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RESEARCH

Fabrication of Hydrophobic Surface on Eri Silk/Wool Fabric Using Nano Silica Extracted from Rice Husk

Mamoni Probha Borah[1,2,3] · Binita Baishya Kalita[2] · Seiko Jose[1] · Sunita Baruah[2]

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Abstract
Silica nano particles were synthesized from rice husk by alkali treatment followed by calcination. The nano particles were characterized with the aid of Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and X-ray Diffraction Spectroscopy (XRD). The eri silk / wool union fabric was coated with the nano silica by hydrothermal method for achieving hydrophobic surface. The SEM and XRD results inferred that the synthesized silica nano particles are spherical and amorphous in nature. SEM, Energy Dispersive X-rays (EDX), and Atomic Absorption Spectroscopy (AAS) analysis qualitatively and quantitatively confirmed the existence of nano silica on the fabric. The nano silica treatment resulted in the enhancement of water contact angle upto 145°. The results are further confirmed by spray test and water absorbency analysis. The nano silica treated fabric possessed good fastness to dry cleaning and washing. The highest contact angle of 145° was observed with the treatment of union fabric with 7.5% nano silica. The AAS results showed 667 ppm of silica on the fabric. The physico-mechanical properties of the silica treated fabric differed marginally from the untreated fabric.

Keywords Contact angle · Hydrophobic textiles · Lotus effect · Nano silica · Union fabric · Surface modification

1 Introduction

Rice is one of the major crops in world. The production of paddy rice results in significant amounts of rice husk and straw. For the manufacture of very pure silicon, silicon nitride, silicon carbide, and magnesium silicide, rice husk is a good source of high-grade silica [7]. When rice husk is burned at temperatures between 550° and 650 °C, it produces rich, amorphous silica ash (SiO_2) [23]. Rice husk ash is essential in industrial applications due to its distinctive properties, which include high porosity, low density, high surface area, and low thermal conductivity [20].

Due to its enormous potential for a variety of end uses, nanotechnology is gaining more and more attention on a global scale as well as in the textile sector [16, 22]. The use of nanomaterials in textiles and the impact of nanotechnology on the textile industry have significantly enhanced fibre and processing technologies to meet social needs [4]. Given their large surface area and high surface energy, nanoparticles are used on textiles to enhance their functional characteristics [11, 15]. One of a solid surface's most crucial characteristics is its wettability, which has traditionally been described using the contact angle. Super hydroponic surfaces have a small sliding angle and a water contact angle of greater than 150 degrees. Due to their small size and high surface energy, nanoparticles are bound to the fabric surface by Vander Waal's forces, giving the fabric a respectable wash fastness [28]. Since water that has adhered to their hydrophobic/super hydrophobic surfaces can be easily removed, these textiles have the potential to be used in both industrial and household textiles.

Statement of Novelty In this study the agro-waste rice husk was used for extraction of nano-silica. The nano silica thus obtained was used to produce hydrophobic finishing on eri silk / wool union fabric. The authors report the first time use of hydrophobic finishing on silk / wool union fabric. This finished fabric could find potential applications in luxury textiles.

✉ Seiko Jose
seiko_jose2005@yahoo.co.in

[1] Textile Manufacture and Textile Chemistry Division, ICAR - Central Sheep and Wool Research Institute, Malpura, Avikanagar, India 304501

[2] Department of Textile and Apparel Designing, Assam Agricultural University, Jorhat, Assam, India 785013

[3] Department of Textile and Apparel Designing, Central Agricultural University, Tura, Meghalaya, India 785013

Silk and wool can be blended with other natural or synthetic fibres to create a wide range of products [1]. Blended fabrics are made to work around some of the inherent flaws of the fibres and ultimately produce the desired qualities. There are reports on the combination of wool and silk with other natural fibres for a variety of uses [9, 26]. Silk is widely used in luxury textiles since it is the queen of textile fibres. Silk fabrics with a hydrophobic finish may be preferred for use in technical textiles and high-end applications. Due to their uneven surface and the space between their fibres, textiles are more difficult to impart a hydrophobic finish to than flat surfaces like metals or glass. Meticulous research attempts have been reported on the hydrophobic and super hydrophobic finishing of textiles using polymers and nano particles [2, 21, 25, 29].

