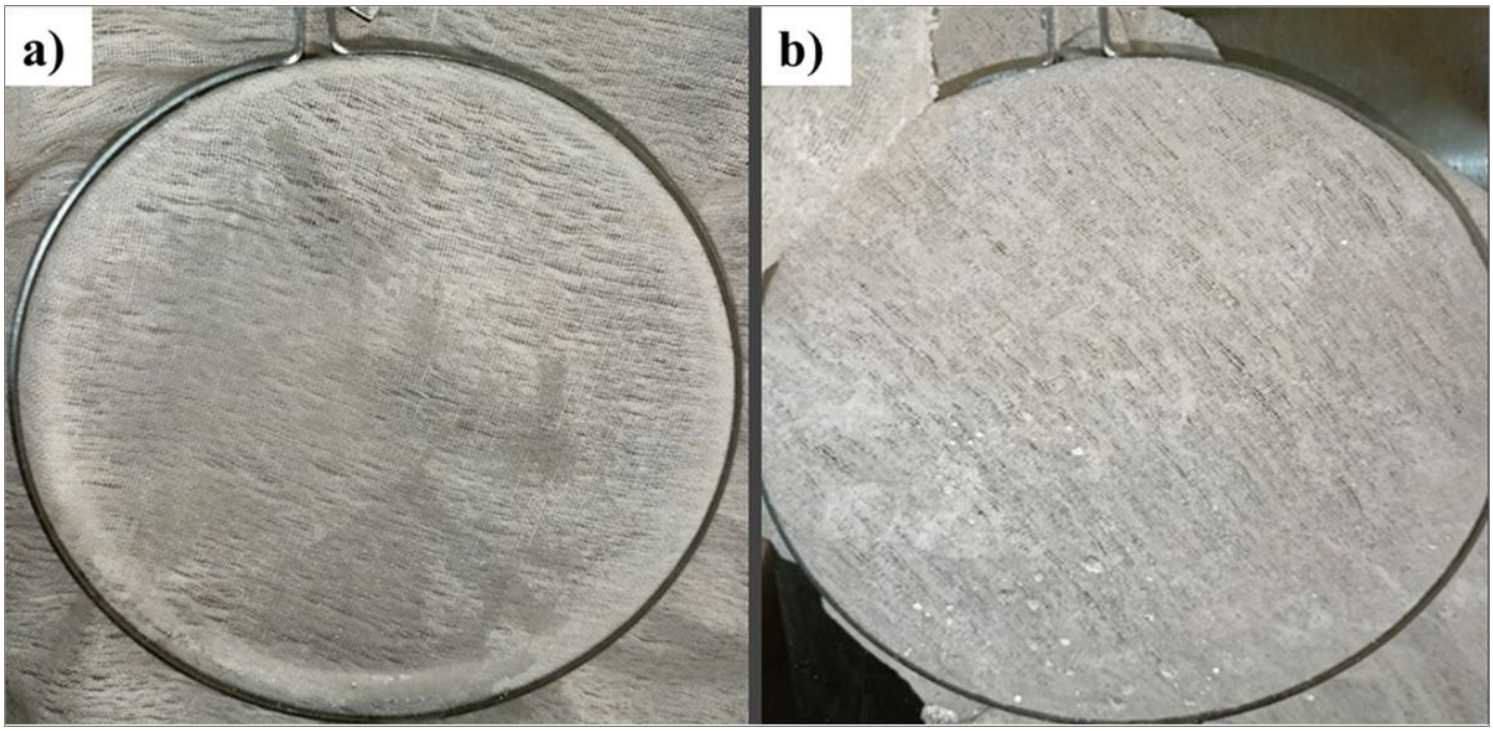

Figure 8

a) Uncoated Sample, b) ZnO & SiO2 Nanoparticle Coated Sample

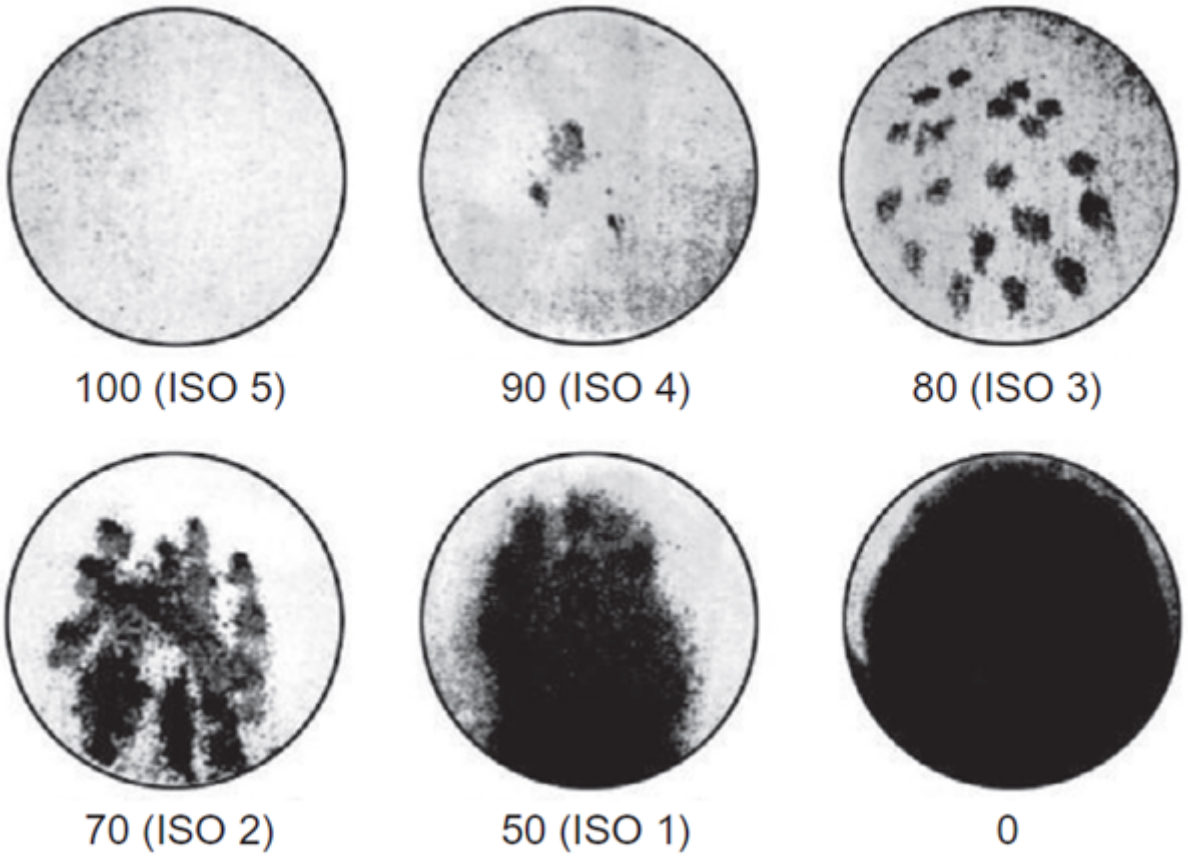

Figure 9

