



Research Article

Development of TiO₂-Functionalized Hemp Cotton Blended Fabric with Enhanced Water Repellency

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KEYWORDS

Blended fabrics,
Functional fabric,
Nanoparticles,
Functional Coatings,
Water-repellent fabrics

ABSTRACT

The development of functional textiles is currently a significant research theme, with many researchers working to develop various functional properties, such as antimicrobial activity, hydrophobicity, and UV protection. This paper is written based on imparting near-super hydrophobicity to a hemp-cotton blended fabric with a ratio of 75% hemp and 25% cotton. Both hemp and cotton are natural fibers, and the fabrics made from these fibers are inherently hydrophilic. To impart hydrophobicity, TiO₂ was synthesized from titanium tetraisopropoxide (TTIP) in isopropyl alcohol. The characterization of the synthesized TiO₂ was validated by XRD analysis of the powder. This TiO₂ was then applied to the fabric by dip-coating, followed by a pad-dry-cure method. The coated fabrics were evaluated through Fourier Transform Infrared (FTIR) spectroscopy and contact angle measurement. FTIR spectra confirmed the successful deposition of TiO₂ nanoparticles, as evidenced by characteristic The treatment is particularly evident in the broadening and slight intensity changes in the region below 1000 cm⁻¹, specifically near 899 cm⁻¹ and stretching toward the 500–600 cm⁻¹ range, which corresponds to the characteristic Ti-O and Ti-O-Ti bridging vibrations. The contact angle measurement shows a significant increase from 65° for the untreated sample to 135° for the TiO₂-coated fabric, indicating nearly superhydrophobic behavior. These findings demonstrate that TiO₂ coating is an effective and environmentally compatible approach for enhancing the functional properties of natural fiber-based textiles. In this study, a minimum amount of chemical is used to enhance the functional characteristics of natural fiber. The use of a prioritized proportion of hemp fiber contributes to the uniqueness of this study.

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1. Introduction

During recent decades, the interest in high-performance, functional textiles has increased significantly due to the growing range of end-use applications in outdoor apparel, protective working apparel, sportswear, and technical textiles. Water repellency, or hydrophobicity, is one of the most practically significant of the many functional properties desired, determining directly whether a fabric will repel water or absorb moisture, and retain its structure and level of comfort in the wet [1, 2]. The natural fabrics that are made using fibers that are natural

and which are also preferred because of their breathability, tactile comfort, biodegradability, and sustainability, are, however, naturally hydrophilic as they have a large number of free hydroxyl (-OH) groups on their cellulose chains that form hydrogen bonds very easily with water molecules [3, 4]. This inherent incompatibility of cellulosic fibers with water and the water repellency that is needed for them to function forms the quintessence of the problem this paper will discuss. Among natural fibers, Cotton is the most popular reinforcement materi-

