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Physical properties of green zno nano coated cotton fabrics

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ABSTRACT

Nanotechnology is a rapidly expanding interdisciplinary field that is frequently considered as a new industrial revolution. Among various nanoparticles, zinc oxide nanoparticles (ZnO NPs) are adaptable semiconducting inorganic materials with two main crystal structures. The use of plant extracts for synthesising metal oxide nanoparticles offers a promising alternative to traditional chemical methods. This study aimed to synthesis eco-friendly zinc oxide nanoparticles using Citrus reticulata (orange) peel powder with two zinc precursors. Cotton fabrics coated with ZnO-1 and ZnO-2 nanoparticles, along with cross-linking agents, showed improved overall performance compared to the control. The treated fabrics displayed fibre swelling, increased weight and thickness (especially with citric acid treatments ZAC1 and ZAC2), and better crease recovery without added stiffness. Tensile strength, elongation, water repellency and colour strength also improved due to nanoparticle deposition, enhancing physical and mechanical properties of the fabric.

Keywords: *Zinc oxide, Control, Treated, Citric acid, Maleic acid, Nanoparticles.*

1. Introduction

The unique and interesting properties of nanomaterials have captivated the interests of scientists and researchers as well as the textile industry, which is vital to the country's development. Nanotechnology partakes in the real commercial potential of the textile industry. This is mainly due to the fact that conventional methods used to impart different properties to fabrics often do not lead to permanent effects, and they will lose their functions after laundering or wearing. Nanotechnology can provide high durability for fabrics because nanoparticles have a large surface area-to-volume ratio and high surface energy, thus providing a better affinity for fabrics and increasing the durability of the function. In addition, a coating of nanoparticles on fabrics will not affect their breathability or hand feel. Textiles that have been developed with nanotechnology are already used in sports, cosmetics, space technology, clothing and material technologies to protect people better in extreme environments.

Metal nanoparticles synthesis is a fascinating subject in nanoscience. In recent decades, several research groups have been interested in various kinds of metal NPs, including iron oxide, silver nitrate, copper oxide and zinc oxide. Metal oxides such as titanium dioxide, zinc oxide, magnesium oxide and calcium oxide are of particular importance among inorganic materials. They are not only stable under extreme conditions but are also usually –recognised as safe materials for humans and animals. Among these, zinc oxide (ZnO) nanomaterials have remarkable potential for applications in many different areas, such as solar cells, sensors, piezoelectric devices, photodiode devices, sunscreens, anti-reflection coatings and photocatalysis [1].

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ZnO NPs show synergistic and enhanced therapeutic efficacy when combined with other therapeutic agents. Zinc oxide is a modifier in the textile industry because of its use for the production of safe garments and all kinds of technical applications [2].

Metal nanoparticles are manufactured utilising a variety of methods, including sol-gel, thermal decomposition, hydrothermal, microwave irradiation and others. However, because of the enormous number of secondary waste products formed as a result of the inclusion of chemical agents for the reduction process, these chemical and physical synthesis techniques are time-consuming, expensive and toxic [3]. Metal nanoparticles derived from plants or plant extracts are more stable than those derived from other sources. Modified organisms have a strong ability to optimise the synthesis of new proteins, enzymes and biomolecules required for the formation and stability of nanoparticles.

Thus, citrus reticulata peel was selected as a catalyst for the synthesis of green zinc oxide nanoparticles. Citrus fruits are commonly used in food processing industries, where tons of pulp, peel and rags are thrown as solid waste during the extraction process. The fruit waste generation not only results in financial losses but also adds to the expense of waste management and disposal. Thus, to minimise the fruit wastage and protect the environment, an experimental study was conducted by using citrus reticulata peel as a catalyst in the green synthesis of zinc oxide nanoparticles with different solvent extracts.

With the advent of science and technology, a new area has developed in the realm of textile finishing. Nanostructures have the potential to improve the physical, mechanical and functional qualities of conventional fabrics. Coating the surface of textiles and clothing with green nanoparticles is one approach to produce a highly active surface that has different functional properties, such as UV-blocking, antimicrobial, self-cleaning properties and other properties.

The washing resistance of functional fabrics developed with nanoparticles is also troublesome. This can be circumvented by utilising cross-linking agents, which not only improve the functioning of fabrics but also their wash endurance. Cross-linking agents also enhance cotton fabrics easy-care characteristics and connect nanoparticles to cotton fibres effectively. Thus, to improve the fabric properties of ZnO nano-cotton fabrics, they are treated with eco-friendly cross-linking agents with a pad-dry curing technique. The addition of cross-linking agents produces a covalent bond between the cellulose chains and nanoparticles, thereby strengthening the fabric's functional properties [4].

2. Materials and Methods

2.1. Selection and preparation process of *Citrus reticulata* peel powder

Nagpur orange is a variety of mandarin orange (*Citrus reticulata*) grown in Nagpur, Maharashtra, India. The fruit has a pockmarked exterior and sweet and juicy pulp. It gives the city of Nagpur its pseudonym, Orange City. The Nagpur oranges blossom during the Monsoon season and are ready to be harvested from the month of December. The orange crop here grows twice a year. The fruit is available from September to December, and it has a slightly sour taste. It is followed by the sweeter Mrig crop in January [5].

For the study, orange fruits of the Nagpur variety were collected from the local market of Dharwad. Collected fruits were cleaned thoroughly using distilled water, the peel and their separated the edible portion, oven-dried at 35°C for 48 h, ground to a fine powder with a laboratory grinder, sieved with ASTM standard mesh of 600 microns of 8 number size and stored in an air-tight container for further experimentation [6].

