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Low-stress mechanical properties of dual bio-pre-mordanted catechu-dyed cotton fabrics subsequently bio-finished with Eucalyptus leaf extract, nano-chitosan and nano-ZnO

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The earlier part of the study, reported elsewhere, evaluated the results of bio-dyeing of cotton fabrics with catechu natural bio-dye using potash alum (K-alum) + Gallnut (GN) dual pre-bio-mordanting to achieve maximum colour strength with overall good colour fastnesses to washing, rubbing and UV-light exposure. The present part of the study evaluates the efficacy of three different bio-finishing (after dyeing) methods using eucalyptus leaf extract, nano-chitosan, and nano-ZnO to improve their antimicrobial and UV resistance properties to the maximum level possible. Skin sensory tactical comfort properties in terms of responses to tensile loading/ stretching, shearing, bending, compression, surface friction (roughness) under low-stress conditions as primary hand values and resultant total hand values of the said dual bio-mordanted and catechu dyed and subsequently bio-finished with above said three different bio-finishes, were evaluated with Kawabata Evaluation System (KES). The said KES analysis revealed an overall increase in thickness (approx. 4- 67 %), weight per sq. centimeter of the fabric (approx. 16- 27 %), and coefficient of friction (approx. 32- 62 %) after application of the said bio-finishes. Still, as per resultant total hand value analysis, all these bio-finished fabrics are found not suitable for men's shirting in summer, but are found suitable for women's dress materials for the winter/autumn season. Simultaneously, Eucalyptus leaf extract bio-finished catechu dyed cotton fabrics had shown excellent UV protection factor of 45-50 with 98-99 % bacterial reduction in specific conditions of treatment, while nano-ZnO bio-finished catechu dyed cotton fabric had also exhibited maximum reduction in bacterial growth (98- 99 %) and nano chitosan did not show that level of very good results for antimicrobial and UV-protective criteria. Thus, both Eucalyptus leaf-extracted and nano-ZnO bio-finished fabrics have shown their potential for use as medical textile applications due to improved functional properties.

Keywords: Bio-finish, Catechu, Eucalyptus leaves, Kawabata Evaluation System, Nano-Chitosan, Nano-ZnO

1 Introduction

The hand value of a fabric, garment, or human skin's sensory feel is an important attribute, apart from its look, colour, style, and appearance. However, the sensory feel of any textiles on the skin of a human wearer is a complex subject. This sensory feel/ comfort is associated with psychological perception of the wearer and partially depends on the low-stress mechanical properties like responses towards tensile loading/ stretching, shearing, bending, compression, surface friction (roughness) under low-stress conditions experienced by touching, stretching, bending, compressing and rubbing (surface roughness) of the fabric[1] by hand. This sensory feel can be objectively measured by the "hand value of garment or fabric" or "fabric handle"[2], which can be determined quantitatively by the Kawabata Evaluation System (KES) or FAST system, etc.

KES Instrument comprises mainly four modules, i.e., KES-FB 1 for tensile and shearing attributes, KES-FB 2 for bending attributes, KES-FB 3 for compression attributes, and KES-FB 4 for surface attributes, i.e., surface frictional (Surface roughness and smoothness properties) to be measured under low-stress conditions. One can measure a total of 16 parameters using all four of these modules. These parameter attributes measured at low-stress conditions under each module give the primary hand value for each attribute.

This total hand value (THV)[3] of textile fabric meant for making a garment is an essential parameter for the selection of fabrics for different wear occasions and seasons like casual/ leisure garments in

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summer and winter or special purpose-specific garments like workwear, sportswear etc. for a feeling of skin sensory feel to human body[4] for the wearer for different purpose and weather conditions with small movement associated with low deformations due to movement of the organ of human body[5].

'The Hand Evaluation and Standardization Committee' (HESC) under the leadership of Prof. Kawabata designed and developed a simulated newer instrumental technique for the estimation of primary hand values and Total Hand values of any textile fabric, by using KES[6] finally to evaluate the THV of fabric concerning low stress tensile, bending, shearing, compression behavior besides its surface characteristics (both surface roughness and geometrical roughness)[7] to obtain an ideal fabric[8] of desirable hand and feel.

Hence, these skin sensory tactile feeling parameters at low-stress conditions like tensile stretching/ extensibility, shearing, bending/ flexibility and surface friction caused due to surface roughness/ smoothness are to be essentially evaluated for assessing total skin sensory/ tactile feel during its wear. This skin sensory feel of fabric also depends on the type of chemical processing done and finishes (temporary and permanent) applied on any textile fabric besides the type of fibers, structure of the yarn, weave type and fabric construction, etc.[6], which are generally fixed for a particular variety of a textile fabric and so it can only be altered by suitable chemical processing or finishing process.

The effect of fibre composition, fabric structure, usual chemical finishes, etc., on the hand value of different textile fabrics was studied by various researchers[9-17]. Researchers had already studied the mechanical attributes (like bending, tensile, shearing, etc.) and surface characteristics after different chemical finishing processes on linen[13], silk[14], and regenerated cellulosic[15] like modal and their blends with cotton and other fibre-based fabrics with the help of KES. The influences of different physical properties on the comfort performance of bedsheet fabrics were also studied[16]. Effect of silicone softener treatment on handloom woven fabrics, by measurement of bending length, surface roughness index by digital image processing technique, and fabric softness assessment by use of fabric feel tester, is reported to evaluate the effects of the application of varied classes of silicone-based compounds to improve feel and hand value[17] to a variety of textile fabrics. Initially, the KES technique was used for men's shirts in different season, etc., and later this technique was expanded to include other types of fabrics for different wear purposes such as materials for women's dresses, in different season, nonwoven fabrics, blankets, and some specialty and functional textiles like disposable diapers, terry fabrics/futon cloth[17] by determining suitable application based formulae utilizing the primary hand values to arrive at total hand value (THV) as per specific end uses for different types of textiles, by putting calculated values of required coded coefficients of different regression equations fit for different end uses to match specific requirement for specific applications and season.

In our earlier part of the preliminary study, reported elsewhere,[18] it is confirmed by the results of bio-dyeing of cotton fabrics with catechu natural dye using potash alum (K-alum) + Gallnut(GN) dual pre-bio-mordanting facilitates to achieve maximum colour strength with overall good colour fastnesses to washing, rubbing and UV-light exposure by the action of two mordants forming a giant complex of [Fibre(cotton) -Mordant-1(K-alum) -Mordant-2(Gallnut) -dye(catechin)]. Use of a single mordant, i.e., either K-alum or gallnut or harda, etc., is known and it gives lower colour strength and poorer colour fastness for natural dyeing even with catechu. While using two different types of natural mordants applied in sequence, the formation of 'Fibre-Mordant-1-Mordant-2-Dye' gigantic bigger complex renders higher fixation of dye (catechin) and consequent higher colour strength with overall higher grade of washing and rubbing fastness, as observed in our preliminary study published elsewhere [18]. So, in the present study, with the expectation of higher fixation of catechu dye with improved colour strength and higher grade of colour fastness to washing and rubbing, etc, two different natural / bio-mordants, i.e., dual mordants, are used as a newer approach in this research. Hence, these two selective natural /bio-mordants [K-alum and Gallnut are used in the present work for pre-mordanting before catechu dyeing. The hydroxyl group of polyphenolic gallo-tannins or -COOH group of Gallic acid may attach through the vacant orbital of Al (from K-alum) by either formation of coordinated covalent bonds and also by creating some weaker hydrogen bond links to form the above-mentioned gigantic bigger complex showing higher colour yield, uniform dyeing and overall higher colour fastness rating, for catechu dyeing at standardized dyeing conditions after the said dual pre-bio-mordanting. While, addition of eucalyptus leaf extract for bio-finishing after K-al + GN dual bio-mordanting followed by catechu dyeing forms a much bigger complex of 'Fibre-Mordant-1-Mordant-2-catechu Dye -Eucalyptus component finish much bigger complex of this catechu bio-dyed and eucalyptus bio-finished [18],[19] cotton fabric sample.

Several important references, including our preliminary work published elsewhere[18], also indicate that Eucalyptus leaf extract is an effective bio-finishing agent for both antimicrobial and UV protection. Additionally, ZnO/ nano-ZnO is very effective for antimicrobial and UV protection finishes. Chitosan/nano-chitosan is also known to possess good antimicrobial properties19. Details of chemical structure and properties, and composition, for Eucalyptus leaf extract[20-24] (Eucalyptus globulus), nano-Chitosan[19,25] (obtained from fish shell-based chitin), nano-Zinc Oxide (obtained from mineral-based relevant natural Zn Ore)[26] are available in our preliminary part of the work reported earlier [18].

From the literature survey, it was revealed that to enhance the antimicrobial and UV protection properties of natural fibres like cotton and jute, suitable antimicrobial agents are chitosan/nano-chitosan, ZnO/nano-ZnO, and Eucalyptus leaf extract, as reported earlier by other researchers for cotton and other textiles. However, these have not yet been applied to catechu-dyed cotton fabrics using dual pre-mordanting.

Therefore, these three bio-finishes, though obtained from different sources having differences in chemical/biochemical criteria, are considered most suitable for application and comparison in their ability to impart the desired antimicrobial and UV-protective properties when applied on catechu-dyed cotton fabrics. Hence, these three bio-finishing agents were selected for the present study, as no comprehensive research has yet been reported on their use for catechu-dyed cotton fabrics with K-alum + Gallnut dual bio-mordanted cotton fabric in this route for producing eco-garments / medical textiles, etc.

So, after identifying this gap in the literature, the present study investigates the application of said dual bio-mordanting followed by post-dyeing bio-finishing using three different bio-finishing agents: nano-chitosan, nano-ZnO, and eucalyptus leaves extract. So, although these three bio-finishing agents are from different sources, there is no bar in making a comparison of their effects/impact on any textile-related low-stress mechanical properties of the treated fabrics, which much depends on their mechanism of physical and chemical interaction with the cellulose of cotton.

So, the novelty of this work lies in the Process development for eco garment/medical textiles by this dual pre-bio-mordanting and subsequent catechu dyeing, followed by bio-finishing with Eucalyptus leaf extract, Nano-ZnO, and Nano-chitosan, all taken from natural sources of comparable eco-safe bio-finishing agents.

From the above background information, it appears that still there is no integrated study available on the determination of hand value of cotton textile fabrics after treatment with different bio-based/ natural materials, i.e., bio mordanting, natural dyeing (with catechu) and biochemical finishing with eucalyptus extract and nano-chitosan and physical intervention with nano ZnO coating with silicone softener and hence the present work is undertaken as an attempt to fill this gap to study the primary hand values and total hand values of these 3 different bio finished cotton woven fabrics to judge the suitability of their use in particular type of garment/end use in specific season.

The present study thus evaluates the efficacy of the said three different bio-finishing (after dyeing) agents using eucalyptus leaf extract, nano-chitosan, and nano-ZnO to improve their antimicrobial and UV resistance properties to the maximum level possible. Skin sensory tactical comfort properties in terms of responses to tensile loading/ stretching, shearing, bending, compression, surface friction (roughness) under low-stress conditions as primary hand values and resultant total hand values of the said dual bio-mordanted and catechu dyed and subsequently bio-finished with above said three different bio-finishes used, were evaluated with Kawabata Evaluation System (KES)[6].

2 Materials and Methods

2.1 Materials

Desized, scoured, and bleached plain weave 100 % pure cotton-cellulosic fabric with 84 ends per inch (20 ends per cm) with warp count of 9.8 tex (Ne 60) and 74 picks per inch (19 Picks per cm) with weft count 10.7 tex (Ne 55), having fabric areal density of 145 g/m^2 and thickness 0.25 mm was procured from NTC Ltd showroom, Kolkata (coded as Sample S1 as control fabric).

Commercial grade Acetic acid (CH_3COOH), Sodium acetate and Sodium acetate- Acetic acid buffer for adjusting 4-4.5, 1 % sodium Hydroxide solution and Sodium chloride (NaCl) as electrolyte/ exhaustive agent were procured from E-Merck (India) to use in the present work.

Natural potash alum (Fitkiri, i.e., K_2SO_4. $Al_2(SO_4)_3.$ $24H_2O$ or simply as K.Al $(SO_4)_2$, $12H_2O$) and Gallnut (maju-phall or Oak Gall, i.e *Quercus Infectoria*) used as bio mordants were procured from the local market (Kagalicharan Dutta & Sons, Burrabazar, Kolkata) to use in the present work.

Catechu (*Acacia catechu*) was purchased from the local market, and purification of Catechu was done as per earlier preliminary work reported elsewhere from the same group of authors[18].