The present work investigates a cost effective method of preparation of hydrophobic surface on eri silk /wool union fabric by using agro-waste rice husk. To the best of the author's knowledge, silk/wool union fabrics do not have a hydrophobic finish. Nano silica is well known for various textiles and composite applications [8, 10, 14]. Here, the nano silica was extracted from rice husk and further coated on eri silk /wool union fabric. The coated fabrics were characterized for its water repellent properties in addition to physico-mechanical properties. The presence of silica on fabric was also established using various analytical techniques.

2 Materials and Methods

The rice husk for the extraction of nano silica was collected from the rice processing industry at Dhemaji, Assam, India. The plain weave fabric with eri silk yarn (2/140 Ne) in warp and merino wool yarn (1/56 Ne) in weft direction was constructed in handloom (80 reed count) having an areal density of 130 g/m^2. The laboratory grade chemicals such as acetic acid (assay-98%), sodium hydroxide (assay-98%), and sulfuric acid (assay-98%) were procured from Merk, India and used without purification.

2.1 Scouring of Union Fabric

The objective of scouring is to remove the alkali soluble impurities from the fabric. This also ensures uniform wettability throughout the fabric during the textile wet processing. For scouring pre-wetted eri silk/wool union fabric was immersed into an open bath containing 1.0% nonionic detergent (Ultravon JU) at 1:20 material to liquor ratio. The scouring was carried out for 30 min with gradual rise of temperature to 60 °C. The scoured fabric was washed in warm water (60 °C) and followed by rinsing in cold water, squeezed and dried at ambient temperature [19].

2.2 Synthesis of Nano Silica from Rice Husk

The synthesis of nano silica from an agro waste rice husk was carried out as per the protocol described elsewhere [12, 20]. In brief, 300 g rice husk was washed with water to remove the impurities. It was then dried in oven until free from moisture. The dried rice husk was carbonized in a glass beaker in a laboratory heating mantle. The char thus obtained was calcinated at 700 °C in a muffle furnace for 6 h to obtain silica. The resulting silica was added to 320 ml of 2.5 M sodium hydroxide solution and reflux for 3 h. The silica solution was filtered and the residue was rinsed with 80 ml of distilled water and the total stock solution was made up to 400 ml.

2.3 Characterization of Nano Silica

The particle size of the synthesized silica nanoparticles was estimated using Malvern Master sizer (3000 model) particle size analyzer by making a dispersion of 0.1% of nano silica in distilled water. The dispersion was sonicated for 30 min prior feeding to prevent agglomeration. The nano silica coating on the fabric surface was analyzed with aid of Scanning Electron Microscope (Nova Nano FESEM-450, the Netherland) with suitable magnification. The fibre and yarn samples were mounted on the metal disc using carbon tape and subjected to gold–palladium sputter coating under a vacuum of 8 to 10 mb pressure before SEM analysis. The EDX analysis was also carried out with the EDX attachment associated with the same instrument. The TEM analysis was carried out with JEOL JEM 2100 with an operating voltage of 200 kV. The FTIR analysis of the nano particles were conducted using Perkin Elmer- Spectrum 2 spectrometer at the weave length from 4000-500 cm^{-1}. The XRD analysis of the synthesized nano silica particles was performed with MiniFlex 600 Rigaku, Japan. The quantitative determination of amount of nano silica imparted during the chemical treatment on the eri silk/wool union fabric was estimated with Avanta GBC Atomic Absorption Spectrophotometer (GBC-Avanta, 2–0-2, GBC Scientific Equipments Pvt. Ltd., Victoria, Australia).