2.2 Optimisation of solvent extractions for synthesis of green ZnO NPs

Nanomaterial has a wide range of applications due to their size and morphology, and have been an important subject in the fields of basic and applied sciences. Among various nanoparticles, ZnO NPs are versatile semiconducting inorganic material with two main crystal structures. It has a wide band gap semiconductor with a band gap energy of 3.3 eV at room temperature. Zinc oxide can be synthesised using different methods like chemical, physical and biological. It has great potential for application in many fields, such as biosensors, cosmetics, drug carriers and antibacterial agents. Based on the results of the quantitative screening of phenolics, the ethanol extract was selected for the examination.

2.2.1 Extraction procedure

2.2.1.1 Ethanol Extraction

Two grams of weighed powder was added to 25 ml of solvent and incubated under agitation at 25 °C for 24 hrs. centrifuged at 5000 rpm for 10 minutes at 5 °C and refrigerated. The supernatant obtained was separated, and the residue was re-extracted with a fresh 25 ml of solvent. The process was repeated, and the supernatants were pooled and the extract obtained was measured and filtered using Whatman filter paper No.1. Extracts were stored in a refrigerator until further analysis.

2.2.1.2 Selection of precursors

Based on the references, Zinc acetate dihydrate and zinc nitrate hexahydrate were the two precursors selected for the study. *Citrus reticulata* peel powder was used as a catalyst for the green synthesis of zinc oxide nanoparticles, and a standardised protocol for green synthesis of zinc oxide nanoparticles. The synthesised green ZnO NPs were characterised for different parameters like particle size and zeta potential using a zetasizer, absorbance peak by UV-Visible spectrophotometer, morphology by Scanning Electron Microscope (SEM) with EDS, material structural characteristics by Atomic Force Microscope (AFM), functional groups with FTIR and structure with XRD.

2.3 Optimisation of treatment conditions for finishing of cotton fabrics

2.3.1 Selection of fabric

Cotton fabric is a type of natural cloth created from the cotton plant's fibres. It comes in a wide range of weaves and is the most versatile and popular fabric in the world. India is the largest producer of cotton in the world, accounting for about 22 per cent of the world's cotton production. Cotton is a breathable fabric that absorbs heat and perspiration from our bodies and allows them to pass through the fabric to the air outside, making it comfortable to the wearer. Thus, medium-weight, pure cotton plain-weaved fabric was used for the application of green synthesised zinc nanoparticles using orange peel extract.

2.3.2 Desizing and scouring

The following recipe was used for one bath scouring and desizing the cotton fabrics. At elevated temperatures, a hydroxide compound and caustic soda mixture can effectively combine desizing and scouring conditions. Desizing is the process of removing the sizing materials present in cotton fabric in order to make it suitable for further processing.

Recipe

Sodium hydroxide - 2 % owf
Sodium carbonate - 2 % owf
Turkey red oil - 2 % owf
Material to Liquor Ratio (MLR) - 1:20
Time - 85 min
Temperature -80 to 90 ºC

The fabric was weighed and the quantities of sodium hydroxide, sodium carbonate, turkey red oil and Material Liquor Ratio (MLR) were calculated. Water was mixed with sodium hydroxide and sodium carbonate, and the solution was heated to 80°. The fabric was then immersed in the solution and treated for 85 minutes at temperatures ranging from 80 to 90 °C. After that, the fabric was thoroughly washed under running water to remove any traces of sodium hydroxide and shade-dried.

2.4 Selection of cross-linking agents

Cross-linking agents are used as durable press finishing agents to produce wrinkle- resistant cotton fabrics and garments in the textile industry.

2.4.1 Citric acid

Citric acid is an organic compound with the chemical formula $C_6H_8O_7$. It occurs naturally in citrus fruits. In biochemistry, it is an intermediate in the citric acid cycle, which occurs in the metabolism of all aerobic organisms. More than two million tonnes of citric acid are manufactured every year. It is used widely as an acidifier, a flavouring agent, and a chelating agent.

Citric acid is an organic tricarboxylic acid present in most fruits, especially lemons and oranges. It is non-toxic and has wide use in the food industry as a safe natural additive since it is used as a preservative. This acid has the capacity to crosslink hydroxyl groups in cellulose and has been reported to occur at high temperatures [7 & 24].

2.4.2 Maleic acid

Maleic acid, or *cis*-butenedioic acid, is an organic compound that is a dicarboxylic acid, a molecule with two carboxyl groups. Its chemical formula is $C_4H_4O_4$. Maleic acid is the *cis*-isomer of butenedioic acid, whereas fumaric acid is the *trans*-isomer. It is mainly used as a precursor to fumaric acid, and compared to its parent maleic anhydride, maleic acid has few applications.

Maleic acid (MA) is a bi-functional carboxylic acid anhydride that forms only one ester linkage with cellulose and is not capable of cross-linking cellulose chains alone. Thus, sodium hypophosphate (NaH_2PO_2) was used as a catalyst to cross-link cellulose chains. The NaH2PO2 reacts with the >C═C< of the MA already esterified with cellulose, thus making it possible to form cross-linking on cotton cellulose by curing at higher temperatures. In this way, phosphorus bonds are cross-linked with cotton cellulose [7].

2.4.3 Pad-dry-cure method

Pad-dry-cure method is a widely used technique for textile finishing and can be used to add a variety of coatings. The fabric was submerged in the nano coating solution and then passed between the rollers in the padding mangle to squeeze out the excess liquid. The finished fabric is then dried and cured. The procedure followed is mentioned below:

Recipe

Material to Liquor Ratio (MLR) - 1:20
Synthesised Zinc oxide NPs - 4 % stock solution
Citric acid/Maleic acid - 2, 4, 6 % each owf
The fabric was immersed in the ZnO nano solution containing both crosslinking agents separately for ten minutes and passed through a pneumatic padding mangle (R. B. Electronic and Engineering, Mumbai) at a speed of 3 m/min with a pressure of 1kg/ cm^2 to remove excess solution, ensuring the wet pick up of 70 per cent. Later, the fabric was shade dried and cured for 10 minutes at 40°C, 50°C and 60°C.