ISO Standard Scale of Spray Rating

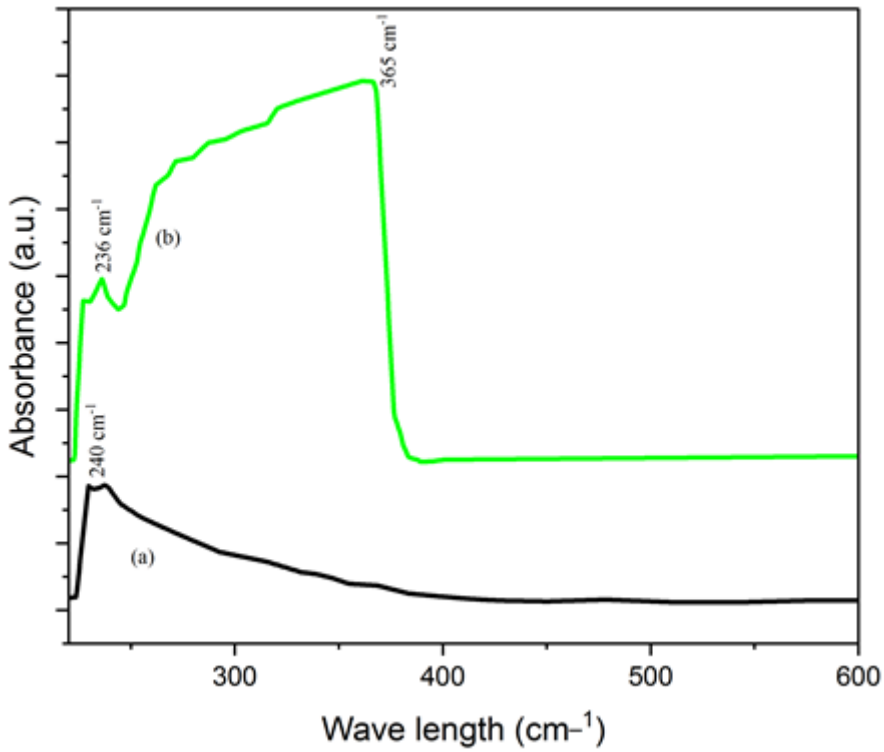

Figure 10

UV-Vis Spectroscopy of (a) Raw Fabric, (b) NPs Treated Fabric

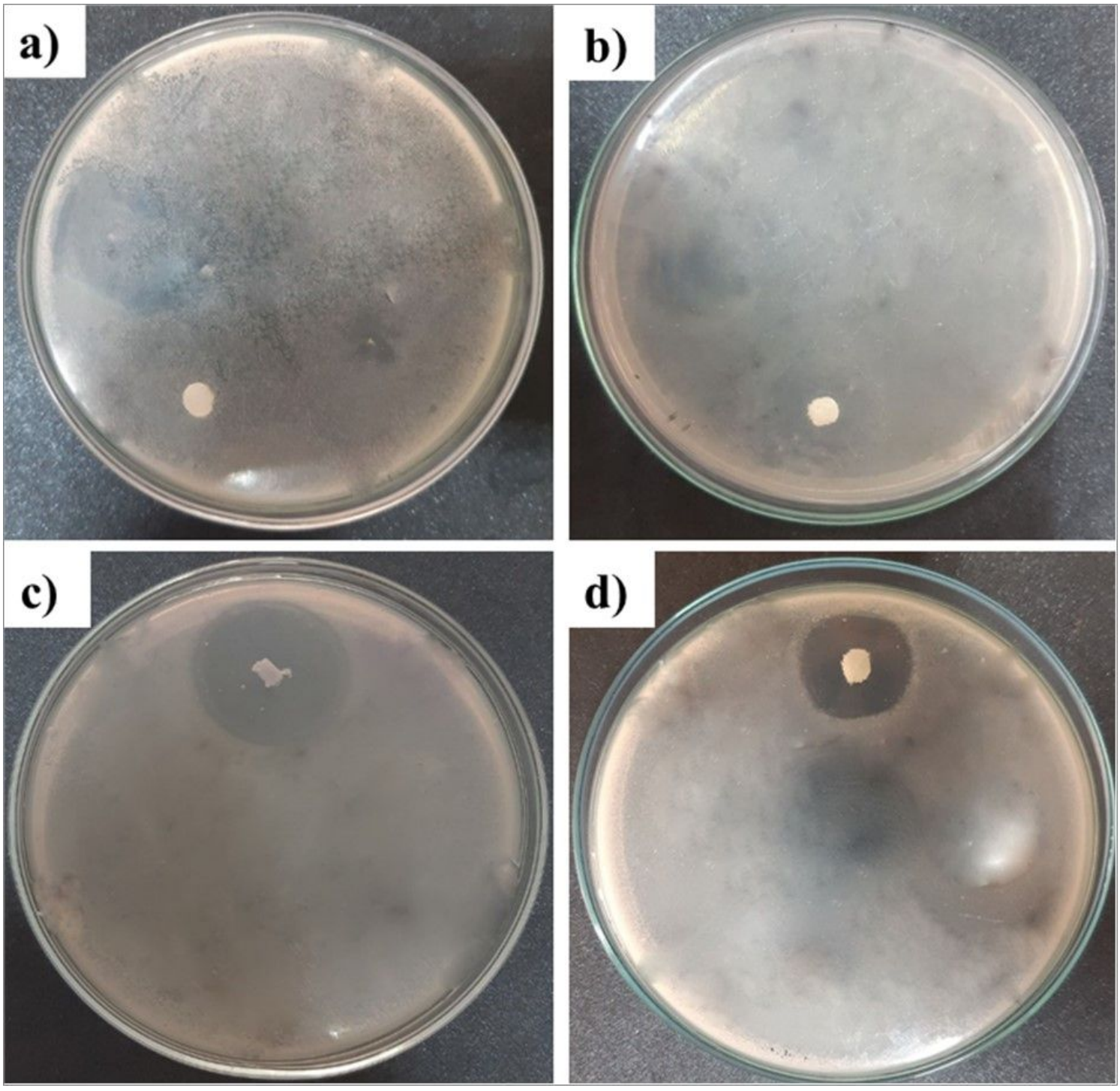

Figure 11

a) Untreated Sample against *S. Aureus,* b) Untreated Sample against *E. Coli,* c) Nanoparticle Treated Sample against *S. Aureus,* d) Nanoparticle Treated Sample against E. Coli