2.4 Application of Nano Silica on Silk/Wool Union Fabric

A dispersion having various concentrations (0.5%, 1.0%, 2.5%, 5.0%, and 7.5% on the weight of the fabric) of nano silica was prepared in a beaker by keeping the MLR 1:20. Dil. H_2SO_4 was added to it until the pH reaches 4.0. Non ionic detergent (1.0 gpl Ultravion JU) was added to it to enhance the stability of dispersion and the dispersion was subjected to high speed agitation (18,000 rpm) in a homogenizer for 10 min. The pre-wetted eri silk/wool union fabric was dipped in it and loaded in the closed

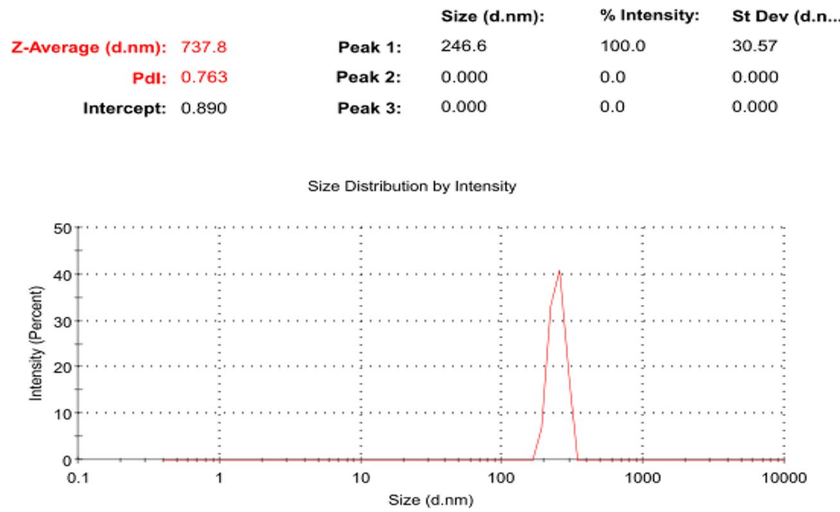

Fig. 1 Particle size analysis of nano silica synthesized from rice husk

Infrared heating machine (Texcare, India). The treatment was performed for 30 min at 90 °C. After the hydrothermal treatment, the union fabric was taken and wrested using a padding mangle by keeping 100% expression. The padded fabric was dried initially at 100℃ and further cured at 150℃ for 2 min. The samples were kept at 25℃ temperature and 65% RH before proceeded to further analysis.

2.5 Analysis of Water Repellency

The water contact angle of nano silica treated fabric was analyzed using Kruss DSA30E instrument (Kruss GmbH, Germany). Spray test was carried out to measured the hydrophobic characters according to FTTS- FA-011 method and graded as per the following scale. No sticking or wetting of upper surface (excellent-100), slight random sticking or wetting of upper surface (very good-90), wetting of upper surface at spray points (good-80), partial wetting of whole upper surface (70), complete wetting of whole upper surface (50) and complete wetting of the whole upper and lower surfaces (0). The water absorbency of the nano silica treated fabric was analyzed by keeping a single drop of water on the fabric sample with the aid of 200 μl micro pipette. The time taken for the complete absorption was noted and recorded. Ten readings were taken by keeping the water drop randomly in various places on the fabric and the average values were recorded [5].

2.6 Analysis of Fabric Properties

The bending length of the fabric samples was determined using a stiffness tester according to ASTM-D-1388 method. The Instron tensile tester (5965, UK) was used to analyzed the tensile and elongation properties of untreated and treated samples following the ASTM D-5035 test method [9]. Crease recovery of the samples was tested according to I.S method: 4681–1968 using the Shirley's crease tester. The