2.4.4. Application of nanoparticles on the cotton fabric

The selected cotton fabric was desized and scoured. Using the pad dry curing method, samples of fabric measuring 25 x 25 cm were prepared and treated with a 4 percent stock of nano-solutions for both the precursors by following the following parameters.

Parameters and variables for coating of cotton fabrics with green ZnO Nps

Parameters	Variables
Nano concentrations (4 %)	5,10,15,20,25,30
Method of application	Pad-Dry-Cure
Eco-friendly Cross linking agents	Citric acid and Maleic acid
Cross linking agents (%)	2,4,6
Curing temperature	40 ºC, 50 ºC, 60ºC

2.5 Laboratory Testing of Fabrics

The fabric samples were tested in the fabric-testing laboratory at the Department of Textile and Apparel Designing, College of Community Science, University of Agricultural Sciences, Dharwad. The ZnO nano-coated fabrics were subjected to laboratory testing for physical and mechanical properties. The data obtained from the tests were statistically analyzed. For the purpose of convenience the fabrics were numbered the details of which are furnished below:

Samples	Treatments
ZnO-1 precursor	Green ZnO NPs with zinc acetate dihydrate precursor
ZnO-2 precursor	Green ZnO NPs with zinc nitrate hexahydrate precursor
Control	Untreated
ZAC1/ ZAC2	ZnO NPs with zinc acetate dihydrate treated with Citric acid
ZAM3/ZAM4	ZnO NPs with zinc acetate dihydrate treated with Maleic acid
ZNC5/ZNC6	ZnO NPs with zinc nitrate hexahydrate treated with Citric acid
ZNM7/ZNM8	ZnO NPs with zinc nitrate hexahydrate treated with Maleic acid

2.5.1. Atmospheric Conditions for Testing

For testing of textile materials in the laboratory, a standard atmosphere has to be maintained [1]. The testing samples were conditioned in an atmosphere with a Relative Humidity of 65±2% and a temperature of 20°±2°C prior to testing for 24 hrs.

2.5.2. Preparation of Test Specimens

The test specimens were prepared by cutting the samples as per the templates and procedures of the Bureau of Indian Standards 1968. Specimen cuts in each direction were scattered as far as possible so that no two warp-way specimens contain the same set of weft yarns. It was ensured that the specimens represented adequately the fabric under test and excluded the areas within 10cm of the selvedges and those with wrinkles or sharp folds.

2.5.2.1 Cloth count

Cloth count in woven textile material is the number of ends and picks per unit area, while the fabric is free from wrinkles and is affected by the yarn count and compactness of the weave. The number of warp and weft yarns in one square inch of the fabric is counted at five randomly selected places across the width and along the length of the test specimens. The region near the selvedge should be avoided because the spacing of the thread is often a little different from that in the body of the cloth [9]. Furthermore, mean values of ends and picks per inch were calculated.
Number of specimens tested: 5 for each warp and weft
Method: Direct counting of threads per unit area, 1 inch
Instrument: Magnifying counting device (Pick glass)

2.5.2.2 Fabric Weight

Fabric weight is expressed as mass per unit area in g/sq.mt. A sample of 5 × 5 cm was cut and weighed on an electronic weighing balance to determine the weight per sq. mt (g) [9]. Furthermore, warp and weft threads were separated and weighed to calculate their respective percentages. The percentage composition of warp and weft was calculated as follows.
Weight of 5 × 5 sample : a (g)
Weight of warp yarns : b (g)
Weight of weft yarns : c (g)
% of warp : b/a × 100
% of weft : c/a × 100

2.5.2.3. Fabric Thickness

The thickness and surface thickness of the fabric are useful indicators of any change or variation in the fabric's handle and appearance [9]. The fabric was kept between the two parallel lines, and a known, arbitrary pressure was applied between the plates and maintained throughout the test. Then the distance between plates was measured by a digital gauge.

The test specimens were conditioned for 24 hours prior to testing. The thickness was measured at least 10 areas, and the mean value was calculated. Care was taken to raise the pressure foot very slowly, maintaining the same pressure.

2.5.2.4 Stiffness

Fabric stiffness is the resistance of the fabric to bending. "Bending length" is the length of the fabric that bends under its own weight to a definite extent. It equals half the length of the rectangular stripe of fabric that bends under its own weight to an angle of 41.5°. The test samples were tested as directed in the BS test method: 3356-1961. A rectangular strip of fabric, 6 inches × 1 inch, was mounted on a horizontal platform in such a way that it hangs like a cantilever and bends downwards. The test specimen was cut with the help of a template, and then both the template and test specimen were placed on the platform with the fabric underneath. Both were pushed forward slowly. The fabric strip began to droop over the platform's edge, and the movement of the templates and fabric continued until the tip of the specimen, as seen in the mirror, cut both index lines. The bending length was read off from the scale mark opposite the zero line engraved on the side of the platform. Five readings were recorded by using Shirley's stiffness tester [9].

2.5.2.5 Crease recovery

Crease recovery is nothing but the allowance of the fabric to recover from the crease. The test samples were tested as directed in IS method: 4681-1968 by using Shirley's crease recovery tester. Samples were cut both warp and weft ways from the fabric with a template, 2 inches long by 1 inch wide. It was creased by folding it in half and placing it under a weight of 2 kg for 5 minutes. The weight was removed, and the specimen was transferred to the fabric clamp on the instrument using forceps and allowed to recover from the crease for 5 minutes. As it recovered, the dial of the instrument was rotated to keep the free edges of the specimen in line with the knife edge. At the end of the time period, as it was allowed for recovery, usually 1 minute, the recovery angle in degrees was read on the engraved scale. Readings were recorded for both warp and weft separately [9].

