SIMULTANEOUS DYEING AND FINISHING OF COTTON FABRICS USING RED OCHER EARTH PIGMENT

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A novel approach to the pad-dry cure method was employed to prepare concurrently dyed and finished cotton samples. The innovative approach integrates a layer-by-layer pigment dyeing procedure for obtaining varying shades of Red Ocher (RO) and functionalities on dyed cotton. Following the dyeing process, advanced characterization techniques, such as Fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA), were employed to confirm the presence of RO and its effect on the fabric. The findings of this unique study indicated exceptional UV protection factors, laundering, and light fastness of dyed cotton. Additionally, an observable trend emerged, indicating an increase in thermal resistance with an augmented number of padded layers, while conversely, a decrease in thermal conductivity was observed. These insightful and novel findings offer exciting opportunities for broader thermal insulation cotton textile applications distinguished by outstanding UV protection and color retention.

Keywords: Red Ocher, cotton, dyeing and finishing, UV protection, thermal properties

INTRODUCTION

The demand for multifunctional textiles has led to significant advancements in fabric treatment processes, aiming to improve aesthetic and functional properties. Among these processes, the pad-dry-cure method stands out for its ability to simultaneously dye and finish fabrics, offering a simplified and efficient approach to textile processing on an industrial scale. Moreover, various research studies show that applying natural dyes on textiles could introduce various functional properties, such as antibacterial, antioxidant, anti-inflammatory, UV protection, and thermal regulation.[1–4] In this context, the unique properties of the Red Ocher, derived from a specific type of soil, are of particular interest. This dye can offer unique properties that can enhance the functionality and aesthetics of textiles.[5] This research, focusing on the simultaneous dyeing and finishing of cotton fabrics using the pad-dry-cure method, specifically employing Red Ocher dye, presents a novel and intriguing approach to fabric treatment.

Red Ocher, a red ochre, is a naturally occurring earth pigment used for centuries due to its rich, vibrant color and eco-friendly nature. Its chemical composition, primarily iron oxide (Fe_2O_3), gives it its distinctive red hue.[6,7] It may also contain varying amounts of silica, clay, and other minerals, contributing to its distinct chemical and physical properties.[8–10] In addition to its aesthetic appeal, RO offers functional properties that make it an excellent candidate for textiles requiring hygienic and UV-protective properties. Its iron oxide composition provides inherent antimicrobial properties, and its non-toxic nature and biodegradability enhance its application as a sustainable dye.[11,12] Despite its potential, its use in textiles remains underexplored, particularly in contemporary dyeing methods. The unique properties of RO, including UV protection and thermal regulation, present significant opportunities for innovative applications in the textile industry. This reiterates the potential of RO, instilling a sense of hope and optimism regarding the future of textile dyeing and finishing.

Cotton is a natural fiber known for its comfort, softness, and breathability, making it a prime choice in the textile industry.[13,14] Its cellulose-rich structure contains numerous hydroxyl groups, readily forming -H bonds with dye molecules, enhancing its affinity for natural dyes. This affinity is further expanded by the fiber's porous nature, allowing for intense dye penetration.[15] Recent research on cotton dyeing and finishing with natural dyes has highlighted various sustainable methods and their benefits. For instance, a study explored *Rosa canina* extracts for dyeing and functional finishing of cotton fabrics.[16] Another study focused on using pomegranate peel extract for dyeing cotton fabrics, highlighting the challenges of natural dye affinity to cotton.[17] Jin *et al*. utilized biomass lotus seedpod waste to extract natural dye and, together with the bio-mordant chitosan, evaluated cotton fabric's dyeing performance and functionality under various conditions.[18] Werede *et al*. investigated the use of Gunda Gundo orange (*Citrus sinensis*) peels, in order to valorize these peels for dye extraction and application on cotton fabric, achieving a maximum dye yield of 40.11% under optimized conditions and demonstrating improved dyeing performance and fabric properties.[19]

This research investigates cotton fabrics' concurrent dyeing and finishing using the pad-dry-cure method, specifically employing RO. This research highlights the practical benefits of using RO in the pad-dry-cure process. It underscores the potential transformative impact of this method in developing sustainable and multifunctional textiles for various industrial applications. The implications of this study could stimulate a new path of sustainable and innovative textile production.