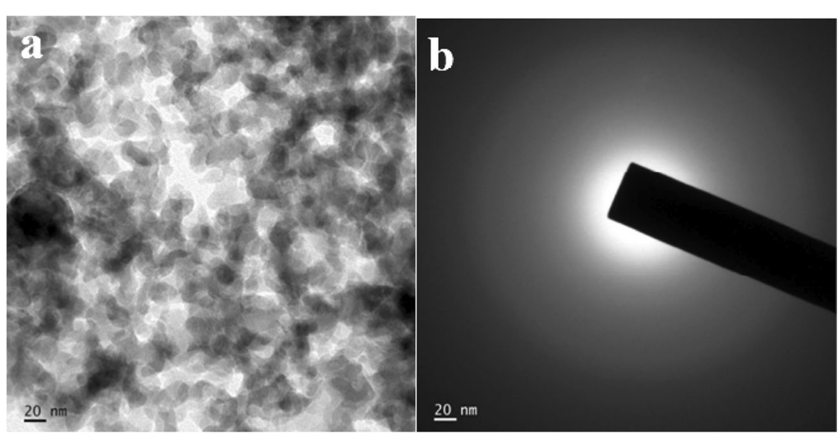

Fig. 2 **a** TEM images of nano silica. **b** Diffraction pattern

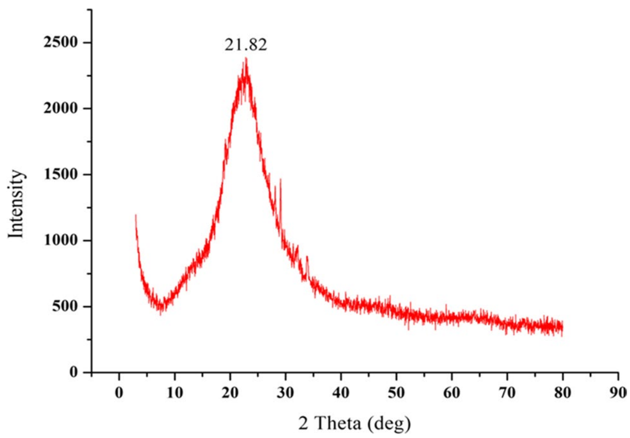

Fig. 3 XRD analysis of nano silica

whiteness, brightness and yellowness of untreated and nano silica-treated union fabric were measured using Premiere Spectra Scan 5100þ computer matching system at 10° observer. The dry cleaning fastness of the nano silica treated fabrics was evaluated according to AATCC 86–1976. The washing fastness of the samples was assessed as per ISO 105 E01. The durability of functional finished fabric sample was tested after different wash cycles (AATCC 124–1996).

3 Results and Discussion

3.1 Particle Size Analysis

The particle size analysis (Fig. 1) of silica nano particles showed an average diameter of 246 nm. The higher particle size may be attributed to the possible connatural properties of agglomeration of silica nano particles.

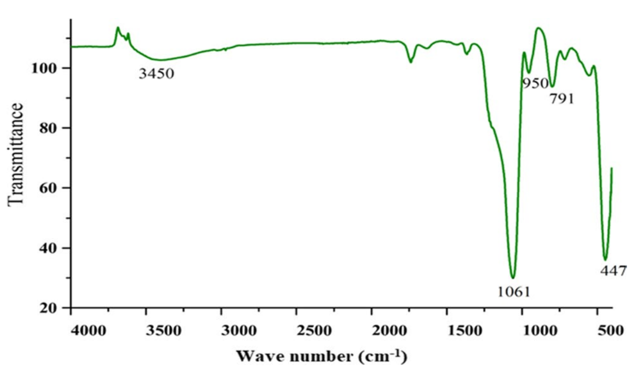

Fig. 4 FTIR spectra of nano silica synthesized from rice husk

3.2 TEM and XRD Analysis

The TEM images of the nano silica extracted from the rice husk is shown in the Fig. 2(a). The TEM image shows irregular geometry of the silica crystals. A network of particles appears to exist. The average crystal size calculated based on TEM analysis is 20–30 nm. From the diffraction patterns, the amorphous region of the synthesized silica is apparent (Fig. 2(b)).

The XRD analysis of nano silica is shown in the Fig. 3. The pattern shows the typical peaks at 2 theta 21.82°, 29.036°, and 64.3°. The broad peak at 21.8 degree indicates the amorphous region of silica [18]. The average crystal size of nano silica estimated using Scherrer's equation by measuring the full width at half maximum (FWHM) of XRD peaks was found to be 90 nm.