2.2 Methods

2.2.1 Methods of Treatment for Sample Preparation

2.2.1.1 Extraction of Catechu, Gallnut and Eucalyptus leaves

Catechu was extracted after pre-soaking of dry catechu chunk in alkali for 1hr[27] minutes to make it easily soluble. Alkali pre-soaked catechu chunk was then taken into a beaker containing a requisite quantity of hot water and was further heated under a water bath at around 60-70 ^{0}C for 60 min, at MLR 1:20, at pH 10, by further addition of 1 % caustic soda, followed by straining it in a 60 mesh nylon filter-cloth. The filtrate was then neutralized with acetic acid to bring the pH to 6-7, to get the final dye liquor of 30 % catechu (On oven-dry mass/weight of source materials of catechu powder/chunk) extract, which is equivalent to 3 % purified dye solution of pure color components of catechu, as evident after purification of dry catechu chunk.

Gallnut (Maju-phal) was extracted in aqueous media from its ground powder of gallnut following optimal extraction conditions at pH 11, Material: Liquor Ratio 1:20, extraction temperature 80 °C and extraction time 45 min[27] and finally it was strained in a nylon 60 mesh filter fabric.

Aqua-ethanolic extract of eucalyptus leaves was prepared in 50: 50 mixture of water and ethanol 100 gm of eucalyptus dried leaves (solid dry mass) were pre-weighed and poured in 1 liter of 50;50 Water: EtOH (Ethanol) mixture for obtaining an aqua-alcoholic extract of eucalyptus (*Eucalyptus globulus Labill.*) leaves at pH-5 to 5.5(controlled by dropwise addition of 10% acetic acid) and subsequently heating at 50^{0}C for 30 min. Finally, this solution was filtered to obtain a light-yellow colored 10 % aqua-alcoholic extract of eucalyptus globulus leaves, as a natural plant-based UV-absorber cum antimicrobial finishing agent [19] for applying on catechu dyed cotton, along with 1/5th lemon juice as a natural acidic catalyst.

2.2.1.2 Dual Pre-Bio-Mordanting and Catechu Dyeing

15 % overall concentration of natural K-alum + Gallnut (75:25) dual pre-mordanted cotton fabric was put into the 30 % catechu extracted dye liquor bath kept at around 30 ºC and continued dyeing with stirring for 10 min at this temperature. Subsequently, heating was continued in the RBE make lab beaker IR dyeing machine to raise the final dyeing temperature from 30 ºC to 70 ºC, and thus, dyeing was continued for a total of 60-90 min. 5gpl NaCl salt was added as an exhausting agent to the dye bath in two installments with 15 min intervals with gradual heating at 2-3 ºC per min at optimum dyeing conditions (Optimum dyeing conditions were obtained from our earlier part of the study communicated showing final dye bath pH as 4.0-4.5 (pH was maintained with acetic acid + sodium acetate buffer), MLR- 1:20, dyeing Time - 60 min and dyeing temperature of 70 ºC and salt concentration -5 gpl). After dyeing was over, dyed cotton samples were rinsed and washed twice thoroughly in running tap water and then lightly soaped in 2 gpl standard non-ionic neutral soap at 50 °C for 15 min for removal of adhered and non-bonded dyes, followed by washing in tap water repeatedly and then subjected to final air-drying to obtain catechu dyed cotton (Sample S2).

2.2.2.2 Bio-Finishing

Selective dual bio-pre-mordanted and catechu-dyed cotton fabric samples were subjected to post-dyeing finishing with 10 % aqua-ethanolic (water: ethyl alcohol-50:50) extracts eucalyptus leaves with further addition of 1/5th of acidic catalyst (5 gpl lemon juice or equivalent citric acid) subjecting to pad (at 100 % weight pick up)- dry (at 100 ºC for 5 min) and cure (at 120 ºC for 3 min) process (Sample S3).

Selective dual bio-pre-mordanted and catechu-dyed cotton fabrics were padded maintaining 100 % wet pick-up using 0.2 % nano chitosan (solubilized in proportionate hot water+ acetic acid + Isopropyl alcohol mixture after homogenization with stirring after addition of Sodium Tri-polyphosphate (STTP) detergent/ surfactant, to prevent partial agglomeration in nano-chitosan suspension) by pad (2 dip-2 nip) -

dry (100 °C for 5 min) -cure (120 °C for 3 min) process for improving wash fastness and rubbing fastness and antimicrobial properties for nano-chitosan finished cotton (Sample S4).

Nano-ZnO suspension (0.2 %) was admixed with 10 ml of hydroxy-methyl-amino silicone (HMAS) emulsion and water to make up the total volume of 100 ml with stirring, to make a homogeneous suspension/ dispersion of nano-ZnO to coat/embed with silicone emulsion containing nano ZnO and was applied on fabric surface as a physical coating interventionin presence of 5gpl lemon juice catalyst (1/5th of nano-ZnO used). Selected samples of dual bio-pre-mordanted and catechu-dyed cotton fabrics were then immersed and batched into nano-ZnO embedded amino silicone suspension, which were then batched for 30 min for well spreading and soaking of the Nano-ZnO + HMAS suspension on the dyed cotton fabrics and then those fabric samples were subjected to padding (at 100 % weight pick up) followed by drying at 100 ^{0}C for 5 min followed by curing at 120 ^{0}C for 3 min to obtain nano-ZnO finished cotton fabric (Sample S5).

2.2.2.3 Summary of the Sample Code of the Selected Samples

Sample Code Nos	Description of sample treatments
S1	Scoured and Bleached Cotton fabric (Control)
S2	Cotton Fabric + 15% Dual Bio-Mordant (K-Alum+ Gallnut) +30 % Catechu Dye
S3	Cotton Fabric+ 15 % Dual Bio-Mordant (K-Alum+ Gallnut) + 30% Catechu Dye + 10% Aqua-ethanolic extract of Eucalyptus Leaves Extract + 5gpl lemon juice
S4	Cotton Fabric+ Dual Bio-Mordant (K-Alum+ Gallnut) + 30% Catechu Dye + 0.2 % Nano-Chitosan finish with 1% Acetic Acid
S5	Cotton Fabric+ Dual Bio-Mordant (Gall nut+ Alum) + 30% Catechu Dye+ 0.2% Nano ZnO finish with 0.04% Citric acid and 2% Hydroxy-methyl-amino silicone (HMAS) emulsion.

2.3 Test Methods

All the selected samples of cotton fabric were conditioned at (65 ± 5) % relative humidity and (27 ± 2) ^{0}C temperature for 48 hrs, as per ASTM standards for conditioning of Textiles[28], before all physical and mechanical testing were done.

2.3.1 UV VIS Absorption Spectral Analysis and Characterization

UV VIS Spectral scans were obtained by wavelength scan of diluted aqueous-ethanolic (water: EtOH 50:50) extract of Eucalyptus leaves (*Eucalyptus globulus Labill.)* and diluted aqueous suspension of nano-chitosan and nano ZnO, using a Hitachi make- U-2000 -UV-VIS absorbance Spectrophotometer, for 190 nm to 1100 nm.

2.3.2 Particle Size Analysis of Nano Chitosan and Nano ZnO

Particle size analysis of Nano-ZnO and Nano-chitosan suspension was obtained using a particle size analyzer (Malvern Panalytical Ltd) as per the Dynamic Light scattering (DLS) method' at ICAR NINFET, Kolkata.

2.3.3 Study of Surface Topology by FE-SEM and TEM Pictures

The high resolution Field Emission Scanning electron microscopy (FE-SEM) of selected samples was carried out by using FEI-Quanta -200F, FEG high resolution Scanning Electron Microscope, using earlier prepared samples for SEM study with Gold-Palladium alloy coating, which were further examined in a Field Emission scanning electron microscope (FE-SEM) with a resolution of 1.0 nm (15kV) with accelerating voltage 0.1 to 30 kV, for untreated and differently mordanted /dyed and bio finished fabrics samples, by taking out fibres from corresponding fabric samples.

High-resolution TEM (Transmission Electron Microscopy) pictures of the surface of selectively treated/mordanted /dyed/bio-finished cotton fabric samples were obtained by using HRTEM (JEOL2M 2100 TEM) instruments following standard methods from SAIF, IIT Mumbai.

2.3.4 Kawabata Evaluation System of Testing of Selective Fabrics

It is an advanced method for assessing low-stress mechanical characteristics like thickness, bending, tensile, shearing, compression, weight per square meter, etc. and surface roughness characteristics of a fabric after conditioning in a standard atmosphere[28.]

Under standardized testing conditions, different low-stress mechanical properties were evaluated by the KES method [6,] covering tensile strength, bending, shearing, compression, as well as fabric dimensions like fabric thickness, and weight, etc. which were evaluated using the KES-FB1 system.

2.3.4.1 Tensile and Shear Properties

For the tensile testing mode of the Auto Tensile and Shear Tester (Model 2012), a unidirectional force of up to 500 gf/cm was applied to a fabric specimen measuring 20 cm × 5 cm. The force was applied between two sets of clamps in a single direction. After reaching the maximum applied force, the instrument allowed the specimen to return to its original position at a constant strain rate. The stretching was unidirectional, but the fabric underwent biaxial deformation. This procedure was repeated for specimens oriented in the perpendicular fabric direction.

In the shear testing mode of the KES-FB1 system, a constant unidirectional tension of 10 gf/cm was initially applied. Subsequently, a shear force was introduced in the transverse direction, inducing shear deformation up to a maximum shear angle of 8 degrees. The shear deformation was then reversed by gradually returning the shear angle to zero degrees.

2.3.4.2 Bending Properties

The bending behaviour of the fabric was assessed using the Bending Tester (Model 1996). Fabric specimens measuring 20 cm × 1 cm were subjected to a standardized curvature of ±2.5 cm^{-1}. Bending and recovery characteristics were evaluated in both the warp and weft directions, as well as on both the face and back sides of the fabric.

2.3.4.3 Compression Properties

The compression characteristics of the fabric were evaluated using the Auto Compression Tester (Model 2012). A compressive load of 50 g/cm^2 was applied to a fabric specimen measuring 20 cm × 20 cm and then gradually released using a movable plunger. From this test, several parameters were determined: fabric thickness at a low compressive load of 0.5 g/cm^2 (T_0), fabric thickness at the maximum load of 50 g/cm^2, work done during compression (WC) and compressive resilience (RC %).

2.3.4.4 Surface Friction characteristics

Surface roughness and surface friction criteria were evaluated using the Surface Tester (Model 1996). Under a preset normal load, the fabric was moved in both forward and backwards directions. Geometrical roughness and the coefficient of surface friction were recorded using electronic sensors integrated into the instrument. All the Kawabata tests done in each of the four modules of the KES instrument are the average of 5 tests for each. All these tests were carried out at ICAR-CIRCOT, Mumbai.

2.3.5 Measurement of UV Protection Factor (UPF)

UV protection factor (UPF) of untreated and treated cotton fabrics was evaluated according to AATCC: 183: /2004 (revised in 2010/2014) method[29], using Lab-sphere Inc., USA, make UV-Transmittance Analyzer:

$$\mathrm{UPF} = \frac{\int_{\lambda 1}^{\lambda 2} E(\lambda)S(\lambda)*d\lambda}{\int_{\lambda 1}^{\lambda 2} E(\lambda)S(\lambda)*Td(\lambda)}$$

$$\text{UV Transmission (\%)} = \frac{\sum_{\lambda 1}^{\lambda 2} T(\lambda)}{\lambda 2 - \lambda 1}$$

where E_λ represent the relative erythemal spectral radiance, S_λ represents the solar ultraviolet radiance, e UV spectral irradiance strength in W.m-2.nm-1, T_λ represents the spectral UV transmission through the fabric, Δ_λ represents the bandwidth of spectral radiance in mm, and λ is the wavelength of spectral radiance in nano-metre. These tests were done at ICAR-CIRCOT, Mumbai.

2.3.6 Measurement of Anti-Microbial Property

Antimicrobial property of untreated and selectively treated cotton fabric samples was evaluated according to the relevant AATCC standard method (AATCC-100-2012)[30] using (i) Klebsiella pneumoniae: AATCC 4352 (Gram +ve bacteria) and also using (ii) Staphylococcus aureus: AATCC 6538 (Gram -ve bacteria) mounting the samples on suitable Petri plates incubated at standard conditions at 37°C incubated for 24 h followed by counting of bacteria colonies and assessing per cent reduction of areas of bacterial growth, using the following formula :

$$R(\%) = (A - B)/A \times 100\%$$

where R represents the % reduction of bacterial growth under test conditions and A and B are the area or numbers of counted bacterial colonies as assessed after the tests were done at ICAR-CIRCOT, Mumbai.

3 Results and Discussion

3.1 Pre-characterization of Bio-Finish Materials Used

3.1.1 Eucalyptus Leaves Extract/Powder

Pre-characterization of catechu dye extract has already been studied and published by us elsewhere[18] in our preliminary work on it. Pre-characterization of aqua-ethanolic (water: EtOH 50:50) extract of Eucalyptus leaves (*Eucalyptus globulus Labill)* was carried out by its ~~UV~~ UV-VIS spectral scan as shown in Fig. 1 below.