al since it provides exceptional hygroscopic properties, mechanical strength, tactile comfort, and easy handling benefits [5]. Hemp is not commonly used in the composite manufacturing industry, but its potential as a reinforcement material shouldn't be ignored. The internal structure of hemp fabric is responsible for a set of complex mechanical and physical properties. Inherently irregular yarns participate in the formulation of the fabric through weaving. The irregularities present in the yarn are difficult to control even under perfect drawing conditions due to the strenuous handling of the fiber [6]. Hemp fabric offers suitable features to be used in composite manufacturing as it possesses impressive mechanical properties, as well as provides good penetration benefit [7]. Nanotechnology provides a promising way of resolving the challenges. The 1-100 nm sized nanoparticles have extremely large surface-to-volume ratios that allow large alteration of surface chemistry and morphology to be achieved with a low load of material [8, 9]. Regarding the case of textile hydrophobicity, the two variables that determine the wettability of a surface are the surface free energy and the surface roughness geometry, which are outlined by the Wenzel and Cassie-Baxter wetting models [10, 11]. When the water contact angle (WCA) is greater than 90° , a surface is described as hydrophobic and superhydrophobic when it exceeds 150° [10]. Depositing nanoparticles onto a fabric surface introduces hierarchical nano-textured roughness, which entraps air beneath water droplets, and the surface is consequently pushed towards a hydrophobic or superhydrophobic state [12, 13]. The applications of these surfaces are well known: water-resistant fabrics are directly applied in outdoor and protective clothing, raincoats, military fabrics, and filtration fabrics, with wetting resistance being one of the foremost performance requirements [14, 15]. TiO_2 nanoparticles have attracted particular attention as a hydrophobic agent of textile substrates, which has been particularly high due to their chemical stability, non-toxicity, low cost, and high affinity to cellulosic fiber surfaces [9, 16]. The sol-gel technique with titanium(IV) isopropoxide (TTIP; $\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$) as a molecular precursor is the most common method of TiO_2 nanoparticles synthesis, as it is simple, low-processing-temperature, and produces high-purity nanoparticles with controllable morphology [13, 17]. During this procedure, TTIP is subjected to controlled hydrolysis and condensation in isopropyl alcohol with water added to it, to produce a colloidal TiO_2 solution. A critical ratio of the alkoxide to alcohol to water is very important in defining the kinetics of hydrolysis, crystallite size, and the surface area of the product [13, 17]. Heat treatment of the precipitate generates anatase crystalline form of TiO_2 that is denoted by a large number of surface hydroxyl groups, encouraging covalent bonding with the hydroxyl-rich surface of cellulosic fibers during the curing step [9, 18]. Previous research has proven that TiO_2 coating is an effective method of conferring hydrophobicity onto cotton and synthetic fiber-based substrates. TiO_2 nanoparticles prepared by the sol-gel method and deposited on

cotton fabric via a dip-coating process yielded a water-repellent and self-cleaning surface, where the enhancement in WCA was directly attributed to the nano-textured surface roughness formed by the coating [11]. Cotton fabric coated with a chitosan/ TiO_2 composite has further been shown to exhibit super hydrophobicity, with TiO_2 nanoparticles providing the hierarchical roughness required to sustain a Cassie-Baxter wetting regime [19]. It has also been confirmed that TTIP-derived TiO_2 exhibits a particularly strong affinity for cellulosic substrates, with anatase-phase nanoparticles bonding effectively to the hydroxyl groups of cotton fibers during thermal treatment [20]. These studies are, however, limited to single-component cotton substrates. Much less attention has been given to hemp (*Cannabis sativa*), a bast natural fiber with a multi-celled structure and inherently rough surface morphology [21, 22], as a substrate for TiO_2 -based hydrophobic functionalization. Importantly, no study to date has investigated TTIP-derived TiO_2 coating specifically on hemp-cotton blended fabrics with a primary focus on hydrophobicity, representing a clear gap in the literature on functional finishing of natural fiber blends. This gap is filled in the current study by the synthesis of TiO_2 nanoparticles using TTIP-solvents through the sol-gel technique and applying these nanoparticles to 75/25 blended fabrics of hemp and cotton using the pad-dry-cure technique. This novelty of the work is attributed to the fact that: (i) TTIP is the TiO_2 precursor that provides a precise control over the size of the nanoparticles and phase; (ii) hemp-cotton blended substrates, the roughness of which at the multi-scale level can synergistically enhance the hydrophobic influence of the TiO_2 nano-texture; and (iii) a focused assessment of hydrophobicity as the main functional outcome. Quantification of hydrophobicity was done by measuring the angle of contact with water. FTIR spectroscopy was used to determine the deposition and surface bonding of TiO_2 , and XRD was used to identify the crystalline phase and measure the crystallite size of the produced nanoparticles.

2. Materials and Methods

Table 1: Fabric specifications

Parameters	Values
Fabric type	Hemp Cotton blend (75% Hemp, 25% cotton)
Fabric structure	1/1 Plain Woven fabric
Yarn linear density	Warp = 20, Weft = 22
Yarn per cm	Warp = 42, Weft = 35
GSM (grams per square meter)	135

2.1 Materials

The fabric samples used in this study were collected from a renowned fabric manufacturing company of India. The fabric sample consists of cotton and hemp fiber in 75 % and 25 % ratios. The fabric composition analysis is presented in Table 1. A 1.5 g/L detergent solution was used to decontaminate the samples. Titanium isopropoxide (IV) ($\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$) and isopropyl alcohol were purchased from the local market.