2.5.2.6 Tensile strength

Tensile strength (Newton force/kg) is the ability of the material to resist or rupture induced by tensile force. It is expressed as force per unit cross-sectional area of the specimen at the time of maximum load. Elongation (%) is the increase in length of the specimen from its initial length, expressed in units of length. The distance that material will extend under a given force is proportional to its original length. Hence, elongation is coated as strain or percentage was assessed for the fabrics. The elongation that a specimen undergoes is proportional to its initial length.

The specimens were tested as directed in IS 1969-Part-1, 2009. The method employed to determine the breaking load and elongation of the material is by using the ravelled strip in the "Unistretch" tensile tester. The specimen was a 5 cm wide piece of fabric prepared initially by cutting the material to a width of about 7 cm, and then the threads were ravelled from both sides until the width attained 5 cm. The test length is 200 nm in between the jaws with a load range of 250 kgf, and an extra length was taken to grip within the jaws.

2.5.2.7 Water repellency

The purpose of the test was to analyse the ability of a fabric to absorb water. It is indirectly related to the comfort properties of the fabric [9]. As per the test method IS 390-1975, the specimen was conditioned prior to testing. There was no specific size of the template used. The specimen was mounted in the specimen holder. The mounted specimen was placed under the spray nozzle on the support provided in the stand for this purpose in such a way that the warp threads were approximately parallel to the direction of the flow of water. Water was poured along the sides of the funnel quickly; the quantity of water was 250 ml at 27±2°C, and it was allowed to spray on the specimen. The specimen holder was removed from the stand when the spraying had ceased. The lowest point of the holder was tapped 3 times in succession against a horizontal surface. Immediately after tapping, it was compared under reflected light with the standard ratings. The number obtained by evaluating all the test specimens was rounded off to the nearest standard rating number and was reported as the rating number of the sample. The test procedure was followed as per the IS 390- 1975.

100 **-** No sticking or wetting of the upper surface
90 **-** Slight random sticking or wetting of the surface
80 **-** Wetting of the upper surface at spray points
70 **-** Partial wetting of the whole of the upper surface
50 Complete wetting of the whole of the upper surface
0-Complete wetting of the whole of the upper and lower surfaces

2.5.2.8 Colour Strength

The colour strength (K/S) values of zinc oxide nano-treated cotton fabrics were measured by using a spectrophotometer (Konica Minolta Spectrophotometer CM-3600d) with interface JAYPAK 4808 (Ver: 01.08.001). The strength of any colourant (fabric) is related to its absorption property. Kubelka–Munk theory gives us the following relation between reflectance and absorbance:

$$\mathbf{K/S = [\{(1-R)^2 / 2R\}]}$$

Where R is the reflectance, K is the absorbance, and S is the scattering. By using the above equation colour strength of different samples was measured.

3. Results and Discussion

3.1 Physical and mechanical properties of green ZnO nano-coated cotton fabrics

3.1.1. Cloth count (Ne)

Cloth count is the number of warp yarns (ends) per unit distance and filling yarns (picks) per unit distance. They are determined using magnifying and counting devices or by unravelling yarns from fabrics. The higher the cloth count, the finer the yarns, and vice versa. In all the samples, the warp-way cloth count was found to be higher than the weft-way cloth count. The data pertaining to the cloth count is given in Table 1. From the data, it is clear that the warp way has a higher cloth count than the weft way. It was also observed that after treatment, the cloth count in both fabrics was reduced. In ZnO-1 and ZnO-2 treated fabrics, the warp cloth count decreases from 30 to 31 (Ne). Irrespective of the application, cotton fabric treated with citric acid showed 31 (Ne), whereas cotton fabric treated with maleic acid showed 30 (Ne).

In the weft direction, cotton fabric treated with zinc acetate and zinc nitrate nanoparticles showed less cloth count (25 to 26 Ne) when compared to untreated fabric (28 Ne).

It is also observed that fabric treated with citric acid has a lower cloth count (25 to 26 Ne) than fabric treated with maleic acid (27 to 28 Ne). The results of the statistical analysis revealed that there is a significant difference between nanoparticles as well as with crosslinking agents at a 1 per cent level of significance.

Regardless of the source for ZnO NPs production or the technique of application, the cloth count of the control sample was found to be higher than the treated fabric samples in both the warp and weft directions (Table 1). The cloth of the nano-coated cotton fabrics synthesised with ZnO-1 and ZnO-2 precursors showed less yarn count than the control fabric. This may be due to the absorption and coating of orange peel powder extract as well as cross-linking agents by the yarns, which resulted in the swelling of fibres, thus reducing the yarn count in both warp and weft directions. According to the study [1], a decrease in the fabric count of the finished fabric treated with the combination of herbal extracts of *Ricinus communis, Senna auriculata and Euphorbia hirta.* The statistical analysis reveals that there exists a significant difference between nanoparticles as well as with the crosslinking agents.