UV VIS absorption Spectra for aqua-ethanolic extract of eucalyptus leaves is shown in Fig. 1 (for an aqua-ethanolic extract of eucalyptus leaves) which shows a large clear and sharp peak at 256 nm in UV-Zone showing a strong presence of uv-absorption

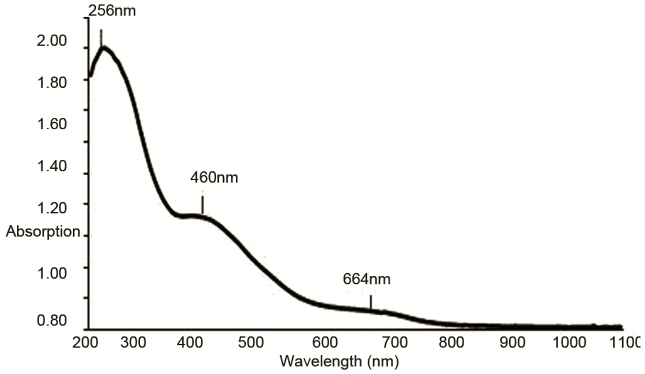


Fig. 1 — UV VIS absorption Spectra for the aqueous-ethanolic extract of eucalyptus leaves

character of all types of flavonoids and tannins present in it, showing large peak height at 256 nm spread over at 220 nm to 370 nm, besides a larger flat hump at 460nm in visible zone, which can be considered as λ_{max}. Another tiny notch, similar to a flattened peak, is present in the visible zone considered, for the minor component of it, at 664 nm. LC-MS analysis of aqua-ethanolic extract and phytochemical analysis are also reported in our earlier part of publication elsewhere, [19], indicating compositional analysis and the total phenolic content in the eucalyptus leaf extract. Another researcher [31] has reported the total phenol content of Eucalyptus as 234 mgGAE/g plant material, and the total flavonoid content is 35 mg RE/g plant material, besides a faint yellow colour of the extract. The antimicrobial and UV-resistant properties of Eucalyptus leaves are believed to be due to the action of flavonoids, Quercitrin, Rutin, Hyperoside, and Eucalyptol present in it [32] .

3.1.2 Nano-Chitosan and Nano-ZnO

Use of biopolymers in bio-finishing of natural fibre-based textiles like cotton, particularly for medical textile applications, holds great importance for sustainable development. In this study, chitosan nano-finish and ZnO nano-finish are utilized, which can further expand the potential applications of these biomaterials in producing eco-garments. This development aims to create green and sustainable antimicrobial and UV-resistant medical textiles that address both environmental and health-related challenges faced by the human wearer, promoting wellness behaviour.

The pre-characterization, including UV–VIS absorption spectral scan of the nano-ZnO and nano chitosan suspension, has been studied earlier by other researchers. ZnO was earlier identified as an n-type semiconductor with a wide band gap (3.37 eV with a UV-VIS absorption peak between the range of 350–380 nm wavelength[33], with a UV-VIS sharp absorption peak also appearing at 378 nm, as a specific characteristic UV-VIS absorption peak of nano ZnO, as shown in Fig. 2(a). While Suspension of nano-chitosan in aqueous media shows its UV absorption spectral scan in Fig. 2(b), showing lesser UV absorption except one peak at 380 nm and no peaks in the visible zone [34]. Both these UV VIS spectral scans are very similar, matching with corresponding previous results in publications of other researchers [33-34], and hence they confirm the purity of both nano-ZnO and nano-chitosan applied on cotton fabrics.

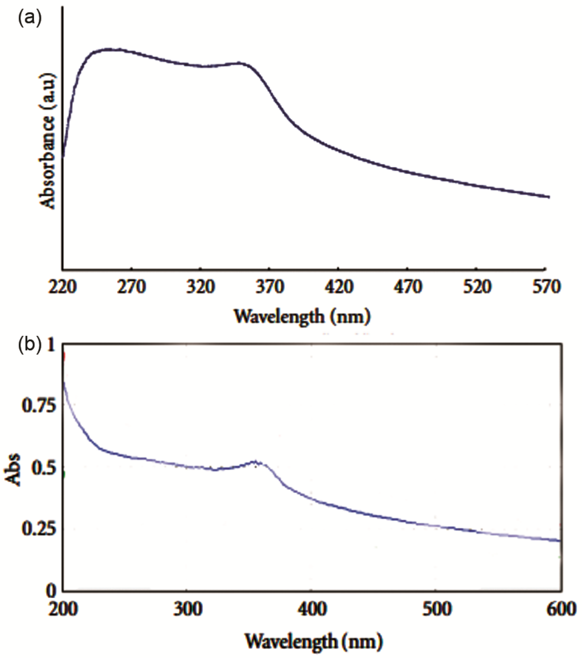


Fig. 2 — (a) UV-VIS Absorption Spectra of Nano-ZnO, and (b) UV VIS Absorption Spectra of Nano-Chitosan

However, for the present work, the most important aspect is the dispersion of nanoparticles after bio-finishing and possible agglomeration of both of them and their impact on modifying surface frictional criteria on the finished fabrics, for which FE-SEM and TEM pictures were taken to be examined. Initially, the suspensions of nano-chitosan and nano-ZnO are characterised by their particle size analysis to understand the overall dispersion of these nano bio finishes and to understand the impact of these bio finishes on catechu-dyed cotton textiles by measuring and analysing their tactile comfort, particularly surface roughness or frictional criteria.

3.1.3 Particle Size Analysis of Nano-Chitosan and Nano-ZnO Suspension

Particle size for nano-Chitosan (volume and intensity) and nano-ZnO (volume and intensity) suspension has been shown in Fig. 3 (a) and 3(b) and Fig. 4 (a) and 4(b), respectively and analysed for their pre-characterization.

Nano-chitosan particles, being more prone to agglomeration and possessing larger aggregate sizes as compared to nano-ZnO, deposited on the fabric surface in a non-uniform manner, and this non-uniform deposition contributed increase in surface abrasiveness and roughness.

As illustrated in Fig. 3(a), the average particle size based on volume distribution was approximately. 2016 nm, with a minimum particle size of 1000 nm and a maximum of 5037 nm, with a polydispersity index of 0.505. likely due to agglomeration. The particle size versus intensity plot shown in Fig. 3(b) presented a similar distribution pattern, supporting the findings observed in the particle size distribution by volume % data. However, there is a minor peak in the range of 100 nm to 200 nm that was also observed in intensity % data, suggesting the presence of a small fraction of truly nano-sized chitosan particles in the suspension. Thus, it indicates that the majority of nano-particles of chitosan fell within the larger size range of 1000 nm to 5037 nm and had an average of 2016 nm, with a small fraction having 100 nm to 200 nm also.

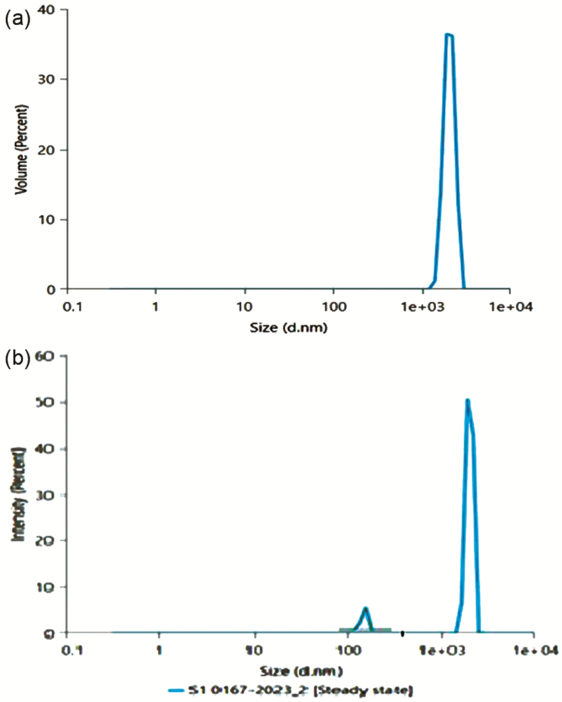


Fig. 3 — (a), (b) Particle size distribution for Nano-Chitosan dispersive Solution (a) by volume % and (b) by Intensity % respectively

As shown in Fig. 4(a), the average particle size of nano-zinc oxide based on volume per cent was approximately 1.025 nm, with a minimum size of 0.83 nm and a maximum of 1.22 nm by volume % data, with a polydispersity index of 1.0, indicating a relatively narrow but uniform particle size distribution at the nano-scale. The particle size versus intensity % plot in Fig. 4(b) revealed a bimodal distribution. The first, tiny, smaller peak corresponded to particles ranging from 0.83 nm to 1.2 nm, representing approximately 8–9% of the total particles, with an average size of approximately 1.025 nm hydrodynamic diameter. The second, more prominent, long height peak, comprising roughly 85–90% of the particles, is in the nano scale, showing particle sizes ranging from 269.5 nm to 520 nm, indicating the formation of a small amount of agglomeration for ZnO nanoparticles.

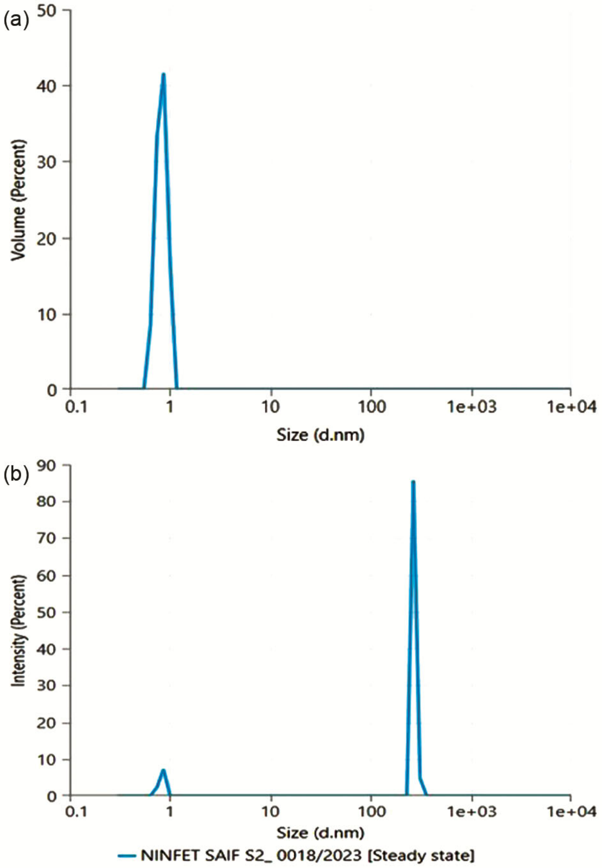


Fig. 4 — (a), (b) Particle size distribution for Nano-Nano-ZnO dispersion (a) by volume % and (b) by Intensity % respectively

Thus, the majority of nano-ZnO particles remain extremely small as nano particles by both volume % and intensity %, but also show some minor agglomeration, leading to the formation of larger clusters of rod-like ZnO nano crystals through coalescence. The combined interpretation of Fig. 4(a) and Fig. 4(b) indicated that nano-ZnO exhibited predominantly very well distribution ZnO nanoparticles, with only limited agglomeration. The least aggregation observed for nano-ZnO suspension may be attributed to the stabilising effect of the amino silicone emulsion as a medium of distribution, which is likely to function as an agglomeration inhibitor, thereby maintaining the dispersion stability and preventing ZnO nano-particle coalescence. It may be noted that there is a difference in particle size and distribution between the solution of Nano-ZnO and Nano-chitosan [35-37].

Some studies showed contradictory reports for the dispersion of nanoparticles of chitosan under different conditions of preparation and treatments. One such previous report [35] indicated that nano-chitosan particles typically exhibit an average size around 275 nm, ranging between 100 nm to 1000 nm or even larger, particularly after homogenization and use of an agglomeration inhibitor. While numerous other reports [36-37] highlighted the strong agglomeration tendency of nano-chitosan particles, which was attributed to their amino-protonated structure in acidic media. This amino-protonated structure promoted electrostatic attraction toward the negatively charged oxygen atoms of water molecules present in the application bath. When cotton fabric was immersed in water, negatively charged sites were generated on the cellulose surface, which further enhanced the coalescence of nano-chitosan particles, leading to increased aggregation on the cotton fabric surface.