3.3 FTIR Analysis

The FTIR spectra of nano silica extracted from the rice husk is as shown in Fig. 4. The spectra depicts characteristic peaks of silica. A broad peak near 3450 cm^{-1} region corresponds to OH stretching. The asymmetric and symmetric stretching modes of Si–O-Si are visible at 1061 and 791and cm^{-1}. The Si–OH bond stretching appeared as a small, but sharp peak at 950 cm^{-1}. The steep peak at 450 cm^{-1} is attributed to the bending frequency of Si–O-Si [10, 23, 24].

3.4 Contact Angle Measurement

The water contact angle of the nano silica treated fabric was measured as described earlier and the results are depicted in Table 1. It is depicted from the contact angle measurement that the bare eri silk /wool union fabric exhibited contact angle of 97.6^O (Fig. 5a). The higher contact angle of the union fabric may be due to the presence of scale structure of the wool fabric, which prevents the easy penetration of water drops inside the fabric [3].

A steep increase in the contact angle was observed with the addition of 1.0% nano silica and it has been raised nearly to 139^O. Further, a gradual increase in contact angle was noted with an increase in percentage of silica and the highest contact angle of 145^O was achieved with 7.5% (Fig. 5b). Since there is no discernible hike in the contact angle was observed between the 5.0% and 7.5% treatment, further higher concentrations were not attempted. The water repellent properties of the nano silica on the eri silk / wool union fabric may be due to the formation of a three-dimensional cross-linked sheath around fibers [27]. The nano silica treated samples were showed marginal

Table 1 Water repellent properties of nano silica treated eri silk / wool union fabric

Fabric description	Contact angle (O)			Spray test		
	Treated	Dry cleaned	Washed	Treated	Dry cleaned	Washed
Control eri silk / wool union fabric	97.6	97.6	97.6	0	50	0
1.0% nano silica treatment	138.95	134.75	93.5	50/70	50/60	50
2.5% nano silica treatment	142.85	135.05	132.25	70/80	50/70	50
5.0% nano silica treatment	143.70	136.3	133.9	80	70/80	70
7.5% nano silica treatment	145.40	139.95	133.8	90	80	80

decrease in water contact angle after dry cleaning and washing (Table 1). However, all the samples retained a contact angle above 130 ^{O}.

3.5 Evaluation of Hydrophobicity by Spray Test

The spray test method rating is a direct indicative measure of the water repellent properties of a fabric. This approach is based on the rating scale from 0–100. The water repellency will be greater the higher the rating. The spray test analysis showed that the control fabric is well- absorbing water and rated as zero. The application of nano silica augmented the water repellency and the maximum rating was observed with 7.5% application (90). The basic water repellent qualities were remained intact despite the rating being slightly reduced by dry cleaning and washing (Table 1). As discussed in the contact angle measurement, the silica nanoparticles prevent the penetration of water molecules inside the fabric structure as well as in the space between the warp and weft yarn and thus, increase the water repellency of the union fabric [17].

3.6 Water Absorbency by Drop Test

The bare eri silk/wool union fabric quickly absorbed the water drop during the water absorbency test (Fig. 5a). The treatment of nano silica improved hydrophobic properties of the union fabric. The water droplets last longer on the fabric surface at higher concentrations of nano silica (5.0% and 7.5%) (Table 2 & Fig. 5b). The table shows a positive correlation between the concentration of nano silica and the retention of water droplets. This perhaps, due to the formation of a silica coated layer on the surface of the fabrics. It is to be noted that five out of ten drops remained even after 60 min, in the case of 7.5% nano silica treatment.