EXPERIMENTAL

Materials

In this research, the laboratory chemicals employed were sourced from Merck Specialties Private Limited, India. The thickener (synthetic polyacrylic acid-based thickener, solid content: 11.06%), binder (acrylic, solid content: 63.7%), and fixer (solid content: 40.76%) were utilized to apply RO on cotton fabric. An ammonia solution at 25% concentration was also employed for specific experimental conditions. Red Ocher was procured from a local market, and the fabric substrate used in the experiments was cotton, sourced from Vardhman Fabrics, India.

Methods

Application of RO on cotton

In this study, the RO was dispersed in water using a thickener and ammonia, which increases the solution's viscosity. This viscosity plays a crucial role in controlling the RO dispersion, ensuring its uniform distribution on the cotton fabric substrate. Since RO exhibits limited affinity for cotton fabric, a binder was introduced to improve color fastness and durability.

A 300 mL dye dispersion was prepared, comprising thickener (0.5%), binder (30 gpl), fixer (0.5%), ammonia (0.5%), and RO (20 gpl). The dispersion was stirred for 2 h at room temperature using a mechanical stirrer at 650 rpm.

Table 1
Add-on percentage of RO-treated cotton samples

Sample No.	Number of layers applied	Add-on (%)
1	1 Layer	1.73
2	2 Layers	5.07
3	3 Layers	7.62
4	5 Layers	11.63

The cotton fabric was padded using a two-bowl padding mangle (RBE Electronics, India) at 90% expression. The padded fabric was then dried at 100 °C for 5 min. Four samples were prepared with 1, 2, 3, and 5 layers of padding, respectively. Each fabric was cured at 170 °C for 1 min, after the drying of the last layer of RO dispersion, to ensure the enhanced durability of the dyed fabric. The add-on percentage was calculated, as shown in Table 1.

As shown in Table 1, the add-on on Sample 1 was 1.73%, representing the additional weight gained by the sample after the application of one layer. As the number of layers increases (Sample 2, Sample 3, Sample 4), the add-on percentages increase, reflecting the application of a higher quantity of chemicals used in the padding operation. These values are crucial for understanding the add-on required to achieve the desired depth of shade and extent of functional properties.

Characterization and testing

Fourier transform infrared (ATR-FTIR) spectroscopy was employed to analyze the functional groups present in RO-dyed fabric, operating in transmittance (%) mode, using a Nicolet iS50 spectrometer from Thermo Fisher Scientific Inc., USA. Additionally, the weight loss (%) of the RO-dyed cotton fabrics was assessed using thermogravimetric analysis (TGA 4000, Perkin Elmer Inc., USA) by heating the samples in a nitrogen atmosphere from 50 °C to 800 °C at a rate of 20 °C per minute. The reflectance (%) of the RO-dyed cotton was measured using a Gretag Macbeth Color-Eye 7000A spectrophotometer. The color strength

(K/S value) related to the fabric's reflectance and absorption properties was calculated using the Kubelka-Munk equation.[20]

The RO-dyed samples' crease recovery angle (CRA) was measured using the AATCC TM66-2017.[21] Fabric samples, sized 40×15 mm, were evaluated using a Shirley Crease Recovery Tester. The RO-dyed cotton samples' laundering fastness was analyzed by following the AATCC TM 61-2007.[22] The test apparatus included an accelerated laundering machine with rotating closed canisters in a thermostatically controlled water bath. The RO-dyed cotton samples' light fastness was assessed through the AATCC TM 16-2004.[23] The RO-treated samples' UV protection factor (UPF) was analyzed using the Australia/New Zealand (AS/NZ) 4399:1996 standard test method. Spectral transmittance of the fabric in the UV range, specifically between 290 and 400 nm, was measured using a spectrophotometer (Labsphere Transmittance Analyzer).[24]

Regarding thermal conductivity and resistance, testing was conducted using a KES-F7 Thermo Labo Tester following ISO 5085-1:1989 standards. Samples were prepared in specific sizes: 20x20 cm for resistance and 10x10 cm for conductivity. Test conditions were maintained at a relative humidity of 65% ± 2% and a temperature of 20 °C ± 2 °C, ensuring consistent and reliable measurements of the fabric's thermal properties. These tests are essential for assessing how effectively the fabric regulates heat transfer, which is crucial for applications ranging from apparel to technical textiles.[25]