3.2 Fabric Constructional Attributes

Control cotton fabric and dual bio-pre-mordanted fabric, catechu dyed fabric and differently bio-finished cotton fabrics are evaluated for changes in fabric construction and dimensional properties like thickness, weight per sq. cm (or g/m^2) by KES-FB1, as shown in Table 1

The untreated Scoured and Bleached control fabric (Sample -S1) had an initial thickness of 0.256 mm with a fabric weight of 145.3 g/m^2. Subsequent treatments applied to Sample S2 to Sample S5 showed an increase in both thickness and Fabric weight in g/m^2 as outlined in Table 1. After treatment with dual bio-mordant, followed by dyeing with catechu dye (Sample S2), showed 16.24 % weight gain and 67 % increase in thickness over Sample S1. This increase in weight and thickness in Sample S2 may be viewed as a combined effect of fabric shrinkage and deposition of bio-mordant/ dye molecules on the surface. Among all, Sample S3 exhibited a substantial 27.25 % enhancement in weight and approximately. 109 % increase in thickness relative to Sample S2. These further enhancements during finishing for Sample S3 may be viewed as entrapping of Eucalyptus leave extract with the help of citric acid as a cross-linking agent. An increase in both thickness and weight for Sample S4 and Sample S5 is due to major agglomeration of nano-chitosan particles and minor agglomeration of ZnO nano-rod particles on the fabric surface, respectively, which are later confirmed by FE SEM imaging.

3.3 Tensile Attributes

The low-stress tensile attributes of control fabric and other treated fabric samples are shown in Table 2.

Table 2 demonstrated that all treated fabric samples exhibited a reduction in LT values compared to the control fabric (Sample S1). Since the LT value represents the elasticity of the fabric, a reduction in this parameter suggests increased extensibility, which in turn implies improved drapability and enhanced wearer comfort. A substantial reduction in LT value was observed for Sample S2 (15.78 %). Statistical analysis indicated that the changes in LT values were significant in both the warp and weft directions at a 95 % confidence level.

Table 1 — Fabric constructional attributes and their changes by different treatments

Sample No.	Thickness mm	Increase in % of thickness	Thickness at max. pressure, mm	Fabric weight, g/m^2	Increase in fabric wt. after treatment, %
Sample S1	0.256	--	0.171	145.30	--
Sample S2	0.429	67	0.339	168.90	16.24
Sample S3	0.536	109	0.368	184.90	27.25 (9.47 % w.r.t. Sample S2)
Sample S4	0.461	80	0.309	170.30	17.20 (0.83 % w.r.t. Sample S2)
Sample S5	0.436	70	0.311	171.80	18.23 (1.72% w.r.t. Sample S2)

S1: Control; S2:Catechu Dyed S3:Eucalyptus finished, S4:Nano-chitosan finished and S5:Nano-ZnO +Amino silicone finished

Table 2 — Results of Low-stress tensile attributes by using KES-FB1A Tensile Tester

Sample No.		LT	Significant at 95 %	WT, gf.cm/cm^2	RT, %	EMT, %
Sample S1	Warp	0.795	----	5.50	47.63	2.77
	Weft	0.674	----	17.07	39.16	10.13
	Avg.	0.735	----	11.28	43.40	6.45
Sample S2	Warp	0.649	YES	8.43	40.14	5.20
	Weft	0.588	YES	11.50	35.67	7.82
	Avg.	0.619	----	9.97	37.90	6.51
Sample S3	Warp	0.688	YES	17.80	25.47	10.35
	Weft	0.688	YES	8.28	37.90	4.96
	Avg.	0.678	----	13.04	31.69	7.66
Sample S4	Warp	0.665	YES	10.38	31.22	6.24
	Weft	0.631	YES	8.82	35.65	5.59
	Avg.	0.648	----	9.60	33.44	5.91
Sample S5	Warp	0.646	YES	11.63	27.53	7.18
	Weft	0.609	YES	10.05	31.58	6.62
	Avg.	0.627	----	10.84	29.56	6.90

*LT- *Linearity of load-extension curve, WT- Tensile Energy, RT- Tensile Resilience, EMT- Tensile Elongation

All treated samples except Sample S3 showed a decrease relative to the control fabric. Sample S3 demonstrated a 15.60 % increase in WT, suggesting improved energy absorption during extension. This may be attributed to the cross-linkage of Citric acid. Conversely, the highest reduction in WT of 14.89 % was observed for Sample S4 compared to the control fabric.

As evidenced from Table 2, Sample S3 demonstrated 15.60 % more resistance to external stress than Sample S1 under low strain conditions, indicating higher stretchability. The EMT results revealed that Sample S3 exhibited 18.76 % more elongation than the control fabric. This increased stretchability was accompanied by a 26.98 % reduction in RT, indicating difficulty in recovering to its original size after removal of tensile forces compared to Sample S1. This may be due to the cross-linking effect of Citric acid used here as a catalyst.

By comparison, RT and EMT values for Sample S3 implied that the finishing treatment increased the stretchability, but the stretch may be mostly permanent. The fabric had around 26.98 % less tensile resilience or less ability to return to its original shape after removal of low external stress. Warp and weft-wise stress-strain diagrams under low stress are shown in Fig. 5 [(a) to (e)].

It appears that there was an increase in tensile elongation for Fig. 5 (c) (Sample S3) compared to Fig. 5 (a) (Sample S1), against the actual stress-strain diagram of control, indicating higher stretchability for Sample S3

3.4 Bending Attributes

The bending attribute for control cotton fabric and dual bio-mordanted and catechu dyed, followed by the three types of finished cotton fabrics, is shown in Table 3.

Comparable data in Table 3 showed a decrease of bending rigidity for Sample S4 (-18.44 %) and Sample S5 (-11.82 %), while a rise was evident for Sample S2 (+13.03 %) and Sample S3 (+4.61 %). An increase in bending rigidity reflects strong resistance against bending force, i.e., difficulty in bending. Samples S2 and S3 exhibited higher resistance to bending as compared to Sample S1.

Relevant data from Table 3 indicated that bending rigidity (B) decreased for Sample S4 (-18.44 %) and Sample S5 (-11.82 %), but the same increased for Sample S2 (+13.03 %) and Sample S3 (+4.61 %). An increased bending rigidity implied higher resistance against bending force, meaning difficult to bend, thus stiffer. Therefore, Samples S2 and S3 were stiffer than Sample S1. The observed increase in bending rigidity for Sample S2 may be attributed to surface deposition of bio-mordant or dye molecules, while in Sample S3, it is likely due to the cross-linking effect of citric acid. In contrast, the reduction in bending rigidity observed in Samples S4 and Sample S5 can be explained by a decrease in both static and kinetic friction, potentially resulting from the smoothening effect caused by the filling of intermolecular gaps within the fibres by nanoparticles. From statistical analysis, it appears that all these test results for B were statistically significant for both warp and weft direction at a 95 % confidence level.

For bending hysteresis (2HB), a reduction in the experimental values was observed after these treatments. As 2HB reflects the ability of recovery of a fabric specimen after bending and a lower value indicates a better recovery characteristic from

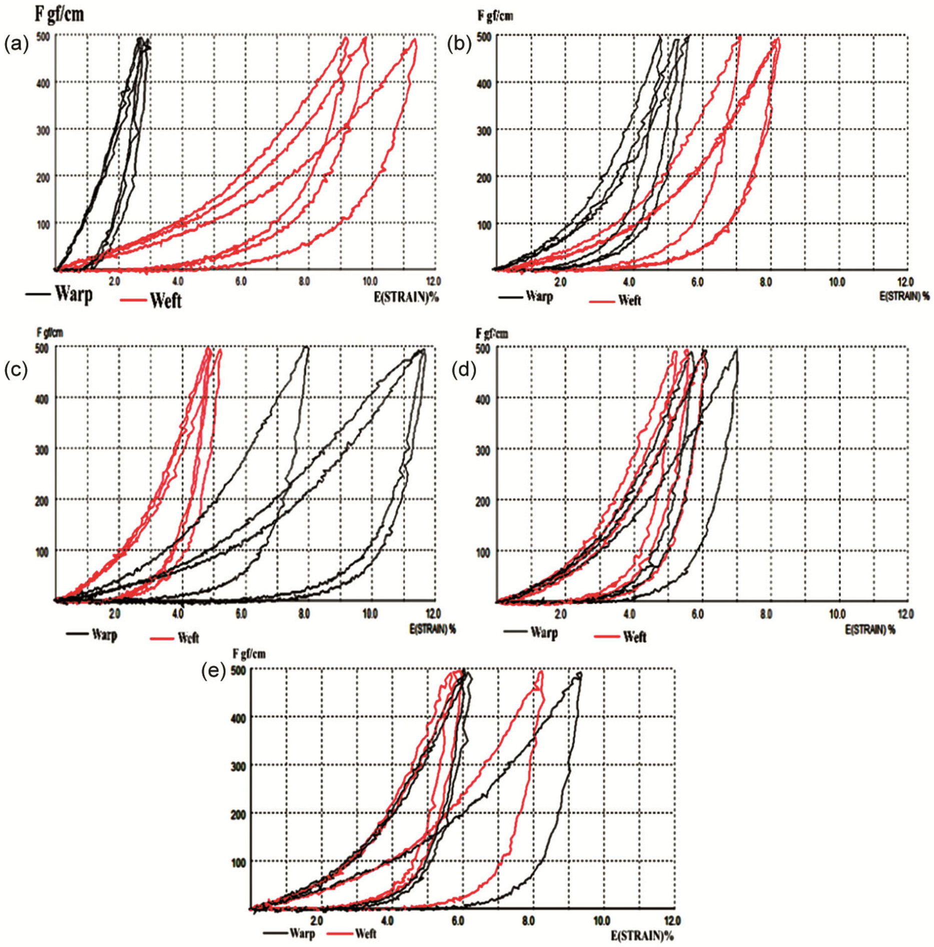


Fig. 5 — [(a) to (e)] Warp and Weft wise Stress-strain diagram for results of low-stress tensile attributes assessed by using KES-FB1A Tensile Tester

Table 3 — Results of Bending attributes assessed by using KES-FB2 bending tester

Sample No		B, gf. cm^2/cm	Significant at 95 %	2HB, gf. cm/cm
Sample S1	Warp	0.0722	-----	0.0667
	Weft	0.0274	-----	0.0291
	Avg.	0.0499	-----	0.0579
Sample S2	Warp	0.0682	YES	0.0623
	Weft	0.0447	YES	0.0369
	Avg.	0.0564	-----	0.0496
Sample S3	Warp	0.0412	YES	0.0383
	Weft	0.0631	YES	0.0637
	Avg.	0.0522	-----	0.0510
Sample S4	Warp	0.0409	YES	0.0315
	Weft	0.0406	YES	0.0371
	Avg.	0.0407	-----	0.0343
Sample S5	Warp	0.0408	YES	0.0322
	Weft	0.0472	YES	0.0446
	Avg.	0.0440	-----	0.0384

B- Bending Rigidity, 2HB- Bending Hysteresis

bending. Both Samples S4 and S5 showed a better bending recovery as compared to the control cotton fabric. Better bending recovery of S4 and S5 may be due to the effect of minor crosslinking. Warp and weft-wise bending diagrams under low stress are shown in Fig. 6(a) to Fig. 6(e).

From the analysis of the bending diagram (Fig. 6(a) to Fig. 6(e)), it is again confirmed that Samples S4 and S5 had better recovery from bending due to their lower 2HB values than Samples S1, S2 and S3 due to some crosslinking effect in S4 by crosslinking of celllulose-chitosan-cellulose and in S5 by crosslinking of cellulose-aledehyde-amino-silicone-cellulose aldehyde besides some minor crosslinking of citric acid used as a catalyst

3.5 Shearing Attributes

Shearing attributes for control cotton fabric and catechu dyed cotton fabrics, and the subsequent 3 types of natural bio-finished cotton fabrics are shown in Table 4.