Table 1. Effect of green ZnO nano-coating on Cloth count

Sample	Crosslinking agents	Cloth count (Ne)	
		Cotton	
		Warp	Weft
Control (Untreated)		32	28
ZnO -1 precursor			
ZAC1	Citric acid	31 (3.12)	26 (7.14)
ZAC2		31 (3.12)	26 (7.14)
ZAM3	Maleic acid	30 (6.25)	27 (3.57)
ZAM4		30 (6.25)	27 (3.57)
ZnO-2 precursor			
ZNC5	Citric acid	31 (3.12)	25 (10.71)
ZNC6		31 (3.12)	26 (7.14)
ZNM7	Maleic acid	30 (6.25)	27 (3.57)
ZNM8		30 (6.25)	27 (3.57)

(Values in parenthesis indicate percentages)

ANOVA Table

Particulars		Warp	Weft
C.D. (0.01)	Nano particles (NPs)	2.667**	0.66
	Cross-linking agents (CLA)	6.00	8.66**
	NPs x CLA	0.66	0.66
C.V. %		4.587	7.556

***Highly significant @ 1 per cent level*

3.1.2 Fabric weight

Fabric weight is often determined by the type of material used, the weave, and the fibre thickness. It is expressed in grams per meter (gsm) or ounces per yard (oz/yd) (oz). It is noticed in Table 20, that an increase in the weight of the fabric was observed after the treatment with ZnO nanoparticles in both the precursors, from 159 to 156 g/m^2 when compared to the control (152 g/m^2). Furthermore, in both the nano-treated fabrics, an increase in percentage was observed in the warp way (51 to 54%) than in the weft way (48 to 49%). Fabrics treated with Citric acid has a considerably higher fabric weight in both ZnO-1 and ZnO-2 treated fabrics when compared to fabrics treated with maleic acid (Table 2). Similarly, fabrics treated with ZnO-2 precursor have shown higher values in warp (52 to 53%) than in weft (46-48 %).

The statistical analysis revealed that there is a highly significant difference at a 1% level between crosslinking agents, but no significant difference was found with nanoparticles. In general, fabric weight influences properties such as fabric thickness, stiffness and thermal conductivity.

Table 2. Effect of green ZnO nano-coating on Fabric weight (GSM)

Samples	Crosslinking agents	Cloth weight (g/m²)		
		Cotton		
		Cloth weight	Warp	Weft
ZA nanoparticles				
Control (Untreated)		152	51	44
ZnO-1 precursor				
ZAC1	Citric acid	158	54 (5.55)	49 (11.3)
ZAC2		159	53 (3.77)	49 (11.3)
ZAM3	Maleic acid	156	52 (1.96)	48 (9.09)
ZAM4		156	53 (3.77)	48 (9.09)
ZnO-2 precursor				
ZNC5	Citric acid	157	52 (1.96)	48 (9.09)
ZNC6		156	53 (3.77)	48 (9.09)
ZNM7	Maleic acid	155	52 (1.96)	46 (4.54)
ZNM8		155	52 (1.96)	47 (6.81)

(Values in parenthesis indicate percentages)

ANOVA Table

Particulars		Warp	Weft
C.D. (0.01)	Nano particles (NPs)	2.667	2.66
	Cross-linking agents (CLA)	10.66*	54.00**
	NPs x CLA	2.66	0.667
C.V. %		2.50	3.67

***Highly significant @ 1 per cent level*

Table 2 shows that the cloth treated with zinc oxide nanoparticles has a greater fabric weight than the control. Furthermore, it is clear that there was an increase in fabric weight in both the warp and weft directions compared to the control. The absorbance of ZnO nanoparticles and orange peel extract, as well as other finishing agents, may account for the weight gain. The results are consistent with the study [10]. Where Cotton fabric treated with lemon peel extract using the exhaust and pad dry cure procedures showed a substantial increase in fabric thickness of 2.7 and 5.5 percent, respectively, when compared to control cotton fabric samples. Furthermore, according to the study [16], it was observed that the application of various herbal extracts on cotton resulted in an increase in weight gain percentage. The interaction between the fabric, nanoparticles, and cross-linking agents had a significant effect on the weight in both the warp and the weft directions. This shows that cross-linking agents have an effect on the weight of the fabric.

3.1.3 Thickness

A fabric's thickness is defined as the distance between its two surfaces under a particular application of force, which can vary if the fabric has a high loft (or is compressible). In the fabrics treated with citric acid, ZAC1 and ZAC2 samples had registered the highest thickness of 0.43 mm, followed by samples ZNC5 and ZNC6 with 0.42mm. The thickness of the fabrics was increased considerably in all the treated samples compared to the control fabric (0.38mm). The fabrics treated with maleic acid showed the highest thickness values in samples ZAM3, ZAM4 (0.42 mm) and lowest in ZAM8 (0.41 mm), followed by ZAM7 (0.41 mm)

thickness. The data pertaining to the thickness of the fabric is furnished in Table 3.
The statistical analysis reveals that there is a significant difference between the crosslinking agents. The difference between nanoparticles was not found to be significant. The thickness of the fabrics was influenced by the yarn count of the threads used. This property seemed to have an influence on fabric weight, stiffness and absorbency.

Table 3. Effect of green ZnO nano-coating on Fabric thickness

Sample	Cross-linking agents	Thickness (mm)
		Cotton
Control (Untreated)		0.38
ZnO-1 precursor		
ZAC1	Citric acid	0.43 (13.15)
ZAC2		0.43 (13.15)
ZAM3	Maleic acid	0.42 (10.52)
ZAM4		0.42 (10.52)
ZnO-2 precursor		
ZNC5	Citric acid	0.42 (10.52)
ZNC6		0.42 (10.52)
ZNM7	Maleic acid	0.40 (5.26)
ZNM8		0.41 (7.89)

(Values in parenthesis indicate percentages)

ANOVA Table

Particulars		Thickness
C.D. (0.01)	Nano particles (NPs)	2.667*
	Cross-linking agents (CLA)	54.00***
	NPs x CLA	0.667
C.V. %		4.244

***Highly significant @ 1 per cent level*

Fabric thickness is the distance between two fabric surfaces under a certain applied pressure, which varies if the fabric has high-loft (or compressible) properties. Table 3 shows that the thickness of the nano-coated fabrics has increased when compared to the control in ZnO-1 and ZnO-2 types of fabrics. Higher deposition of ZnO NPs, as well as the extract of orange peel powder, may have caused the cloth to thicken. It is also confirmed by statistical analysis that there exists a significant difference between cross-linking agents, but not with the treatments.