RESULTS AND DISCUSSION

Color strength

Figure 1 and Table 2 present data illustrating cotton fabric's colorimetric and dyeing characteristics subjected to varying RO-padded layers. L*, a*, and b* values represent the color coordinates, where L* indicates lightness (higher values indicating lighter shades), a* denotes the green-red axis (positive values for red and negative for green), and b* signifies the blue-yellow axis (positive values for yellow and negative for blue). The L* value decreased with the increase in RO layers, indicating darker coloration due to the application of a higher quantity of RO. The value of a* increased with more dye layers, suggesting a shift towards red hues as the dye concentration on the fabric intensifies. Similarly, the b* value increased with additional layers, indicating a trend toward yellow hues.[26]

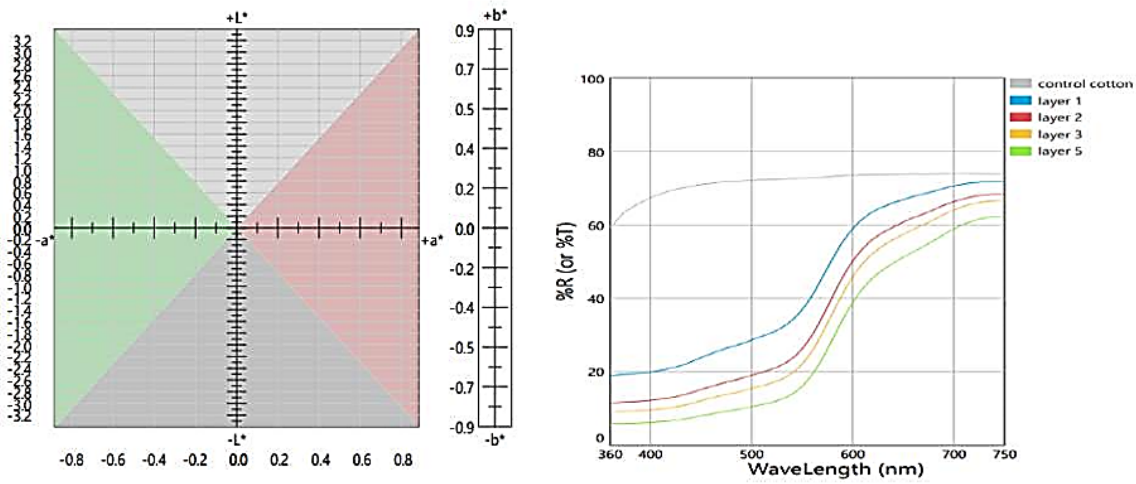


Figure 1: L*, a*, b*, and K/S values of RO-treated cotton fabric samples with different layers

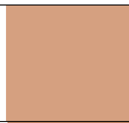
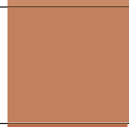
The K/S value represents the ratio of the absorption coefficient to the scattering coefficient, indicating color strength. It significantly increases as the number of RO layers rises, from 0.079 in the control sample to 7.122 in the 5-layer sample. This increase demonstrates enhanced dye application, fixation, and distribution within the fabric structure, resulting in deeper and more saturated colors. The observed trends in L*, a*, b*, and K/S values confirmed that increasing the number of RO layers improved the depth of shade and enhanced color fixation on dyed cotton, resulting in darker fabric. This multilayer dyeing approach minimizes surface irregularities and optimizes the RO-treated cotton's overall color quality and strength.[27,28]

Crease recovery angle (CRA)

Table 2 illustrates the crease recovery angle (CRA) values for cotton fabric samples subjected to different numbers of RO padding layers. CRA measures the fabric's ability to recover from creasing, with higher angles indicating better crease resistance and fabric resilience. The fabric without treatment exhibited a CRA of 137°, indicating its limited ability to recover from creasing. The CRA value decreased with the increasing number of RO padding layers.

The decrease in CRA may be attributed to several factors influencing fabric crease recovery. The additional layers of RO and binder can increase the stiffness of the fabric, making it harder for the fabric to recover its original shape after creasing.[29] The weight of the added RO and chemicals may change the fabric's internal structure due to the presence of RO dye on the surface, the penetration of RO in the inter-fibre and inter-yarn gaps, which can act as anchors for holding the binder film, contributing to reduced fabric resilience. Multiple layers may restrict the fabric's ability to flex and recover from creasing. However, the stiffness of the treated cotton fabric was not significant, and the prepared product is suitable for apparel and technical textiles.