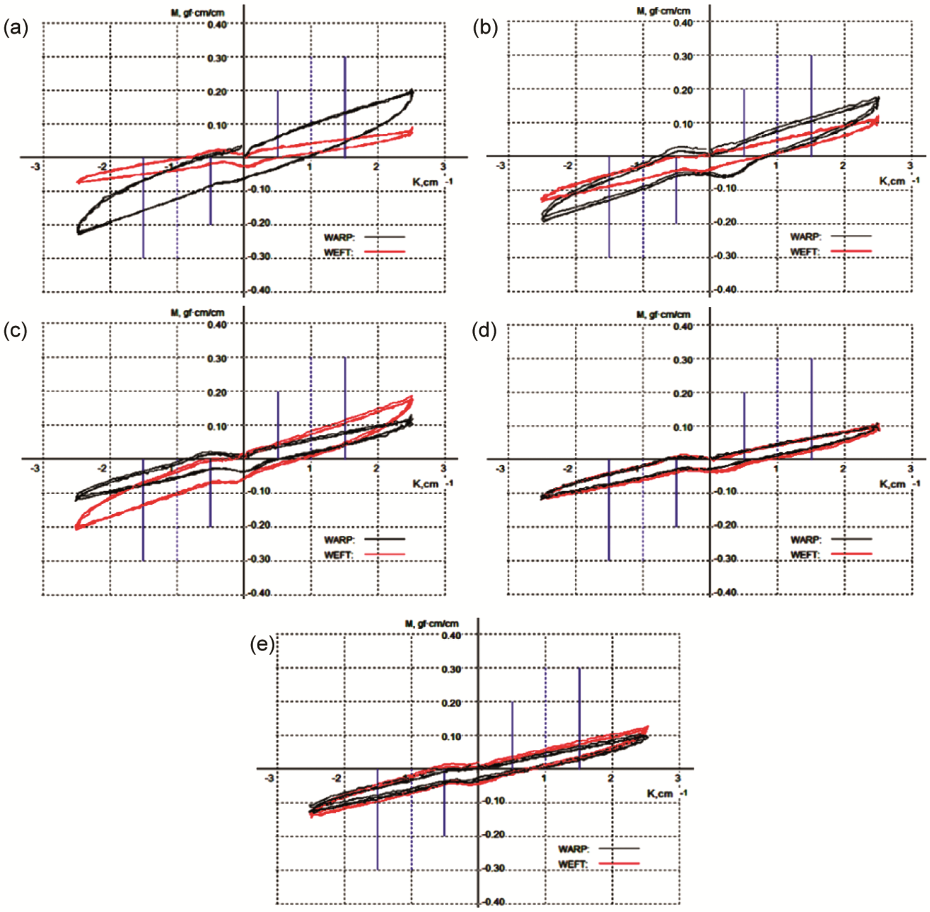


Fig. 6 — [(a) to (e)] Warp and Weft-wise bending diagrams under low stress conditions as assessed by using KES-FB2 bending tester

Table 4 — Shearing attributes as assessed by using KES-FB1 by shear tester

Sample No.		G, gf/cm. deg	Significant at 95 %	2HG, gf/cm	2HG5, gf/cm
Sample S1	Warp	0.81	----	2.10	2.94
	Weft	0.71	----	1.98	2.72
	Avg.	0.76	----	2.04	2.83
Sample S2	Warp	0.47	YES	0.93	1.63
	Weft	0.44	YES	1.08	1.75
	Avg.	0.45	----	1.01	1.69
Sample S3	Warp	0.55	YES	1.72	2.72
	Weft	0.49	YES	1.26	2.25
	Avg.	0.52	----	1.49	2.48
Sample S4	Warp	0.40	YES	0.97	1.55
	Weft	0.39	YES	0.89	1.45
	Avg.	0.39	----	0.93	1.50
Sample S5	Warp	0.36	YES	0.78	1.22
	Weft	0.33	YES	0.67	1.11
	Avg.	0.35	----	0.73	1.16

*G- Shear Rigidity, 2HG- Hysteresis of shear force at a shear angle, 2HG5- Hysteresis of shear force at ±50 shear angles

As per relevant data in Table 4, reductions in shear rigidity (G) values were observed in all the bio-finished samples as compared to Sample S1. Reduction in the value of shear rigidity (G) implies diminishing of resistance to shearing force, meaning the material under test exhibits softness with improved drapability. From statistical analysis, it was evident that changes in G were significant for both warp and weft direction at a 95 % confidence limit.

The data thus indicated a consistent reduction in shear rigidity (G) among all bio-finished samples. Maximum substantial reduction of G was observed for Sample S5 (-53.95 %), suggesting that Sample S5 would exhibit the highest degree of softness among all treated samples. The enhanced softness in Sample S5 was attributed to the incorporation of an amino silicone softener, which served as a dispersing and film-forming agent used to fix nano-ZnO particles. In contrast, minimum reduction of G was observed for Sample S3 (-31.58 %) might be due to the minor crosslinking between cellulose and citric acid (used as a catalyst), besides possible crosslinking of cellulose and polyphenolic compounds / gallic acid present in eucalyptus leaf extract.

The values of 2HG and 2HG5 represent the ability of the fabric to recover from low and high shear

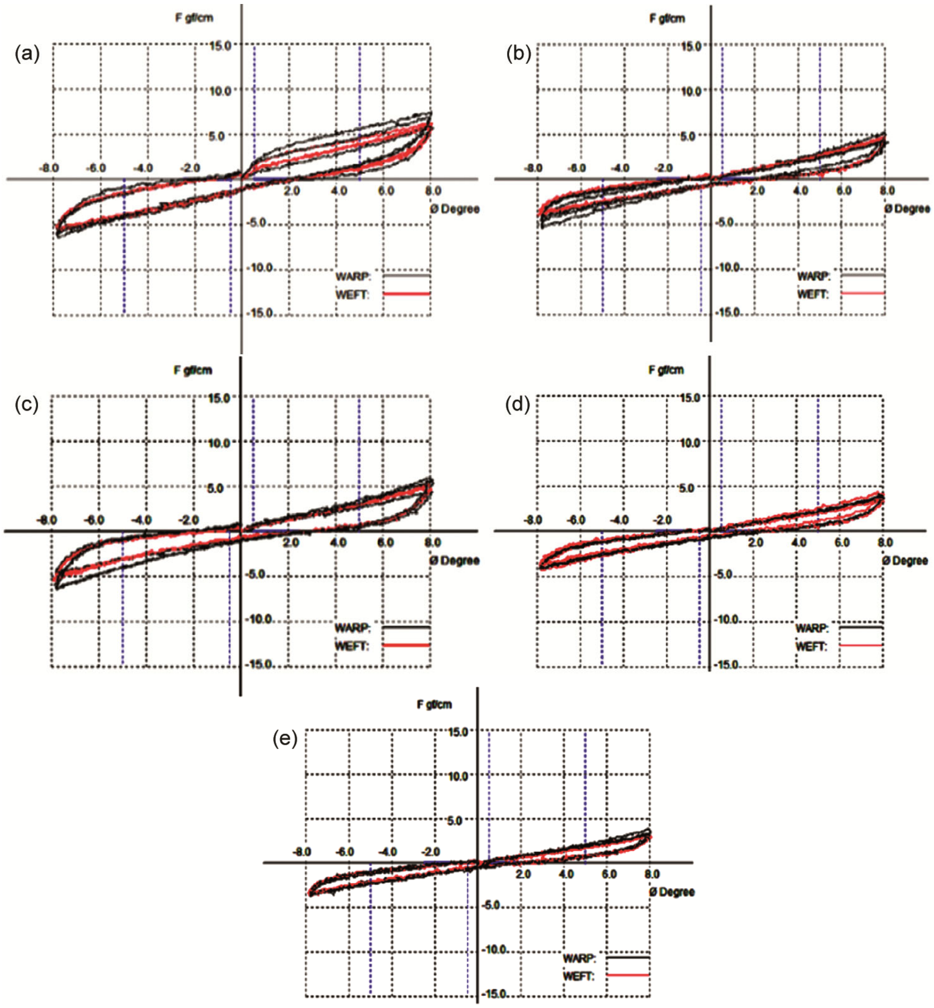


Fig. 7 — [(a) to (e)] Warp and Weft-wise shearing diagrams under low stress conditions as assessed by using KES-FB1 by shear tester

stress, respectively. Lower value indicates a higher ability to recover from shear stress, meaning less restriction for sliding against each other of constituent fibres. In the present work, both 2HG and 2HG5 values were reduced for all the treatment fabrics, indicating a reduction in recoverability from shear stress as compared to Sample S1. The reduction of 2HG and 2HG5 values was maximum for Sample S5 (-64.22 % and -59.01 % respectively), indicating less stiffness than other finishes imparted due to the incorporation of an amino silicone softener. Warp and weft directional shearing diagrams under low stress are shown in Fig. 7(a) to Fig. 7(e).

From the analysis of the shearing diagram (Fig. 7(a) to Fig. 7(e)), it is observed that there is a reduction of both 2HG and 2HG5 values for all the above said treatments done as compared to control fabric and the maximum reduction of 2HG and 2HG5 values is observed and confirmed for sample No S5

Table 5 — Compression attribute as assessed by KES-FB3A Compression tester

Sample No.		LC	Significant, at 95 %	WC, g/cm^2	RC, %
Sample S1	Avg.	0.300	----	0.094	46.84
Sample S2	Avg.	0.350	YES	0.166	44.23
Sample S3	Avg.	0.273	YES	0.251	33.72
Sample S4	Avg.	0.297	YES	0.186	44.53
Sample S5	Avg.	0.260	YES	0.210	40.22

*LC- Linearity of load-extension curve for compression test, WC- Compression energy and RC- Compressional resilience

3.6 Compression Attributes

Compressive attributes for control cotton fabric and different types of natural bio-finished cotton fabrics are shown in Table 5.

From the analysis of the data in Table 5, it was observed that the linearity of load-extension value (LC) under compression for Sample S2 was increased by 16.67 % relative to Sample S1, but Samples S3, S4, and S5 exhibited a reduction in LC value as compared to Sample S1. The LC value serves as an indicator for fabric compressibility, a higher LC value signifying the fluffiness and enhanced compressibility of the fabric specimen. Sample S2 demonstrated higher compressibility than other treated fabric specimens. LC values for Samples S3, S4 and S5 were reduced as compared to Sample S1, indicating a reduction in fluffiness or alteration in fabric thickness after application of those treatments/ finishes. This may be viewed as cross-linking of Citric acid (Sample S3) and surface deposition of nanoparticles (Sample S4 and Sample S5). Statistical analysis indicates that all changes in LC values, except for Sample S4, are statistically significant at the 95 % confidence limit.

Corresponding low-stress compression behaviour diagrams for control fabric and differently treated/ bio-mordanted, dyed and bio-finished cotton fabrics are shown in Fig. 8(a) to Fig. 8(e).

The compression energy (WC) value represents the energy required to compress a fabric specimen up to its maximum compression force. Sample S2 exhibited a 76.60 % increase in WC as compared to Sample S1, indicating more floppiness and a higher energy requirement for compression. This increase may be attributed to the deposition of dual bio-mordant on the fabric surface. Reduction in compressional resilience (RC) was observed, showing a diminished ability to recover after removal of compressive force relative to the control fabric mainly due to some minor crosslinking effect occurred.

The most significant reduction in RC was observed for Sample S3 (28.01 %), indicating a higher chance of permanent deformation under compression where possible crosslinking between cellulose and polyphenolic compounds/ gallic acid present in eucalyptus leaf extract was assumed to be much predominant in Sample S3. In contrast, Sample S4 showed the minimum reduction in RC (4.93 %).

3.7 Surface Attributes

Surface attributes for control, bio-mordanted, catechu dyed, and differently bio-finished cotton fabrics are shown in Table 6.

Data of Table 6 indicated an increase in both Coefficient of Friction (MIU) and Geometrical Roughness (SMD) for all the said bio-finished fabrics. This increase can be attributed to surface modifications resulting from the application of various natural and bio-based agents, including

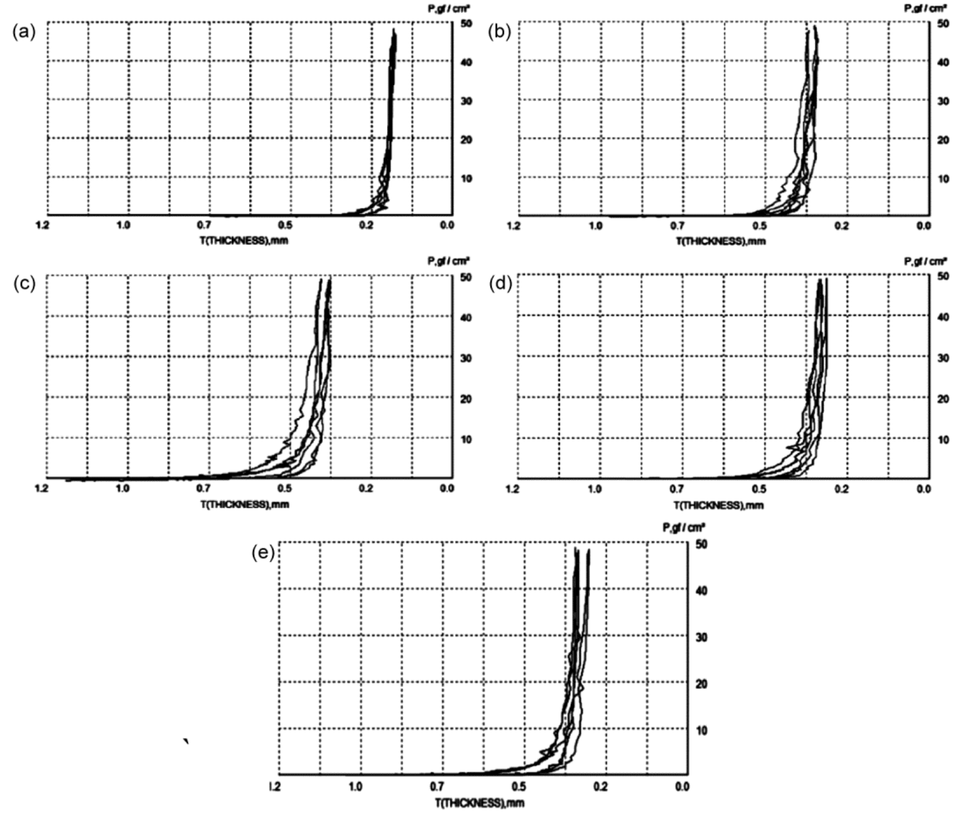


Fig. 8 — [(a) to (e)] Low-stress diagrams of compression behaviour as assessed by KES-FB3A compression tester

potassium alum (a natural metallic mordant), gall nut tannins, catechu dye powder and bio-finishing materials such as eucalyptus extract, chitosan (nano-chitosan) and nano-ZnO embedded in an amino silicone emulsion. Maximum increases in MIU (178.38 %) and SMD (76.61 %) were observed in Sample S4, likely due to the deposition of larger, coagulated nano-chitosan particles on the fabric surface. In contrast, Sample S5 exhibited the smallest increases in MIU and SMD, which may be attributed to the enhanced surface smoothness imparted by Hydroxy-Methyl Amino Silicone films and the uniform dispersion of fine nano-ZnO particles across the fabric surface.