2.2 Experimental Procedure

In this experiment, the precursor solution was prepared using 97% rated Titanium isopropoxide (IV) ($\text{Ti}(\text{OCH}(\text{CH}_3)_2)_4$) and 99.5% rated isopropyl alcohol. 250 ml alkaline distilled water with varying pH levels was added as the hydrolysis catalyst. TTIP, alcohol, and water were mixed at a 5:3:1 molar ratio. This solution went under vigorous stirring. HNO_3 was added to adjust the pH level of the solution. 0.2 moles HNO_3 solution was prepared in a separate flask using distilled water. Afterwards, acidic solution was added in the precursor solution under vigorous stirring. This mixture was maintained under reflux for 6h at 85°C . Once completed, white material precipitated in the solution. The precipitation was washed with distilled water and centrifuged. Finally, the precipitation was dried at 60°C and calcined at 500°C for 3 h. The TiO_2 nanoparticles were coated on samples using the pad-dry-cure technique. The samples were treated with a 1% concentration of TiO_2 solution at a 1:50 liquor ratio for 10 minutes. A specific amount of TiO_2 was mixed with water to create a nanoparticle solution. Then, the sample was immersed in the solution and continuously agitated, allowing TiO_2 particles to enter the fabric. Padding the samples in a padder machine resulted in a 75% pickup rate. Finally, the samples were dried at 100°C for 5 minutes and then cured at 120°C for 3 minutes.

2.3 Fourier Transform Infrared (FTIR) Spectroscopy

Fourier transform infrared spectroscopy was performed to identify the functional groups in the Dyes and Chemical Engineering Lab, BUTEX. The test was conducted using the PerkinElmer Two FT-IR spectrometer in the 400 to 4000 cm^{-1} frequency range, which covers the mid-infrared region.

2.4 XRD (X-ray Diffraction)

XRD analysis was conducted at the Textile Materials Engineering Lab, BUTEX. The XRD analysis of the prepared sample of TiO_2 nanoparticles was done using a Bruker-made diffractometer, Cu-K α X-rays of wavelength (λ)= $1.5406\text{ \AA}$. The diffractometer was running at 40 kV and 40 mA to determine crystalline size and phase identification. Data were taken in the range of $2\theta \approx 10^\circ$ to $2\theta \approx 80^\circ$, with a step of 0.1972° . The Debye-Scherrer equation was used to measure the crystalline size and dimensional structure of TiO_2 nanoparticles.

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

2.5 Water Contact Angle

The wetting characteristics of untreated fabric and TiO_2 -coated fabric were examined using the AATCC 79-2000 method. A high-resolution CMOS camera was used for instant capture of each droplet. The Contact Angle Meter CAM 01 software then calculated the contact angle.

3. Results and Discussion

3.1 XRD Analysis

The X-ray diffraction (XRD) pattern of the synthesized TiO_2 nanoparticles, recorded over a 2θ range of 10° – 80° using Cu K α radiation ($\lambda = 1.5406\text{ \AA}$), is presented in **Figure 1**. All diffraction peaks were indexed by comparison with the JCPDS reference cards for anatase TiO_2 (JCPDS #21-1272) and rutile TiO_2 (JCPDS #21-1276), confirming the presence of a mixed anatase–rutile phase. The dominant diffraction peaks at $2\theta = 27.47^\circ, 36.12^\circ, 39.23^\circ, 41.28^\circ, 54.37^\circ, 56.68^\circ$, and 69.06° are attributed to the (110), (101), (200), (111), (211), (220), and (301) planes of rutile TiO_2 , respectively. The peaks at $2\theta = 25.34^\circ$ and 64.10° correspond to the (101) and (002) planes of the anatase phase. The sharp, well-resolved nature of all diffraction peaks confirms a high degree of crystallinity in the synthesized TiO_2 [23, 24].