Table 2
Color values and fastness ratings of RO-treated cotton fabric samples

Sample	L*	a*	b*	K/S value	CRA (°)	Laundering fastness	Light fastness	Image
Control	88.34	0.07	1.57	0.079	137	--	--	--
No dye	88.37	0.02	2.76	0.090	--	--	--	--
1 Layer	70.46	17.28	24.63	1.610	135	4-5	4-5	
2 Layers	63.08	22.68	29.2	3.149	132	4	4-5	
3 Layers	59.63	24.85	30.94	4.251	128	4-5	5	
5 Layers	53.72	28.13	33.56	7.122	115	4-5	5	

Washing fastness

Table 2 displays the washing fastness ratings on a 5-point scale for cotton fabric samples treated with different numbers of RO padding layers. Washing fastness refers to the fabric's ability to retain its color and appearance after repeated washing cycles, indicating the durability of the color on dyed textiles. The ratings indicate "good" to "excellent" laundering fastness as the number of RO padding layers increases. This suggests that the fabric retained its color well after washing, with minimal fading. The laundering fastness rating depends on RO fixation and distribution. The role of the acrylic binder is significant in holding RO on cotton. This prevents the removal of RO during washing treatments. As RO had no affinity for cotton fabric, the tinting on the white fabric was negligible. The chemicals used in the dyeing process, such as fixers and binders, can contribute to better attachment and stability of the RO on the fabric, improving laundering fastness.

Light fastness

Table 2 presents the light fastness ratings on a 5-point scale for cotton fabric samples treated with different numbers of RO padding layers. Light fastness refers to the fabric's ability to retain its color when exposed to light, indicating the durability of the RO against fading. The samples with 1 layer and 2 layers showed ratings of 4-5, indicating very good to excellent light fastness. This suggests that the fabric retained its color well when exposed to light, with minimal to no fading observed. The samples with 3 and 5 layers received a rating of 5, indicating excellent light fastness. Fabrics treated with this number of RO layers showed no or minimal color change even after prolonged exposure to light. This fixation of RO to the fabric fibers was facilitated by fixers and binders, which enhance the fabric's resistance to fading when exposed to light. The higher ratings observed with increased RO layers indicate improved light fastness due to the presence of a higher quantity of RO on the fabric. Generally, pigments possess good light fastness, and since Red Ocher is a water-insoluble inorganic pigment, an excellent rating of light fastness was achieved.

UV protection factor

Table 3 presents the UV protection factor (UPF) values and ratings for cotton fabric samples treated with different numbers of RO padding layers. UPF measures the effectiveness of a fabric in blocking ultraviolet (UV) radiation from the sun, specifically UV-A and UV-B rays, which are known to cause skin damage and aging.

Table 3
UV protection factor (UPF) values and ratings of RO-treated cotton

Sample	UPF values	UV-A blocking (%)	UV-B blocking (%)
Control sample	6.50	80.09	85.86
No dye	7.16	80.20	87.46
1 Layer	24.88	95.44	96.24
2 Layers	45.48	97.53	97.84
3 Layers	51.59	97.89	98.09
5 Layers	92.50	98.81	98.92

The control sample served as baseline, it had a UPF of 6.50, indicating poor UV protection. In contrast, the sample with no RO (no dye) slightly improved to a UPF of 7.16 due to the addition of a cross-linked binder layer. Moreover, the sample with 1 layer of RO dye showed a significant improvement in UPF to 24.88, indicating good UV protection. This enhancement is attributed to the UV-blocking composition of RO contributing to UV protection. Further, the sample with 2 layers showed UPF of 45.48, achieving an excellent UV protection.

Furthermore, the samples with 3 and 5 layers exhibited even higher UPF values, reaching 51.59 and 92.50, respectively, indicating excellent UV protection. These excellent results might be due to the RO containing metal oxides or other UV-absorbing substances that effectively blocked UV radiation by absorbing, reflecting, or scattering it away from the skin. Also, multiple RO layers may ensure better penetration and distribution of UV-blocking agents throughout the fabric, maximizing their effectiveness.