3.1.4 Crease recovery

It is the property of a textile material by which it can return to its former shape after being creased. The measure of crease resistance is specified quantitatively in terms of crease recovery angle. From Table 4, it can be observed that the crease recovery angle of all the samples showed a significant increase in both warp and weft directions when compared to control fabrics. It is clear that the highest values were obtained in sample ZAC1 (113°), followed by ZNC6 (113°) and ZAC2 (112°) in warp way for the fabrics treated with ZnO nanoparticles along with citric acid, whereas for ZnO nano-coated fabrics with maleic acid, ZAM4 (113°), followed by ZAM3 (112°), ZNM7 (112°) and ZNM8 (112°) values were registered.

Table 4. Effect of green ZnO nano-coating on Crease recovery angle (^{o})

Sample	Crosslinking agents	Crease recovery angle (^{o})	
		Cotton	
		Warp	Weft
Control (Untreated)		110	92
ZnO-1 precursor			
ZAC1	Citric acid	113 (3)	98 (6.5)
ZAC2		112 (1.81)	96 (4.34)
ZAM3	Maleic acid	112 (1.81)	96 (4.34)
ZAM4		113 (2.72)	100 (8.69)
ZnO-2 precursor			
ZNC5	Citric acid	113 (3)	97 (5.43)
ZNC6		113 (3)	98 (6.5)
ZNM7	Maleic acid	112 (1.81)	96 (4.34)
ZNM8		112 (1.81)	97 (5.43)

(Values in parenthesis indicate percentages)

ANOVA Table

Particulars		Warp	Weft
C.D. (0.01)	Nano particles (NPs)	0.667	2.66
	Cross-linking agents (CLA)	20.66	82.66**
	NPs x CLA	0.667	18.66**
C.V. %		4.709	6.826

***Highly significant @ 1 per cent level*

In the weft direction, a higher crease angle was registered in samples ZAC1 (98°), ZNC6 (98°), followed by ZNC5 (97°) and ZAC2 (96°). Similarly, higher crease recovery angles were registered with maleic acid compared to control fabrics. This might be attributed to the higher stiffness of the cotton fabrics treated with nanoparticles. Crease recovery angle was increased both in warp and weft directions due to the nano treatment.
Statistical values from the table indicate that there is a significant difference between the nanoparticles, crosslinking agents and nanoparticles / crosslinking agents. There was an impact of treatment on the crease recovery.
In general, the treated samples recovered their crease recovery faster than the untreated samples. Cloth treated with citric acid had a higher crease recovery angle than fabric treated with maleic acid, regardless of the presence of ZnO nanoparticles (Table 4). The presence of cross-linking chemicals may have obscured or eliminated hydrogen bonds, resulting in an increase in crease recovery. The nano-treated fabrics showed improved crease recovery, tensile strength, elongation, air permeability, and tear strength compared to untreated fabric. The statistical results revealed that there is a significant difference between nanoparticles and crosslinking.
According to the study [12], the effect of eco-friendly cross-linkers on cotton fabric using metal oxide nanoparticles via the pad-dry-cure method with and without UV irradiation was evaluated as a fixation procedure. ZnO, in combination with CA and MA, exhibited the greatest improvement in crease recovery, followed by MgO and CaO. The addition of these nanoparticles also enhanced the fabric's tear strength, handle and antibacterial properties.

3.1.5 Stiffness

Stiffness is a special property of a fabric. It is the tendency of the fabric to keep standing without any support.

It is a key factor in the study of handling and drape. From Table 5, it is observed that all the treated fabrics have increased their stiffness after the treatments compared to the control fabric. Among all the samples in ZnO-1 precursor -coated fabrics, ZAC2 has the highest stiffness in both warp (2.6 cm) and weft (1.9 cm), followed by ZAC1, ZAM4 and ZAM3.

In samples of ZnO-2 precursor treated, higher readings were observed in ZNC6 with 2.5 cm in the warp direction and 1.9 cm in the weft direction, followed by ZNC5, ZNM7 and ZNM8. It is also found that fabrics treated with citric acid have achieved higher stiffness values compared to maleic acid in both the ZnO-1 and ZnO-2 treated fabrics. Therefore, the increase in the stiffness for the first and second nano-treated samples with two precursors might marginally make the fabric stiffer, thereby reducing fabric mobility. The statistical analysis shows that there is a significant difference between crosslinking agents, but not between nanoparticles and cross-linking agents.

Table 5. Effect of green ZnO nano-coating on Stiffness (cm)

Sample	Crosslinking agents	Stiffness (cm)	
		Cotton	
		Warp	Weft
Control (Untreated)		2.3	1.7
ZnO-1 precursor			
ZAC1	Citric acid	2.5 (8.69)	1.8 (5.88)
ZAC2		2.6 (13.04)	1.9 (11.76)
ZAM3	Maleic acid	2.4 (4.3)	1.8 (5.88)
ZAM4		2.5 (8.69)	1.8 (5.88)
ZnO-2 precursor			
ZNC5	Citric acid	2.4 (4.3)	1.8 (5.88)
ZNC6		2.5 (8.69)	1.9 (11.76)
ZNM7	Maleic acid	2.4 (4.3)	1.8 (5.88)
ZNM8		2.4 (4.3)	1.8 (5.88)

(Values in parenthesis indicate percentages)

ANOVA Table

Particulars		Warp	Weft
C.D. (0.01)	Nano particles (NPs)	0.007	0.007
	Cross-linking agents (CLA)	0.487**	0.287**
	NPs x CLA	0.007	0.007
C.V. %		8.507	4.254

***Highly significant @ 1 per cent level*

From Table 5, it is clear that the stiffness of the treated fabrics did not differ significantly compared to the control fabrics. The ZAC2 sample was recorded with the highest stiffness values, followed by ZAC1, ZAM4 and ZAM3.