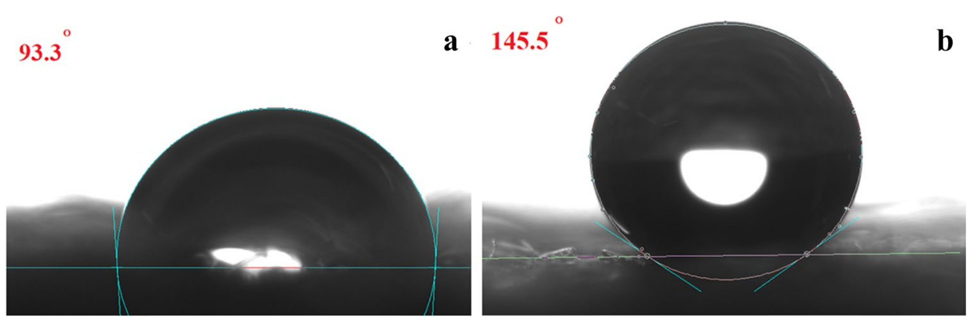

Fig. 5 **a** Water contact angle of (a) control. **b** 7.5% nano silica treated union fabric eri silk / wool union fabric

Table 2 Drop test for water repellency on eri silk /wool union fabric

Sample description	No. of drops	1 min	3 min	5 min	7 min	10 min	30 min	60 min
Control eri silk / wool union fabric	10	0	0	0	0	0	0	0
1.0% nano silica treatment	10	10	8	8	6	2	0	0
2.5% nano silica treatment	10	10	9	9	9	8	7	4
5.0% nano silica treatment	10	10	10	10	10	9	8	5
7.5% nano silica treatment	10	10	10	10	10	9	8	5

The results are arithmetic mean of five determination of each sample

Table 3 AAS analysis of union fabrics treated with nano silica

Sample description	Silica (ppm)
Control eri silk /wool union fabric	-
1.0% nano silica treatment	316.75
2.5% nano silica treatment	300.50
5 0.0% nano silica treatment	425.75
7.5% nano silica treatment	667.00

3.7 Atomic Absorption Spectroscopy

The quantitative estimation of nano silica treated fabric was performed with AAS and results are depicted in the Table 3. Between 1.0% and 2.5% of nano silica application, there was no discernible difference in the silica content, which is measured at about 300 ppm. However, 5.0 and 7.5% nano silica application resulted 425 and 677 ppm. The results show that, in comparison to the applied concentration, the nano silica concentration in the treated fabric has increased in a corresponding manner. Since AAS is a digestive method, it can be said that nano silica is having a good adhesion with the union fabric. The higher water contact angle of 7.5% nano silica treated fabric may be due to the higher silica content on the fabric.

3.8 SEM and EDX

The nano silica synthesized from rice husk formed small spherical shaped particles (Fig. 6(a)). From the images, it is found that majority of the synthesized particles are having similar size and shape. Figure 6(b) depicts the deposition of silica nano particles on the wool fibre surface. It is apparent from the figure that the surface of the fibre is thoroughly coated with silica nano particles of different sizes. Certain particles are found to be agglomerated, which may be due to the propensity of nanoparticles for agglomeration.

It was also noticed that there were formations of non-uniform small pits, micro-craters, etc., along with nano silica deposition on fabric surface layer. As a result of nano silica coating, the surface roughness of the fiber got enhanced, which helped in achieving hydrophobicity of union fabric [5, 13].

The control eri silk /wool union fabric showed the presence of elements such as, carbon (71.28%), oxygen