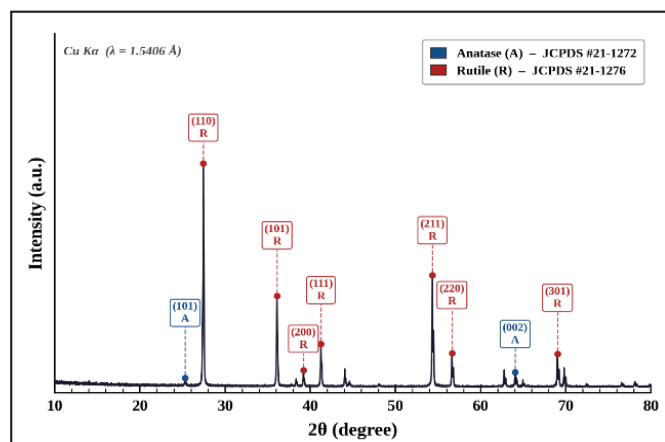


Figure 1. XRD graph of Titanium dioxide

The lattice parameter (a) of the synthesized TiO_2 was calculated using Bragg's law in conjunction with the Miller indices of each identified reflection, and the results are summarized in **Table 2**. For a simple cubic approximation, the lattice constant was derived from the relation $a = \lambda\sqrt{s} / (2 \sin \theta)$, where $s = h^2 + k^2 + l^2$. The average lattice parameter was determined to be $a = 4.679\text{ \AA}$, which is in close agreement with the reported JCPDS reference value for rutile TiO_2 ($a = 4.593\text{ \AA}$), with a deviation of only 1.88%. This small deviation is physically significant: it indicates the presence of residual lattice strain and/or minor structural distortion at grain boundaries introduced during the sol-gel synthesis and low-temperature calcination at 200°C . The consistency of the calculated lattice parameter with the

Table 2: TiO₂ Lattice Parameters

Bragg's angle			Simple Cubic			s		Ratio	Lattice constant (Å)	Average a (Å)
2θ	θ	sinθ	h	k	l	h ² +k ² +l ²	sin ² θ/s	λ ² /4a ²	a=√(λ ² /4(sin ² θ/s))	4.6795
27.4673	13.73365	0.237408699	0	0	0	2	0.028181445	0.03	4.5886	
36.11674	18.05837	0.309985721				4	0.024022787	0.02	4.9699	
41.28177	20.640885	0.35250951				6	0.020710492	0.02	5.3526	
54.36923	27.184615	0.456859085				8	0.026090028	0.03	4.7689	
64.10445	32.052225	0.530692038				10	0.028163404	0.03	4.5900	
56.67528	28.33764	0.474666529				12	0.018775693	0.02	5.6216	
69.06019	34.530095	0.566839037				14	0.022950464	0.02	5.0847	

reference value confirms the successful formation of crystalline TiO₂ without significant phase contamination, and validates the phase assignment made from peak indexing[25]. Without significant phase contamination, and validates the phase assignment made from peak indexing.

The average crystallite size (D) of the TiO₂ nanoparticles was estimated from the XRD peak broadening using the Scherrer equation.

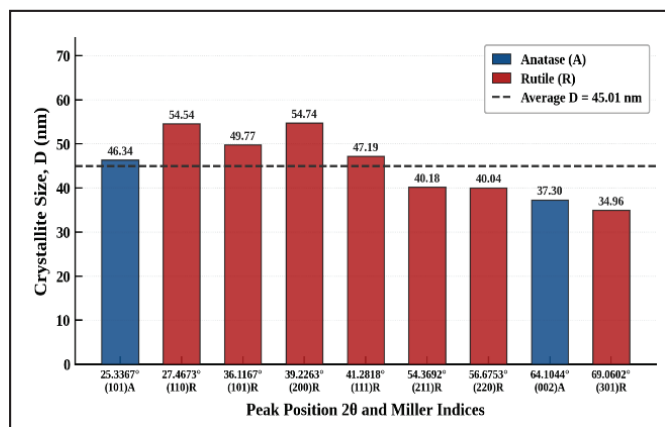
$$D = \frac{0.9 \times \lambda}{\beta \cos \theta} \quad (2)$$

where K = 0.9 is the Scherrer shape factor, λ = 1.5406 Å is the X-ray wavelength, β is the full width at half maximum (FWHM) of the diffraction peak in radians, and θ is the Bragg angle [26, 27].