The cumulative effect of these factors results in significantly improved UPF values and ratings as the number of RO layers increases. This makes the RO-treated cotton fabric a more protective material against harmful UV radiation, which is crucial for applications in sun-protective clothing and outdoor textiles.

Thermal resistance and conductivity

Table 4 presents the thermal resistance and thermal conductivity values for cotton fabric samples treated with different numbers of RO padding layers. Thermal resistance measures the fabric's ability to resist heat flow, while thermal conductivity indicates how well heat is conducted through the fabric.

The control fabric sample had a thermal resistance of 0.059 m^2K/W and a thermal conductivity of 0.063 W/m/K, and served as the baseline for comparison. As the number of RO layers increased, the thermal resistance of the treated fabric samples increased, whereas the thermal conductivity decreased with the increase in layers. The corresponding thermal resistance increased, and thermal conductivity decreased with more dye layers, which can be attributed to several factors. For instance, the RO coats the fabric surface, adding a layer that can impede heat flow. This coating effect enhanced thermal resistance. The addition of dye layers increases the overall mass of the fabric, contributing to higher thermal resistance. The metal oxides present in the dye possess inherent thermal resistance properties, further enhancing the fabric's ability to resist heat flow. The pad-dry-cure dyeing process can alter the textile structure, creating a more tortuous path for heat to travel, thereby increasing thermal resistance. Metal oxides can also affect the reflection or absorption of thermal radiation, contributing to the overall thermal resistance of the fabric.

Table 4 shows that the relationship between thermal resistance and thermal conductivity is inversely proportional. As thermal resistance increases, the fabric's ability to conduct heat decreases. This makes the RO-treated fabric suitable for applications where heat insulation is desired, such as in curtains or tarpaulins, to keep indoor environments cooler by restricting heat transfer from outside. In conclusion, the dyeing process with RO containing metal oxides significantly enhanced the thermal resistance of the fabric, while reducing its thermal conductivity. This improved thermal performance can introduce cooling functionalities to textiles, making them beneficial for various practical applications in temperature regulation.

Table 4
Thermal resistance and thermal conductivity values of RO-treated cotton fabric samples

Sample	Thermal resistance (m^2K/W)	Thermal conductivity (W/m/K)
Control sample	0.059	0.063
1 Layer	0.062	0.058
2 Layers	0.065	0.051
3 Layers	0.066	0.049
5 Layers	0.068	0.044