This demonstrates that nanoparticles are uniformly dispersed across the fabric without adding bulk and thereby increasing stiffness. The existence of a cross-linking agent and ZnO NPs is responsible for this trend. Without a cross-linking agent, the hydrogen bonds between cellulose chains may break under stress, allowing the chains to slip against each other. The increase in bending length of the treated samples is also attributed to hydrogen bonds in cellulose, which are converted to covalent bonds after cross-linking [13]. The statistical analysis also reveals that there exists a significant difference between crosslinking agents, but not significant with nanoparticles and nanoparticles/ cross-linking agents, indicating that the application of ZnO nanoparticles has changed the property of the cotton fabric. In addition, numerous researchers have shown that the presence of cross-linking agents and herb extract might produce an increase in cotton bending length [14].

3.1.6 Tensile strength

Among all the samples, green nanoparticles of ZnO-1 precursor-treated fabrics have shown better tensile strength. ZAC2 had registered higher tensile strength in both warp and weft directions with 66.3 kgf and 2.52 percentage elongation, followed by ZAC1 65.5 kgf (2.35%) and ZAM3 65.3 kgf (2.57% elongation). Similarly, in samples treated with ZnO-2 precursor, the highest strength is registered in ZNC6 with 64.8 kgf, 2.32 per cent elongation, followed by the ZNC5 sample with 64.1 kgf, 2.3 per cent elongation. The lower values were obtained for fabrics treated with maleic acid, ZNO-2 precursor in ZNM8 63.3 kgf, 2.2 per cent, followed by ZNM7 62.9 kgf, 1.97 per cent elongation Fig.1.

The load and elongation were increased in both warp and weft directions after the treatment for all ZnO-1 and ZnO-2 samples when compared to the control. However, the results of ANOVA showed there was a significant difference between the crosslinking agents and nanoparticles.

Table 6. Effect of ZnO nano-coating on Tensile Strength

Sample	Crosslinking agents	Tensile strength			
		Warp		Weft	
		Strength (Kgf)	Elongation (%)	Strength (Kgf)	Elongation (%)
Control (Untreated)		62.4	1.9	51.97	1.3
ZnO-1 precursor					
ZAC1	Citric acid	65.5	2.35	56.1	1.67
ZAC2		66.3	2.52	56.67	1.72
ZAM3	Maleic acid	65.3	2.37	55.21	1.6
ZAM4		62.8	2.57	53.9	1.58
ZnO-2 precursor					
ZNC5	Citric acid	64.1	2.3	52.6	1.47
ZNC6		64.8	2.32	53.92	1.56
ZNM7	Maleic acid	62.9	1.97	52.89	1.5
ZNM8		63.3	2.2	53.65	1.5

ANOVA Table

Particulars		Warp		Weft	
		Strength (kgf)	Elongation (%)	Strength (kgf)	Elongation (%)
C.D. (0.01)	Nano particles (NPs)	0.327	0.004	8.85	0.03
	Cross-linking agents (CLA)	30.32	0.89	34.34	0.31
	NPs x CLA	0.12	0.001	4.01	0.01
C.V. %		2.63	17.43	2.60	9.14

***Highly significant @ 1 per cent level*

In general, all the treated samples exhibited greater tensile strength than the untreated fabric. Among the treated samples, irrespective of cross-linking agents, fabric finished with ZnO NPs synthesised by ZnO-1 precursor showed higher tensile strength compared to the untreated fabric. The load and elongation were increased in both warp and weft directions after the treatment for all the samples when compared to the control. The increase in the tensile strength is possibly due to the slight rupture of cellulosic chains. Higher elongation for breakage of cotton fabrics loaded with ZnO nanoparticles is related to the presence of the metal nanoparticles bonded to hydroxyl groups of cellulose chains in the cotton fibres. This is also because of the fact that pulling the fabric with additional metal nanoparticles requires more force [15].

The results support the study [16], which developed zinc oxide nano-coated fabrics and studied their mechanical and antibacterial properties using pomegranate peel extract. The results showed that an increase in tensile strength could be owing to the strong contact between PVA and the two components, which improved the mechanical strength of the fabrics.

3.1.7 Water repellency

Water repellent is a state characterised by the non-spreading of water globules on textile material. Table 7 shows that following treatment with ZnO nanoparticles in both precursors, the rating of the control sample from complete wetting of the whole of the upper and lower surfaces was increased to complete wetting of the upper surface, demonstrating that there was an impact on water repellency after nano-coating. The results support the findings of study [17], which shows improvement in water repellency may be due to the deposition of nanoparticles, which hinder the flow of water through the fabric.

Table 7. Effect of green ZnO nano-coating on Water repellency

Sample	Crosslinking agents	Water repellency
Control (Untreated)		0
ZnO-1 precursor		
ZAC1	Citric ac id	50
ZAC2		50
ZAM3	Maleic acid	50
ZAM4		50
ZnO-2 precursor		
ZNC5	Citric ac id	50
ZNC6		50
ZNM7	Maleic acid	50
ZNM8		50

The ratings of the treated samples were increased over the control fabric. Table 25, clearly indicates that there was an impact of nano-coating on the water repellency of cotton fabrics, which has enhanced the rating of repellency compared to the control. This may be due to the deposition of nano-particles on the fabric, which hinders the flow of water through the fabric and thus increases the repellency of water. The results are consistent with the study [3], which assessed the ultra-violet protection and wettability properties of nano zinc oxide (ZnO) with acrylic binder on the cotton fabric using the pad-dry-cure method. Test results showed a significant improvement in the UV-absorbing activity of the ZnO-coated cotton fabric. The coatings of nanomaterials also enhance hydrophobic properties by changing the wetting capability of the cotton fabric surface.