The FWHM values of all nine identified peaks ranged from 0.150° to 0.276°, and the resulting crystallite sizes are detailed in Table 3. The calculated crystallite size varied from 34.96 nm to 54.74 nm across individual peaks, with a Scherrer average of D = 45.0 nm. Following the Warren correction for instrumental broadening (β_{instr} ≈ 0.08° for the Bruker diffractometer) [6], the instrumentally corrected average crystallite size is 50.2 nm. Both values are well within the nanoparticle regime (< 100 nm), confirming the nano-scale nature of the synthesized TiO₂. The variation in crystallite size across different peaks is expected, as the Scherrer method is sensitive to peak-specific broadening and to the anisotropic growth of crystallites in different crystallographic directions.

Table 3: Crystallite Size Estimation

Peak position 2θ (°)	FWHM β (°)	λ (Å)	D (nm)	Average D (nm)
27.4673	0.14996	1.5406	54.54	45.01
36.11674	0.16788		49.77	
41.28177	0.17988		47.19	
54.36923	0.22227		40.18	
64.10445	0.25128		37.30	
56.67528	0.2254		40.04	
69.06019	0.27581		34.96	
25.33668	0.1757		46.34	
39.22635	0.15408		54.74	

**Figure 2.** Crystallite Size Bar Chart

To further validate the crystallite size estimation and to decouple the contributions of crystallite size and microstrain to the total peak broadening, a Williamson–Hall (W-H) analysis was performed **Figure 3**. In the W-H method, $\beta \cos \theta$ is plotted against $4 \sin \theta$ for all peaks; the y-intercept yields the size term ($K\lambda/D$) and the slope yields the microstrain (ϵ) [28].

The W-H plot produced a linear fit with a y-intercept corresponding to a crystallite size of $D = 79.9$ nm and a slope corresponding to a microstrain of $\epsilon = 9.2 \times 10^{-4}$. The near-zero microstrain value confirms that peak broadening in this sample is dominated by crystallite size effects rather than strain-induced broadening. The larger D value obtained from W-H analysis compared to the Scherrer method is consistent with literature, as the W-H approach accounts for strain and tends to provide a volume-weighted size estimate. Taken together, the Scherrer and W-H analyses consistently confirm that the synthesized TiO_2 nanoparticles are crystalline and fall within the nano-scale range (< 100 nm). The narrow FWHM values (0.15° – 0.28°) observed in the diffractogram are therefore physically consistent with well-crystallized particles in the upper nanometer range, as expected for a sample calcined at 200°C .

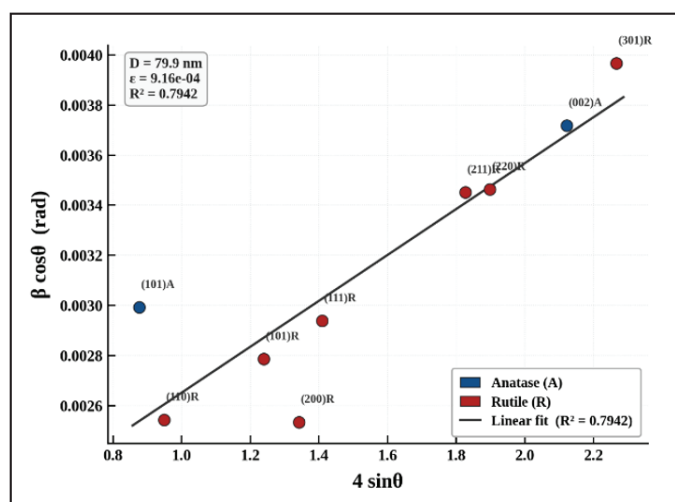


Figure 3. Williamson–Hall Plot

3.1 Analysis of Wettability

The wettability of textile materials was evaluated using a water droplet method, considering one side of both the treated and untreated fabrics. As shown in **Figure 4(a)**, the untreated fabric exhibited a contact angle of 65.84° , while the TiO_2 -treated fabric displayed a significantly higher contact angle of 135.27° , as shown in **Figure 4(b)**. When the contact angle (θ) is around 90° , the fabric surface is considered hydrophobic, while a value between 145° and 150° indicates a superhydrophobic surface [10]. In this experiment, the measured contact angle of 135° suggests that the treated fabric is nearly superhydrophobic. The increased contact angle is attributed to the TiO_2 coating on the fabric surface.