Table 6 — Surface Frictional / Roughness properties as assessed by using KES-FB4 by Surface attribute tester

Sample No.		MIU	Significant at 95%	MMD	SMD, μm
Sample S1	Warp	0.067	----	0.0089	5.948
	Weft	0.155	----	0.0466	3.622
	Avg.	0.111	----	0.0277	4.785
Sample S2	Warp	0.186	YES	0.0167	6.883
	Weft	0.212	YES	0.066	8.960
	Avg.	0.199	----	0.0413	7.922
Sample S3	Warp	0.267	YES	0.0644	8.725
	Weft	0.268	YES	0.0256	7.317
	Avg.	0.267	----	0.0450	8.021
Sample S4	Warp	0.332	YES	0.0515	9.970
	Weft	0.287	YES	0.0237	6.932
	Avg.	0.309	----	0.0376	8.451
Sample S5	Warp	0.195	YES	0.0588	9.327
	Weft	0.146	YES	0.0142	6.350
	Avg.	0.171	----	0.0365	7.838

*MIU-Coefficient of friction, MMD-Deviation in Coefficient of friction, SMD-Geometrical roughness

The corresponding increases in MIU and SMD values for chitosan-treated fabrics developed a rougher surface texture, which may result in a reduction in tactile comfort or increased abrasive discomfort for the wearer. Statistical analysis confirmed that all observed differences in both the warp and weft directions are statistically significant at the 95% confidence level.

Corresponding surface characteristic diagrams under low stress conditions are shown in Fig. 9(a) to Fig. 9(e). These Fig. 9(a) to Fig. 9(e) displayed the trend of variation in Coefficient of friction (MIU) and geometrical roughness (SMD) for control fabric and differently treated/ bio-finished cotton fabrics. From these diagrams, it was observed that variations of both MIU and SMD were found to be increased in all the treatments, and Sample S4 (Fig. 9(d)), i.e., nano-chitosan treatment, shows the highest increase in surface roughness (higher MIU& SMD).

It is thought appropriate to examine FE-SEM images and high-resolution TEM images with scales to measure sizes of surface deposited particles of nano-chitosan and nano-ZnO on catechu-dyed cotton fabrics, determining the actual sizes of both adhered/fixed nano, micro and agglomerated particles deposited on the fibre surface.

So, for analysis of surface topology after both nano-chitosan and nano-ZnO bio-finish on catechu dyed cotton fabrics, HR-TEM and FE-SEM pictures of nano-chitosan and nano-ZnO bio-finished cotton fibre surface are shown in Fig. 10(a) to 10(d), which also correlates with the reasons for surface roughness/smoothness of the samples.

Fig. 10(a) shows the HR-TEM picture of agglomerated nano chitosan, and the same is

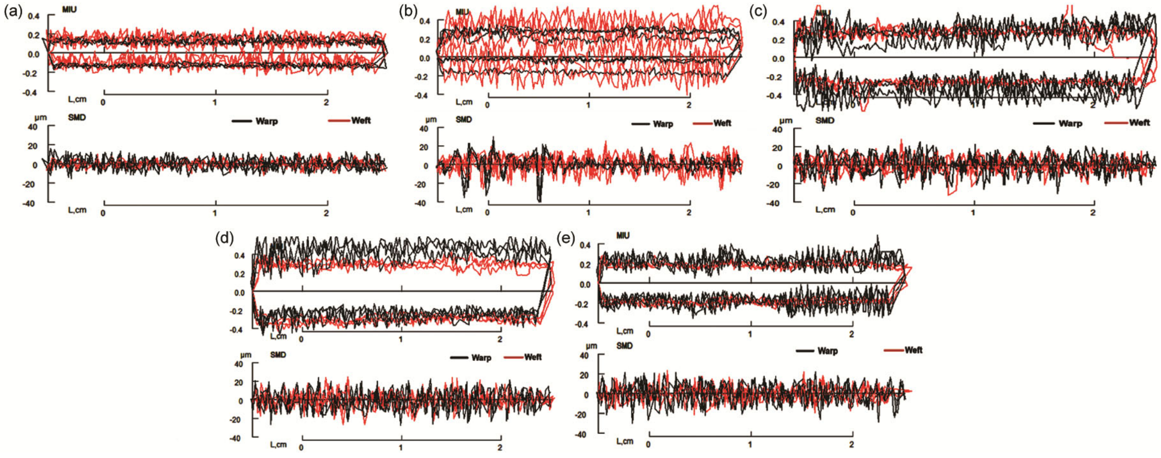
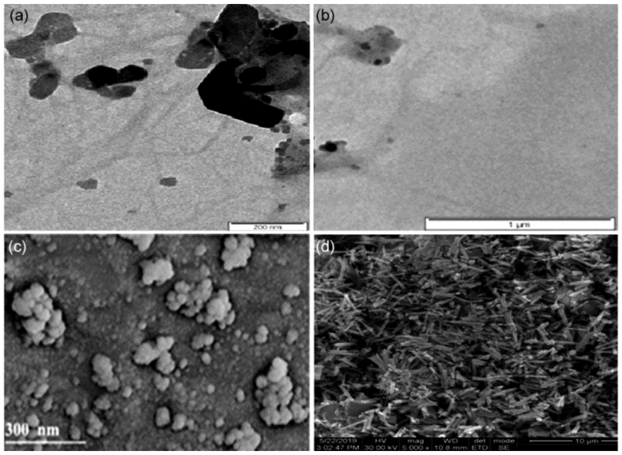


Fig. 9 — [(a) to (e)] Low-stress diagrams of Surface Frictional/ Roughness behaviour as assessed by using KES-FB4 by Surface attribute tester



S4: Nano-chitosan finished Cotton fabric and S5: Nano-ZnO +Amino silicone finished cotton fabric

Fig. 10 — (a) HR-TEM Picture of Sample S4 (nano-chitosan finished cotton, showing major agglomeration); (b) HR-TEM Picture of Sample S5 (nano-ZnO + amino-silicone finished cotton showing very minor agglomeration), (c) FE-SEM Picture of Sample S4 (for nano-chitosan finished cotton) showing very high agglomeration of nano-chitosan, and (d) FE-SEM Picture of Sample S5 with very little agglomeration, i.e mostly non-agglomerated deposit of nano-ZnO (showing crystals of nano-rods structure of ZnO)

confirmed from the FE-SEM picture in Fig.10(c), which shows the high agglomeration of nano-chitosan for the Sample S4 fabric surface. While for Nano-ZnO treated cotton fabric surface, Fig. 10(b) shows the HR-TEM picture of uniformly dispersed mostly as non-agglomerated physical deposit of nano ZnO crystals and the same is confirmed from FE-SEM picture in Fig.10(d) showing uniform deposition of nano-ZnO crystals array of nano rods of ZnO nano particles, which shows the rod-like nano-ZnO crystal deposited uniformly over the surface of Sample S5 fabric surface.

The particle sizes as revealed from scales of corresponding scales of FE-Sem and TEM images for nano-chitosan finish sample ranges from 200 plus nm to a maximum of sizes of 5000 plus nm (i.e., within the range of 200 –5200 nm with average around 1000 plus nm) with few small amounts in tiny nano form ranging 100-300 nm. So, the nano chitosan suspension, after homogenization with dispersing agent before application, has improved its retention of nano-sized chitosan particles, though most of them are agglomerated and distributed non-uniformly, increasing its MIU and SMD for nano-chitosan finished Sample (S4).

While particle sizes as revealed from scales of corresponding scales of FE-Sem and TEM images for nano-ZnO range from 0.83 nm to a maximum of sizes of 520 nm (i.e., within the range of 1–520 nm) with a uniform distribution of nano-ZnO rod-like tiny crystal particles embedded in amino silicone films across the fabric surface. This uniform distribution contributed to a relatively smooth fabric surface, as evidenced by only a minor increase in the coefficient of friction (MIU) and minimal impact on the fabric's bending properties for the Nano-ZnO finished sample(S5). This effect may be attributed to the presence of a flexible and smooth film formed by the hydroxy-methyl-amino silicone emulsion, which serves as a medium of application and acts as a physical coating of uniformly distributed nano-ZnO particles. The resulting film not only enhanced dimensional stability but also maintained a soft and comfortable fabric hand, making it suitable for draping women's dresses.

3.7.1 Chemical and Physical Interaction of Finishes applied on cotton:

The above results of key mechanical properties may be related to the chemical and physical interaction of the said finishes applied on cotton, such as Eucalyptus leaf extract containing ample polyphenolic compounds, which can form an ether link with the -CH_2OH group of cotton cellulose, with or without some crosslinking potential with adjacent cellulose chains.

Nano chitosan finishes with the amine group of chitosan, can be attached to minor aldehyde groups of bleached oxy-cotton, forming an aldimine link with or without potential crosslinking potential with adjacent -CHO in bleached oxy-celluloses.

While Nano-ZnO powder forms a physical coating under an amino silicone-based self-polymerizing film formed with some crosslinking potential between amino silicone and minor aldehyde groups of bleached oxy-cotton, forming an aldimine link.

3.8 Total Hand Value (THV) analysis

The Total Hand Value (THV-KN-303-Ds-Summer) of control fabric and catechu dyed and subsequent differently bio-finished cotton fabrics were carried out to judge the suitability of these bio-finished cotton fabrics for men's summer shirting using the corresponding data obtained for primary hand qualities (KN-202-DS) in terms of Koshi (Stiffness), Shari (Crispness), Fukumari (Fullness and Softness) and Hari (Anti-drape Stiffness) using Corresponding regression formulae to calculate THV (KN-303-Ds-SUMMER) values, as shown in Table 7A.

Based on the primary hand quality parameters Koshi (stiffness), Shari (crispness), Fukumari (fullness and softness), and Hari (anti-drape stiffness) and corresponding THV (Total Hand Value) data for KN-303-Ds-SUMMER standard KES formulae for men shirting, as shown in Table 7A, indicate that all Samples S1 to S5 were not found suitable for men's shirting fabrics intended for summer use.

Further to judge the suitability of these catechu-dyed differently bio-finished cotton fabrics for women's dress materials, preliminary hand qualities were calculated using the KN-203 LDY Summer and KN-203 LDY Winter KES standard formula to determine corresponding Total Hand Values (THV-KN-302-Summer and THV-KN-302-Winter) and are shown in Table 7B and 7C. These evaluations were based on experimental data obtained through the Kawabata Evaluation System, indicating primary hand attributes, which were then used to determine Koshi (stiffness), Numeri (smoothness), and Fukumari (fullness and softness) etc. as per standard KES formulae and the resultant THV values obtained for Samples S1, S3, S4 and S5 were presented in Table 7B for the summer–spring season and Table 7C for the winter–autumn season for women's dresses.

Analysis of the THV (KN-302-Summer) results of women's dress materials in summer revealed that the control cotton fabric (Sample S1) exhibited a Total Hand Value of only 2.49, with a little increase in THV values for Samples S2 and S3. However, some improvements in THV values were observed in Samples S4 and S5, with THV values rising to 3.76 and 4.47, respectively. These THV results thus indicated that Sample S5 (Nano ZnO-finished) demonstrated its suitability for women's dress materials for the summer–spring season, showing a better quality of Sample S5 than Sample S4 (Nano -Chitosan finished) for Women dress in Summer and other Samples S1 to S4 were not considered suitable for women dress in summer amongst all the samples studied.