3.1.8 Colour strength

The colour strength property is characterised by the absorption in the visible region of the spectrum and expressed as a colour strength value.

From Table 8, it is clear that the colour strength (K/S) values of ZnO-treated fabrics were compared with untreated samples, showing a change in the reflective values and K/S values. The colour strength values were increased from 2.32 to 3.86 K/S when compared to the control at 2.32 K/S. It is also clear that fabrics treated with the ZnO-1 precursor had higher K/S values than those treated with the ZnO-2 precursor.

The L* values of the ZnO-treated samples decreased from 83.43 to 81.88 compared to the control at 85.99. This indicates that the fabrics are becoming lighter after the treatment. The a* values of the treated fabrics have shown a significant difference from redness to greenness with -1.47 to -0.74 when compared to the control (3.09). The b* values of the treated fabrics have shown improvement in values after the treatment, from bluer (-11.81) to yellower (6.9 to 8.58).

Table 8. Effect of green ZnO nano-coating on colour strength of the fabric

Sample	Crosslinking agents	Colour strength			
		K/S	L*	a*	b*
Control (Untreated)		2.32	85.99	3.09	-11.81
ZnO-1 precursor	Citric acid	3.21	81.93	-0.74	8.58
		2.74	83.18	-1.47	7.53
	Maleic acid	2.55	83.43	-1.09	6.9
		3.86	81.95	-0.87	8.45
Both	CA/MA	3.22	82.79	-1.11	7.61
ZnO-2 precursor	Citric acid	2.56	82.81	-1.33	6.63
		2.55	83.04	-1.34	6.71
	Maleic acid	3.16	82.35	-1.35	8.48
		3.08	81.88	-1.12	8.19
Both	CA/MA	2.845	82.36	-1.232	7.81
Both	Citric acid	2.71	82.72	-1.22	7.54
	Maleic acid	3.49	82.43	-1.1	7.88

K/S (Colour strength), L [(+) Lightness / (-) Darkness], a* [(+) Redness / (-) Greenness], b* [(+) Yellowness / (-) Blueness*

ANOVA Table

Particulars		K/S	L*	a*	b*
C.D. (0.01)	Nano particles (NPs)	0.146	0.050	0.041	0.385
	Cross -linking agents (CLA)	1.926**	30.35**	45.90**	1033**
	NPs x CLA	1.623*	3.739**	0.469**	3.476
C.V. %		13.567	0.534	49.564	90.872

***Highly significant @ 1 per cent level*

In general, the colour analysis of the cotton fabrics treated with ZnO nanoparticles showed a difference in colour values when zinc oxide nanoparticle concentrations are varied. The statistical analysis of the colour strength shows a significant difference between crosslinking agents and nanoparticles/ crosslinking agents.

The colour strength (K/S) of the nano-coated cotton fabric has increased after the treatment compared to the control. It is also observed from Table 8, that higher K/S values were obtained in ZnO-1-treated fabrics compared to ZnO-2-treated fabrics. The higher k/S values show the existence of nano metal particles on the cotton fabric. The L*, a* and b* values of the treated fabric were decreased compared to the control, showing that the fabrics developed were showing lightness, yellowness, and greenness. The findings are congruent with study [17], who used pad dry cure method to evaluate the mechanical properties of jute fabric using a manufactured colloidal nano-Zinc solution. Higher K/S values were reported, indicating the presence of nano metal particles for colour affinity towards the substance (Table 8).

The results are consistent with the study [18]. The use of pomegranate peel extract in the functionalization process led to an increase in the K/S values, indicating enhanced colour intensity compared to the untreated cotton sample.

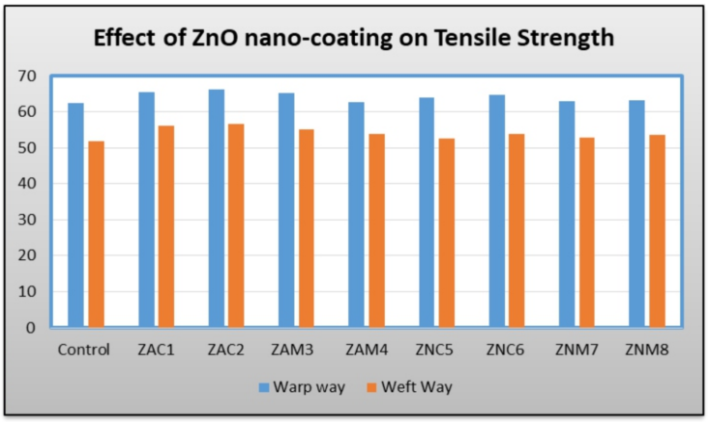

Fig. 1. Effect of green ZnO nano-coating on Tensile Strength

4. Conclusion

Nano-coating of cotton fabrics with ZnO-1 and ZnO-2 using orange peel extract and cross-linking agents improved overall fabric performance compared to the control. Treated fabrics showed reduced yarn count due to fibres swelling, increased weight and thickness (especially with citric acid treatments ZAC1 and ZAC2) and enhanced crease recovery without altering stiffness. Tensile strength and elongation improved in both warp and weft, while water repellency ratings increased due to nanoparticle deposition. Colour strength (K/S) values were higher in ZnO-1 than ZnO-2, with reduced L*, a*, b* values, imparting lighter yellow-green shades. Overall, ZnO nano-coating, on cotton fabrics has enhanced physical, mechanical and aesthetic properties of cotton fabrics without compromising softness.

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