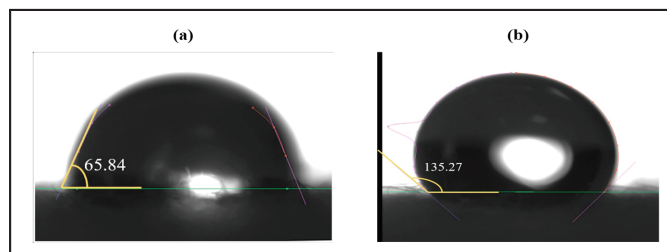


Figure 4. Water contact angle of 75/25 Hemp Cotton blended fabric (a) untreated, (b) sample with TiO_2 coating.

3.2 FTIR Analysis

Figure 5 shows the FTIR spectrum of the TiO_2 -coated 75/25 hemp–cotton blended fabric. The FTIR spectra of the TiO_2 treated 75/25 Hemp/Cotton blend show the characteristic cellulosic backbone which is marked by O–H stretching (3337 cm^{-1}), C–H stretching (2901 cm^{-1}), and C–O–C pyranose ring vibrations (1030 cm^{-1}) interfacing with the inorganic functionalization [29]. The treatment is particularly evident in the broadening and slight intensity changes in the region below 1000 cm^{-1} , specifically near 899 cm^{-1} and stretching toward the 500 – 600 cm^{-1} range, which corresponds to the characteristic Ti–O and Ti–O–Ti bridging vibrations [30]. Furthermore, the slight shift or reduction in the 1735 cm^{-1} (hemicellulose carbonyl) and 1636 cm^{-1} (absorbed water) peaks [31] suggests that the TiO_2 particles may be coordinating with the hydroxyl groups of the fiber, potentially displacing some bound water or masking certain organic functional groups through the surface.

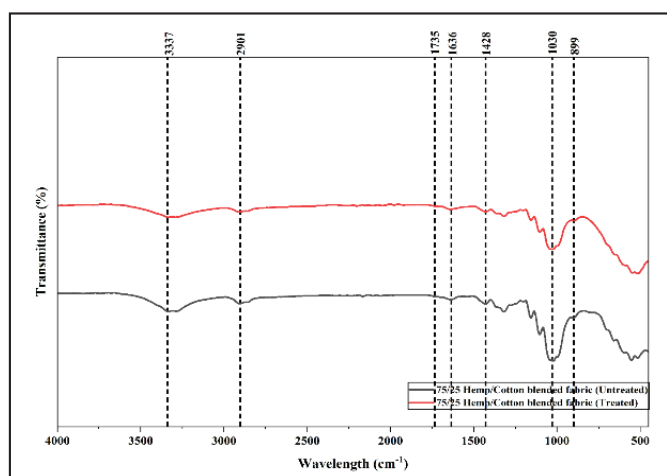


Figure 5. FTIR spectra of TiO_2 -coated 75/25 Hemp Cotton blended Fabric.

4. Conclusion:

In this study, TiO_2 nanoparticles were successfully synthesized and integrated into hemp–cotton blended fabrics through an efficient, low-chemical consumption process. The treatment transformed the naturally hydrophilic fabric (initial contact angle of 65°) into a highly water-repellent surface with a contact angle of 135° . This shift is attributed to the creation of

a nano-textured surface and lowered surface energy. FTIR analysis confirmed robust Ti–O bonding, ensuring the coating. While this study primarily focuses on achieving near-superhydrophilicity, the versatile nature of TiO₂ suggests that this functionalization could be expanded. By incorporating specific additives or optimizing the nanoparticle synthesis, it is possible to impart additional multi-functional properties such as antimicrobial activity, self-cleaning capabilities, and UV protection thereby creating a high-performance, 'smart' textile through further chemical analysis.

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