THV results (as per KES standard KN-302-Winter for women dresses) shown in Table 7C, for all the catechu dyed and differently bio-finished fabric samples intended for women's dress materials for the winter-autumn season, revealed insightful positive trends regarding fabric handle quality. THV data in Table 7C

Table 7A — Total Hand Value (THV-KN-303-Ds-Summer) of control fabric and dyed bio-finished fabrics

Sample No.	Primary Hand Values (KN-202 -DS) for Cotton-Based Men's Shirting Fabrics				THV (KN-303-Ds-SUMMER)
	Koshi (Stiffness)	Shari (Crispness)	Fukurami (Fullness & softness)	HARI) Anti-drape stiffness)	
Sample S1	4.79	2.69	12.80	3.76	-0.15
Sample S2	4.13	2.30	14.12	2.69	-0.69
Sample S3	4.23	1.73	14.85	3.40	-1.19
Sample S4	3.28	1.18	14.75	1.66	-1.02
Sample S5	2.85	1.76	15.45	1.22	-1.18

S3: Eucalyptus leaf finished, S4: Nano-chitosan finished and S5: Nano-ZnO +Amino silicone finished

Table 7 B — Total Hand Value (THV-KN-302-Summer) for control, dyed and bio-finished cotton fabrics for summer women's dress materials

Sample No	Primary Hand Values (KN-203 LDY) for Summer for Cotton-Based Women's Dress			THV(KN-302-Summer)
	Koshi (Stiffness)	Numeri (Smoothness)	Fukurami (Fullness & softness)	
Sample-S1 SS1	4.65	8.60	5.70	2.49
Sample S2	5.45	5.29	8.70	2.57
Sample S3	4.79	4.69	8.29	2.70
Sample S4	4.79	4.29	8.70	3.76
Sample S5	5.85	6.84	8.74	4.47

S3: Eucalyptus leaf finished, S4: Nano-chitosan finished and S: Nano-ZnO +Amino silicone finished

Table 7 C — Total Hand Value (THV-KN-302-Winter) for control, dyed, and differently bio-finished cotton fabrics for winter women's dress materials.

Sample No	Primary Hand Values (KN-203 LDY-Winter) for Cotton-Based Women's Dress			THV (KN-302- Winter)
	Koshi (Stiffness)	Numeri (Smoothness)	Fukurami (Fullness & softness)	
Sample S1	4.79	4.29	8.70	3.76
Sample S2	5.70	5.97	8.60	4.08
Sample S3	5.46	5.78	8.86	4.05
Sample S4	5.12	6.06	8.89	4.08
Sample S5	5.06	6.85	9.41	4.53

*THV – Total Hand Value -KN-302-Winter, on a 1-5 scale, where 5 is best and 1 is poor.

indicated that the initial THV for Sample S1 was 3.76, and THV results were found to increase to 4.08 for Sample S2 (dual pre-mordanted and catechu dyed cotton), indicating an enhancement in the perceived hand characteristics. While. Post-treatment with Eucalyptus leaf extract (Sample S3) and nano chitosan finished (Sample S4) maintained the THV at a similar level within 4.05 to 4.08, with negligible variation, suggesting limited additional effect on overall hand value, but acceptable hand qualities for use in women's dresses in the winter-autumn season. In contrast, application of nano-ZnO finished sample combined with amino silicone emulsion (Sample S5) as a best finishing treatment showed the enhancement of THV results up to 4.53 on a five-point scale (1-5). This significant increase suggested a considerable improvement in the fabric's tactile qualities, making it well-suited for women's light-weight winter-autumn dress materials.

Thus, it may be concluded that Sample S5 had emerged as the most favorable/suitable, exhibiting the highest THV results for both summer and winter seasons for applications as women's dress materials. This superior performance was primarily attributed to enhanced "Fukurami" (fullness and softness) in combination with optimized stiffness, smoothness and overall tactile fullness, making fabric ideally suitable for the designated apparel category.

The combined effects of these preliminary hand qualities had been illustrated through the "Fukurami" (fullness & softness) as shown in Table 7C, showing Sample S5 having the highest Fukurami value, indicating superior tactile attributes desirable for winter apparel. Additionally, the THV (by KN-302-Winter) of the fabrics, subjected to various treatments including mordanting, dyeing with catechu dye and bio-finishing, consistently ranged between 4 and 5. This range was substantially higher than the initial THV of 3.76 recorded for the untreated control Sample S1, highlighting the potential of these differently bio-finished /post-treated cotton fabrics for women's dress materials suitable for autumn-winter wear, offering enhanced skin sensory tactical comfort.

To provide a summary of a comparative analysis of all the samples studied in the present work, by Spider Charts (also known as a snake diagram), a relevant comparative THV diagram of the control fabric and dual pre-mordanted and catechu-dyed fabrics and differently bio-finished cotton fabric samples was presented in Fig. 11(a) to 11(e). These visual representations of summative variations in fabric hand

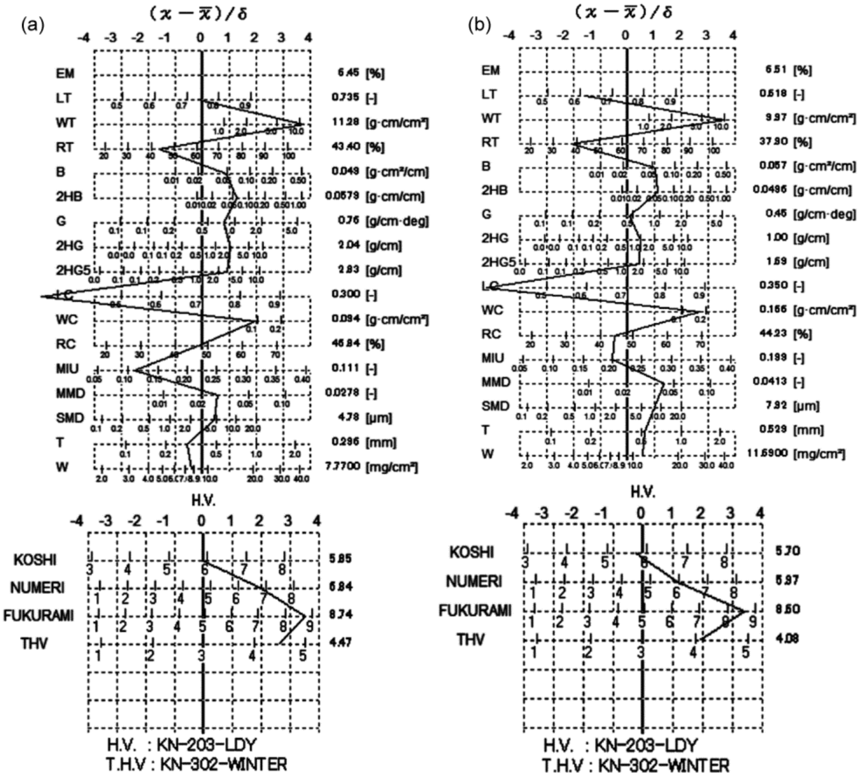


Fig. 11 — (a) Snake diagram for THV for Sample S1, (b) Snake diagram for THV for Sample S2, (c) Snake diagram for THV for Sample S3, (d) Snake diagram for THV for Sample S4, and (e) Snake diagram for THV for Sample S5 (*Contd.*)

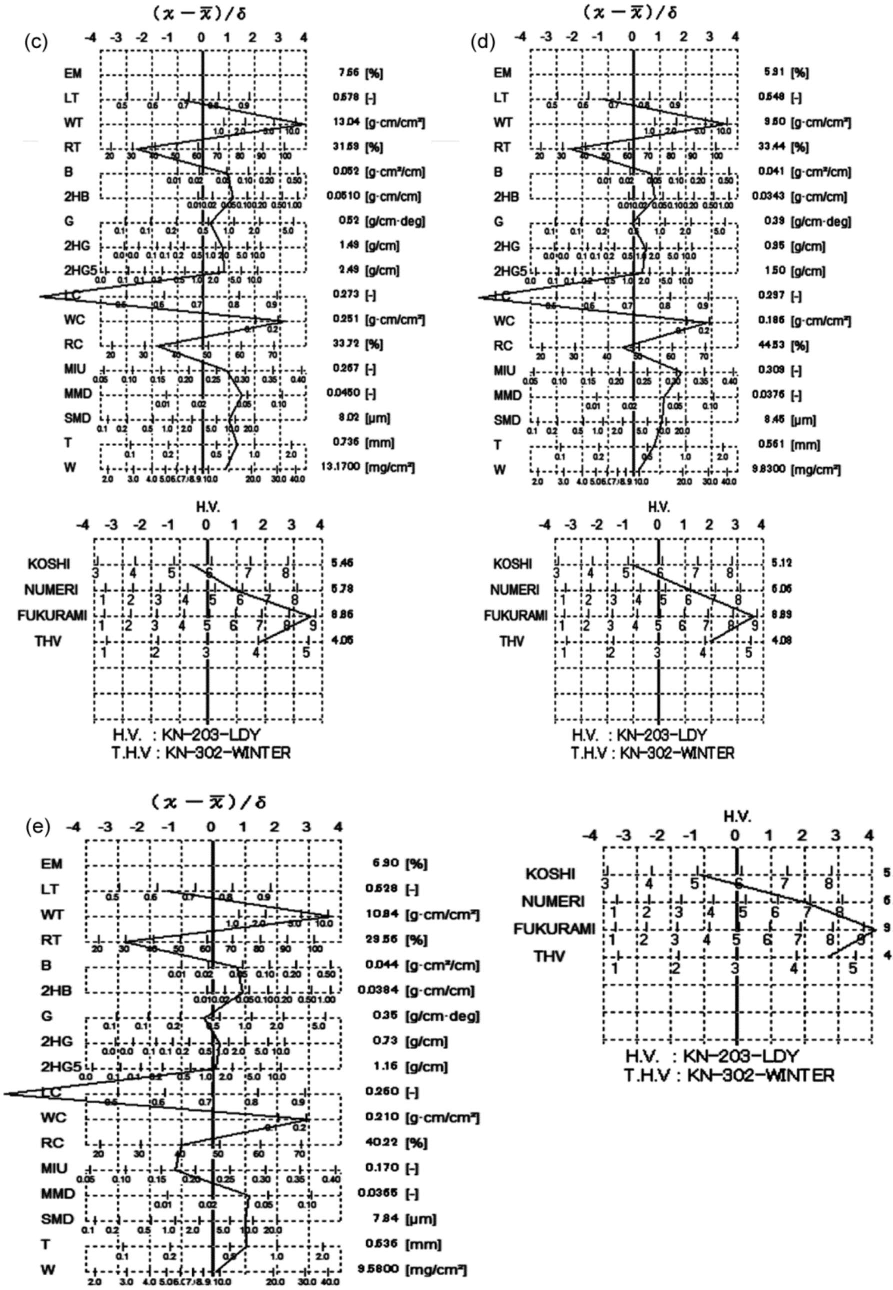


Fig. 11 — (a) Snake diagram for THV for Sample S1, (b) Snake diagram for THV for Sample S2, (c) Snake diagram for THV for Sample S3, (d) Snake diagram for THV for Sample S4, and (e) Snake diagram for THV for Sample S5

qualities for all the samples individually supported the interpretation of observed results showing varied improvements in tactile comfort performance by each sample, as shown below in Fig. 11(a) to 11(e).

Based on the KN-201-LDY evaluation of THV using the Kawabata Evaluation System for women's dress materials, a medium-weight cotton fabric with THV scores above 4.0 is considered suitable for high-quality sensory comfort for use in drapery women's apparel. The THV of the differently finished fabrics is primarily influenced by some key mechanical and surface properties under low stress conditions, particularly MIU (coefficient of friction), EM (extensibility), WT (weight) and LC (linear extensibility), etc.

In the Present study, further analysis of the THV (KN-303-Winter) through a spider chart (Fig. 11(a) to 11(e)) revealed that the primary distinctions between the differently treated cotton fabric samples and the control sample arise from variations in type of finishes. The spider chart effectively visualized these variations, allowing for a comparative assessment of the impact of each finishing treatment on fabric handle.

Analysis of the corresponding spider chart further revealed that control cotton fabric after K-alum + gallnut dual pre-bio-mordanting and subsequent catechu dyeing followed by three different bio-finishing treatments, showed a noticeable decrease in WC (compressional energy) and RC (compressional resilience) in the treated cotton fabrics as compared to the untreated control sample, (vide relevant data in Table 5). The reduction in WC indicated lower fabric compressibility, suggesting a stiffer structure, while the reduction in RC signified a diminished ability of the fabric to recover its original form after the removal of compressive force.

Conversely, an increase in MIU (coefficient of friction), MMD (mean deviation of the coefficient of friction) and SMD (geometrical surface roughness) was observed in the treated samples, which is maximum for Sample S4. These increases in MIU and SMD suggested a higher degree of surface roughness, likely resulting from the surface deposition of mordanting agents and relevant bio-finishing materials, whose effects were dependent on particle size and distribution. Additionally, the increase in fabric thickness and weight was confirmed by the incorporation of these mordants, dyes and bio-finishing materials, particularly in Samples S3, S4 and S5, thereby supporting the conclusion that surface and structural modifications significantly influenced the tactile and key mechanical behaviour of the treated fabrics under low stress conditions.