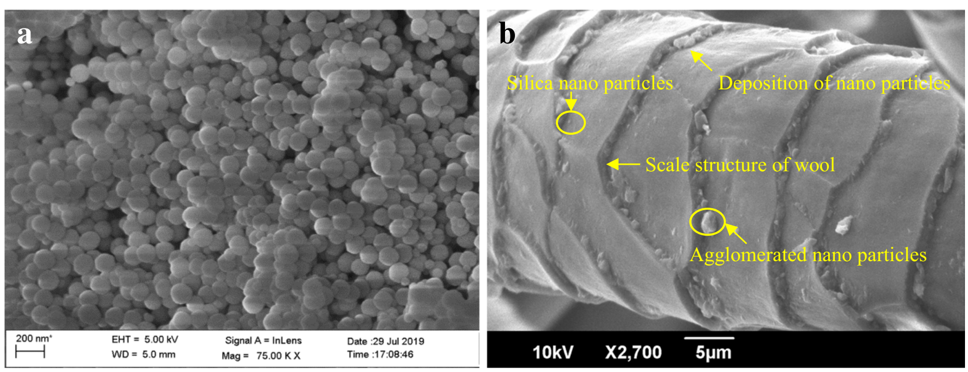

Fig. 6 SEM images of (**a**) nano silica (**b**) nano silica coated union fabric

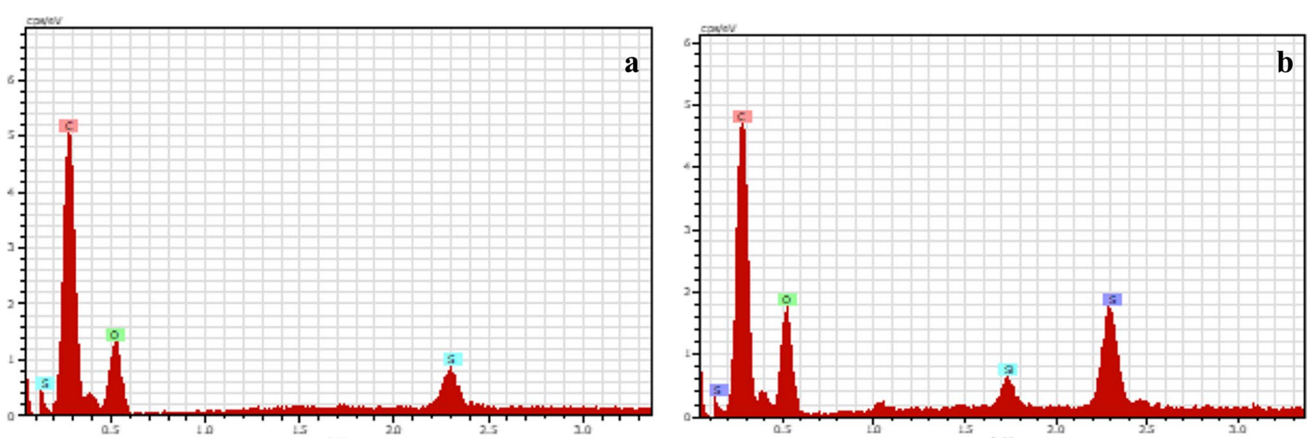

Fig. 7 **a** Control union fabric and **b** 7.5% Nano silica coated union fabric

Table 4 EDX of control union fabric and nano silica treated union fabrics

Fabric description	C [at. %]	O [at. %]	S [at. %]	Si [at. %]	Total
Control eri silk /wool union fabric	71.28	28.28	0.44	-	100
7.5% Nano silica treated union fabric	68.43	30.31	0.93	0.32	100

(28.28%), and sulphur (0.44%) (Fig. 7(a)). Additionally, EDX detected the existence of Si in the coated fabric (7.5% treated) as shown in the Fig. 7(b). The presence of sulphur is detected probably due to the presence of cystine linkages in the wool fibre. The coated fabric detected 0.32% silicon. Since nano SiO_2 contains oxygen, the oxygen content was also increased to 30.31% (Table 4).

3.9 Physico-Mechanical Properties of the Fabric

The surface appearance properties of the nano silica coated eri silk /wool union fabric are shown in the Table 5. Since the fabric was scoured, but not bleached, the basic whiteness index of the control fabric was found to be 31 only. The application of nano silica particles at different concentration didn't alter the basic whiteness, yellowness and brightness properties and it remains almost intact.