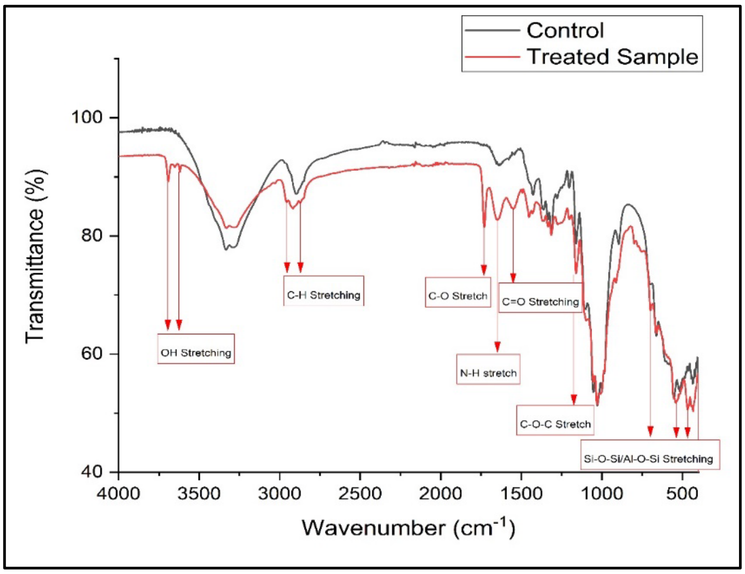


Figure 2: ATR-FTIR spectra of control and treated cotton fabric samples

Fourier transform infrared (ATR-FTIR) spectroscopy

As shown in Figure 2, the OH stretching at 3400 cm^{-1} indicates the presence of hydroxyl groups (–OH), often associated with water or alcohol. This noticeable change in the intensity suggests possible changes in the hydroxyl group content of cotton fabric. The C-H stretching at ~2900 cm^{-1} corresponds to the stretching vibrations of C-H bonds, typically found in alkanes. This may indicate the hydrocarbon structure of RO dye. A strong peak at ~1700 cm^{-1} is characteristic of carbonyl groups (C=O) and is associated with carboxylic acids and esters. The C-O stretch between ~1200-1300 cm^{-1} indicates the presence of C-O bonds in carboxylic acids and esters. These shifts or intensity changes are associated with carbonyl and ether or ester groups in the acrylic-based thickener and binder. The N-H stretch around ~1500-1600 cm^{-1} is associated with the stretching vibrations of N-H bonds, characteristic of the amines and amides content of the fixer used in the treatment. The C-O-C stretch (~1100 cm^{-1}) may correspond to the asymmetric stretching of C-O-C bonds associated with the esterification reaction between cotton and acrylic binder. The Si-O-Si/Al-O-Si stretching (~500-1000 cm^{-1}) in the broad region indicates the presence of silicate or aluminosilicate compounds of fixer.

The FTIR spectra indicate differences between the control and treated samples. The observed changes in peak positions and intensities suggest modifications in the functional groups, possibly due to the RO treatment applied to the sample. These changes could involve the introduction of new functional groups or alterations in the existing ones.

Thermogravimetric analysis

The thermogravimetric analysis (TGA) of the treated cotton fabric sample indicates higher thermal stability than that of the control sample. The treated sample exhibited better stability in the temperature range from 50 °C to 270 °C. This could be due to RO layers, which might form a protective barrier, enhancing the fabric's resistance to thermal degradation.[38] Despite its initial stability, the treated sample degrades earlier at around 300 °C. This could be attributed to the decomposition of the binder layer or the interaction between the dye and cotton fibers at elevated temperatures. Beyond 380 °C, the treated sample again showed improved thermal stability. This might be because the higher stability of RO contributing to a stable layer that protects the underlying cotton fibers.[36]

The treated fabric maintained a higher residual weight throughout the temperature range from 400 °C to 600 °C, suggesting that the RO imparts a more thermally stable residue than the control sample.

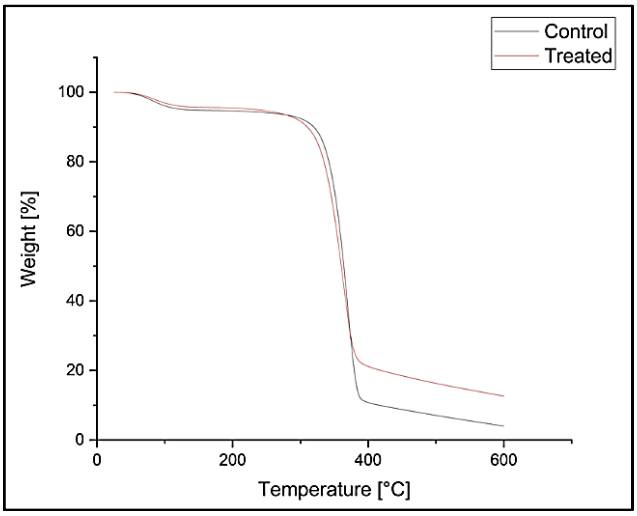


Figure 3: TGA curves of the control and RO-treated cotton fabric sample

CONCLUSION

This research investigated the use of Red Ocher for simultaneous dyeing and functional finishing of cotton fabric. Significant enhancements were confirmed in the treated cotton fabric samples compared to the control, through comprehensive characterization and testing. FTIR spectra showed the presence of functional groups of RO on the dyed cotton fabric. TGA analysis revealed that the treated cotton fabric exhibited higher thermal stability across a wide temperature range. The UV protection factor (UPF) tests demonstrated that increasing the number of dye layers significantly enhanced the fabric's ability to block UV-A and UV-B radiation. The K/S value measurements confirmed that the multilayered dyeing approach improved color strength with higher K/S values. Laundering and light fastness tests showed that the treated fabric retained its color well under various conditions, indicating good durability and stability of the dye on the cotton fibers. The treated fabric samples exhibited increased thermal resistance and decreased thermal conductivity, suggesting that the RO enhanced the thermal stability and introduced cooling functionalities to the fabric. This makes the treated cotton fabric suitable for thermal insulation applications, such as curtains and tarpaulins. Overall, incorporating RO layers significantly enhanced the functional properties of cotton fabric, making it more resistant to thermal degradation, better at blocking harmful UV radiation, and more durable in terms of color retention.

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