3.9 Analysis of UV protection and Antibacterial Performance

Results of UV protection and Antibacterial performance for the control fabric and these three differently bio-finished cotton fabrics are shown in Table 8.

Based on the findings of the analysis of Antibacterial performance and UV protection property, Samples S3 and S5 may also be considered for medical textile applications, too, due to their notable antimicrobial activity and ultraviolet (UV) protection criteria, the results of which are shown in Table 8.

From relevant data in Table 8, it was observed that the application of a dual bio-mordant treatment followed by Catechu dyeing (Sample S2) resulted in a substantive 2-3 times increase in the Ultraviolet Protection Factor (UPF value up to 10-15) but this treatment did not yield any notable reduction in the growth of gram-positive bacteria. In contrast, the application of Eucalyptus leaf extract (Sample S3) produced the highest observed enhancement in UV protection, with 9-10 times increase showing excellent UPF results up to 45-50.

Additionally, this eucalyptus leaf extract finishing treatment achieved bacterial reduction of approximately 99% for gram-positive bacteria and 98 % reduction in gram-negative bacteria, indicating almost highest antimicrobial performance too. These findings thus highlighted the effectiveness of specific eucalyptus leaf bio-finishing treatments, resulting in the highest level of enhancement in both UV protection and antimicrobial functionality of cotton fabrics, While for Sample S5, the application of Nano-ZnO finish in combination with silicone emulsion resulted in only a moderate 5 times increase in UPF up to 25, but demonstrated very good antibacterial performance (at par with sample S3) showing gram-positive bacterial reduction up to 99 % and reduction in gram-negative bacteria up to 98.8 %.

Thus, nano-Zno finish is proven to be very good in tactile comfort and antimicrobial criteria, but its UV protection performance is moderately good only (Not

Table 8 — Results of UV protection and Antibacterial properties of the selected samples

Fabric and Treatments	UV Protection Analysis (AATCC-183-2004/2010 method)			Bacterial reduction (AATCC-100-2012 method)	
	UV-A Trans, %	UV-B Trans, %	UV protection factor	Klebsiella Pneumoniae: AATCC 4352 (Gram-negative bacteria)	*Staphylococcus aureus*: AATCC 6538 (Gram +ve bacteria)
Sample S1	14.5	16.1	5	Nil Reduction	Nil Reduction
Sample S2	9.57	8.47	10-15	46.4%	Nil Reduction
Sample S3	2.08	1.81	45-50	98.02%	99.34%
Sample S4	4.15	3.38	25-30	56.34%	76.22%
Sample S5	4.25	3.60	25	98.80%	99.42%

S3: Eucalyptus leaf finished, S4 :Nano-chitosan finished and S5 : Nano-ZnO +Amino silicone finished

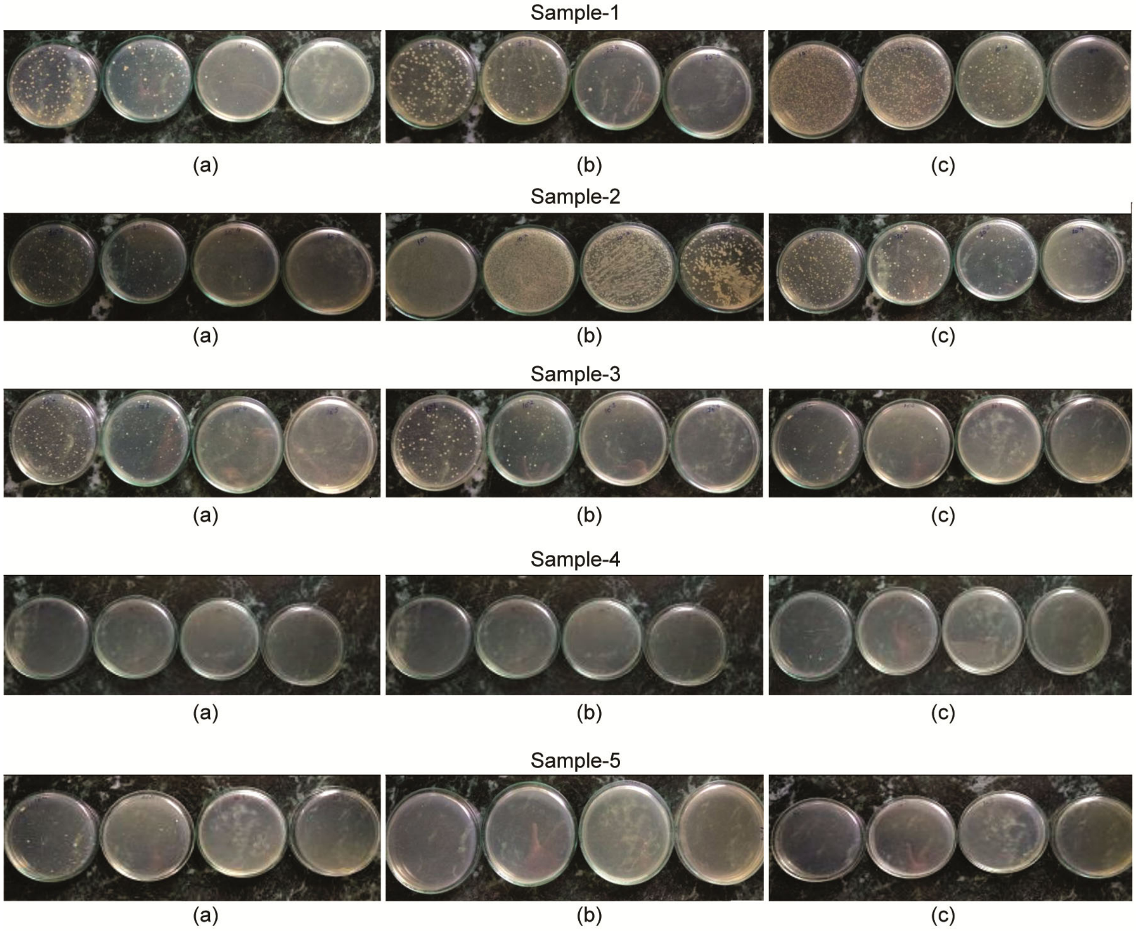


Fig. 12 — Pictures of Petri plates of corresponding S1 to S5 samples for Antibacterial Test as per AATCC-100-2012

excellent like eucalyptus finish). Corresponding antimicrobial test results are shown in Table 8, with corresponding pictures of Petri plates shown in Fig. 12.

The above results/characteristics thus reinforced the potential application of Samples S3 and S5, i.e., Eucalyptus leaf extract finished sample and also nano ZnO-based finished sample, in producing antimicrobial bio-functional medical textiles, particularly for surgical gowns of women doctors and women patients. But for Vitamin D-deficient women patients, for sunbathing in the winter season, eucalyptus finished fabrics are more suitable than nano-ZnO finished cotton fabrics, for its moderate range of UV protection results of sample S5.

While nano-chitosan finished cotton fabrics (Sample S4) exhibited higher surface friction, stiffness in bending behaviour, and late bending recovery, as well as much lesser antimicrobial performance than Samples S3 and S5. However, UV protection is moderately good (UPF value up to 25-30); this finish does not encourage its use in either summer or winter women's dress materials and nor for medical textiles.

However, overall THV results for women's dresses are equally good for this sample S3 as compared with the other two finishes (Sample S4 and Sample S5) studied in the present work.

3.10 Chemical and Physical Interaction of Different Bio-Finishes for Antimicrobial and UV Protection Properties

It is reported earlier in literature[27] that Epi-gallocatechin-3-gallate, having a polyphenol structure present in *Acacia catechu,* may crosslink with many proteins and attach to the peptidoglycan layer of cell walls of gram-positive bacteria, resulting in damage to its cell wall and causing deterioration of the crosslinking peptides, cytoplasmic lipids, and proteins. This is also mentioned in previous literature[27,38], that "the presence of saponins, tannins, and polyphenol colour components in catechu dye may have played a major role in imparting UV blocking property since they have free radical scavenging capability.

It is reported in earlier literature that 1,8-cineole (Eucalyptol) and polyphenolic flavanoids present in eucalyptus, fruit, bark and leaves have strong antimicrobial effects against many bacteria. Besides containing the known antioxidant eucalyptol as one of the constituents, alpha-pinene, beta-pinene etc., present in the extract of eucalyptus, are believed to have both antibacterial and antioxidant properties[39] too and hence, S3 sample shows superiority for both UV rays protection and prevention of bacterial growth (for both gram-positive and gram-negative bacteria vide results in Table 8).

Nano chitosan finish treatment on pre-mordanted and catechu-dyed cotton fabrics does not show good bacterial protection results (Table 8) with corresponding petri-plates shown in Fig. 12 for S1 to S5 samples, though chitosan is known to be a good antimicrobial agent. This may be due to the reduction of zeta potential (between +ve charged chitosan containing amine/ammonium groups in acidic media and -ve charged cell wall of bacteria), in this case by possibly combining +ve charged chitosan with -ve charged bacteria) and hence the antimicrobial action of chitosan is reduced. Also, nano-chitosan cannot render the desired UV protection due to the lower UV absorption criteria of chitosan, as observed from the UV-VIS spectral scan of chitosan solution (as shown in Fig. 2(b)).

Nano ZnO finish shows very good results in antimicrobial criteria, as also reported by earlier workers[26] which corroborates the present findings. This high antimicrobial effect of ZnO is believed to be due to the photobleaching action of ZnO, not allowing microbes to adhere /stick to the Nano-ZnO or ZnO-coated cellulose surface to be attacked by any bacterial species. Some improved UV protection of nano-Zno finished cotton within a limit can be explained by its UV-VIS spectral scan in Fig. 2(a), showing broader UV absorption peak at entire UV-Visible Zone without any specific zone of UV absorption in particular, not able to protect a particular harmful UV zone, after a certain limit of UV absorption by nano ZnO in general.

4 Conclusion

This study evaluated the Total Hand Value (THV) of cotton fabrics treated with dual bio-mordants gall nut and natural potassium alum (K-Alum) followed by catechu dyeing and then three types of bio-finishing treatments (eucalyptus leaf extract, nano-chitosan, and nano-ZnO combined with amino silicone emulsion) using the Kawabata Evaluation System (KES) under low-stress mechanical conditions.

The application of these treatments resulted in a noticeable increase in fabric thickness and weight per unit area, confirming the effective deposition of the bio-mordant, dye and finishing agents onto the cotton substrate.

Treated samples exhibited a general reduction in the linearity of load-extension (LT), tensile energy (WT) and tensile resilience (RT) along with an increase in tensile elongation (EMT). These changes indicated enhanced stretchability and reduced stiffness, although the fabrics demonstrated slower recovery, suggesting semi-permanent elasticity depending on the treatment type.

Decrease in bending rigidity (B) and bending hysteresis (2HB) was recorded in Samples S4 and S5, indicating improved flexibility and better recovery from bending deformation due to decrease in both static and kinetic friction, potentially resulting from the smoothening effect caused by the filling of intermolecular gaps within the fibres by nanoparticles, hence suggesting a softer and more pliable fabric structure.

All treated fabrics showed a decline in shear rigidity (G) as well as in 2HG and 2HG5 values, reflecting improved drapability. Sample S5 demonstrated the most pronounced enhancement, with a 53.95% reduction in G and substantial decreases in 2HG (64.22%) and 2HG5 (59.01%). These improvements were primarily attributed to the softening effect of amino silicone in the nano-ZnO finishing formulation.

Overall reduction in compressional resilience (RC) was observed across all samples, with Sample S3 showing the highest reduction (28.01%) after treatment with eucalyptus extract. This suggests enhanced fabric softness and fullness. The highest reduction for Sample S3 may be viewed as cross-linking of Citric acid.

An overall increase in both the coefficient of friction (MIU) and surface roughness (SMD) was noted, likely due to the surface deposition of finishing agents. While these changes may enhance functionality, they could reduce comfort during warm-weather wear due to increased skin friction.

Treated fabrics, especially those with eucalyptus extract and nano-ZnO, showed the highest improved ultraviolet (UV) protection and antibacterial activity, registering their potential suitability for medical textiles as antimicrobial and UV protective applications.

THV analysis indicated that these treated fabrics were unsuitable for men's summer shirting but showed partial suitability for women's summer dresses. Their optimal application was identified in women's winter-autumn apparel, where greater tactile and thermal comfort is preferred. Among all samples, Sample S5 (catechu-dyed and finished with nano-ZnO and amino silicone emulsion) achieved the highest THV score of 4.53 (on a 1–5 scale), making it the most promising for women's winter-autumn garments.

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