The mechanical properties of the fabric were assessed as described earlier. The control union fabric recorded a bending length of 1.08 cm in the warp and 1.24 cm in the weft direction. After nano silica coating, the bending length registered slight increase in both warp and weft direction with the increase in the concentration of nano silica. This may be due to the stiffness got imparted on the union fabric during the treatment. The silica particle may also cover the surface of the fibres and stiffen them by blocking the pores between them. Crease recovery refers to the ability of the fabric to return to its original shape after removing the folding deformations [6]. The crease recovery angle was also found to registered marginal difference after coating of nano silica. Since eri silk yarn was used in the warp direction, the tensile strength of the control fabric was found to be significantly higher than the weft direction. There is a steep reduction in the tensile strength (warp direction) of the fabric was observed after the treatment with 1.0% nano silica. However, further enhancement of silica concentration didn't mark any notable reduction in the tensile strength. The elongation of the fabric remained almost intact after nano silica treatment.

4 Conclusion

Herein, spherical-shaped nano-silica particles were synthesized from rice husk and applied on eri silk- wool union fabric. The main objective of the study was to enhance the hydrophobicity of the union fabric and the utilization of rice husk for the development of high end applications. Among various concentrations of nano-silica applied on the fabric, the 7.5% application exhibits the highest water contact angle of 145°. The spray test and an analysis of a single drop of water provided additional support for the contact angle measurements. The nano-silica treatment resulted in a marginal reduction in the tensile strength of the fabric, however; most of the physico-mechanical properties were found intact. The dry cleaning and washing slightly reduced the hydrophobicity of the coated fabrics meantime the coated fabric retained the basic hydrophobic character. The presence of nano-silica on the fabric was confirmed by SEM, EDX, and AAS analysis. The application of nano-silica up to 7.5% couldn't produce a super hydrophobic surface, however; it reached near to it.

Table 5 Surface appearance and physico-mechanical properties of nano silica treated union fabrics

Sample Description	Surface Appearance			Bending Length (cm)		Crease recovery (°)		Tensile Strength (MPa)	
	Whiteness Index	Yellowness Index	Brightness Index	Warp	Weft	Warp	Weft	Warp	Weft
Control eri silk /wool union fabric	31.01	24.13	55.21	1.08 *(3.02)*	1.23 *(6.95)*	99.85 *(0.15)*	98.00 *(0.08)*	272.00 *(6.21)*	92.03 *(6.02)*
1.0% nano silica treatment	30.83	23.89	56.42	1.10 *(0.54)*	1.26 *(9.05)*	99.40 *(0.35)*	101.80 *(0.88)*	223.61 *(31.92)*	94.26 *(4.06)*
2.5% nano silica treatment	30.17	24.07	55.65	1.14 *(5.05)*	1.30 *(5.27)*	101.60 *(0.26)*	101.40 (0.35)	221.44 *(5.90)*	94.11 *(1.27)*
5 0.0% nano silica treatment	31.23	24.04	56.45	1.18 (2.78)	1.35 (5.64)	107.25 (0.35)	102.0 (0.29)	218.00 (0.77)	95.00 (3.16)
7.5% nano silica treatment	29.07	24.90	55.00	1.31 *(13.60)*	1.37 *(4.72)*	102.75 (0.17)	101.00 (0.64)	216.76 *(2.08)*	95.22 *(2.03)*

The data in the table represent the average of five tests, and values in the parenthesis indicate CV%

Thus, it can be concluded that rice husk, which is an agro residue can be used for the preparation of nano-silica and the resultant nano-silica can produce a good hydrophobic finishing on textiles with reasonable washing fastness.

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Author Contributions Mamoni Probha Borah - Preparation of samples, testing, manuscript preparation Binita Baishya Kalita - Technical guidance Seiko Jose - Design of experiment, manuscript correction, testing Sunita Baruah - Testing and manuscript correction.

Data Availability Not applicable.

Code Availability Not applicable.

Declarations

Ethical Approval Ethics committee approval is not required for reported work.

Consent to Participate All authors are hereby declare their consent for the participation

Consent for Publication All authors are hereby declare their consent for the publication

Competing Interests The authors declare no competing interests.

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