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JCHR (2024) 14(3), 518-529 | ISSN:2251-6727



Surface Modification of Cotton Fabric Using TiO2 Nanoparticle Dispersions Synthesized Through Spray Deposition.

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ABSTRACT:

(Received: 04 February 2024

Revised: 11 March 2024

Accepted: 08 April 2024)

KEYWORDS

TiO₂ nanoparticles, hydrophilic surfaces, Superhydrophobicity, Dip coating, Sol-Gel method, antibacterial properties, textile. Super hydrophobicity is of interest for practical applications such as water repellence, self-cleaning, stain resistance, and antibacterial properties. Due to its excessive hydrophobicity nature, Titanium Dioxide TiO_2 metal was selected as surface coating material. Initially, Titanium Dioxide nanoparticles were synthesized by the substantial route of using freshly obtained Egg Albumen (EA) proteins by Sol-Gel method. The Synthesized TiO₂ nanoparticles (NPs) were further characterized by the FTIR, SEM, and AFM. The Synergistic super hydrophilic surfaces study was done by super hydrophobic through spray deposition of nanostructured TiO_2 over textiles. Repellence behavior was studied using static and dynamic contact angles and also coating is tested for mechanical durability and thermal stability. The method with egg albumen provides us with a cheap, green, and repeatable route for the uniformly synthesizing and mechanical properties of the TiO₂ NP's representing the basis for a high-quality and well-durable hydrophobic fabric preparation therefore that influence of the modification procedure on textile handling futures

1. Introduction

1.1 General:

Superhydrophobic surfaces, characterized by a water contact angle surpassing 150° and a droplet sliding angle below 10° [02], have garnered significant attention in recent decades due to their versatile applications. These applications include but are not limited to anti-corrosion, anti-icing, drag reduction, and non-wetting properties. The attributes of flexible superhydrophobic textiles, such as effective selfcleaning, breathability, non-wetting properties, and lightweight construction, have become prominent in various sectors, spanning technical, industrial, medical, military, and everyday life applications. These range from beach umbrellas, shade shelters, and camping gear to clothing and advertising materials [06]. Integrating ultraviolet (UV)-shielding [18] properties in superhydrophobic textiles further enhances their practical applications.

Drawing inspiration from the superhydrophobic properties observed in lotus leaves, the combination of particular surface topography and low surface energy can give rise to superhydrophobic surfaces [7]. Numerous superhydrophobic surfaces incorporate some roughness, frequently achieved by integrating inorganic nanoparticles like TiO_2 [21]. Renowned for their exceptional UV protection, TiO_2 nanoparticles can be utilized in surface hydrophobication alongside low surface energy substances [25] like silicones or fluorocarbons to develop multifunctional textiles exhibiting both UV-shielding and superhydrophobic attributes.

Young's equation:

$$\cos\theta_{\rm Y} = (\sigma_{\rm sv} - \sigma_{\rm sl})/\sigma_{\rm lv}$$

 θ_{Y} = Young's contact angle σ_{sv} = surface tension (energy per unit surface) of the solid-vapor interface www.jchr.org

JCHR (2024) 14(3), 518-529 | ISSN:2251-6727



 $\sigma_{\rm sl}$ = surface tension of the solid-liquid interface

 σ_{lv} = surface tension of the liquid-vapor

A smooth, flat, stiff, and homogeneous surface is assumed for the measurement of the "contact angle (CA)," (θ), when a water droplet is present.

Wetting phenomena:

The electrical dipole property of liquid water is the main reason for its comparatively high surface tension. Water's tendency to stick to surfaces, even itself, is one simple explanation for its dipole behaviour [25].



Figure 1: Force diagram of Young's equation and associated Contact Angle measurement.



Figure 2: Hydrophilic and hydrophobic wetting conditions.

Surfaces that interact with water droplets are classified as hydrophilic (attracting water) or hydrophobic (repelling water). Water droplets prefer to disperse or moisten surfaces with high surface energy chemistry, like glass. On the other hand, water droplets bead up on surfaces with low surface energy chemistry, such as Teflon [19].

A surface is said to be hydrophobic when a drop of water sticks more to itself than to it; as a result, the water drop will bead up and create a contact angle larger than 90° . On the other hand, a surface is said to

be hydrophilic and a water drop with a contact angle of less than 90° would stick to it more than to itself. Whereas a drop of water that shows no interaction with a surface (no wetting) will bead up into a perfect spherical (disregarding gravity) and have a contact angle close to 180°, a drop that completely wets a surface will have a contact angle close to 0° [11]. These situations are depicted in the pictures in Figure.

Dynamic contact angles (advancing and receding)

A water drop on a slanted surface with advancing (θ_a) and receding (θ_r) contact angles is depicted in Figure 3. The contact angle hysteresis (θ) is the difference between the advancing and receding Cas [04]



Figure 3: Advancing and receding dynamic contact angles.

Roll-off angle:

The roll-off angle (θ_R) is the angle at which a water drop rolls off a slanted flat surface. Roll-off angles of superhydrophobic surfaces are typically less than 5 degrees. On the other hand, very good superhydrophobic surfaces can show roll-off angles of less than one degree [09]. A water drop with a roll-off angle of roughly 5 degrees is shown in Figure 4.



Figure 4: Angle of roll-off of water drop.

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JCHR (2024) 14(3), 518-529 | ISSN:2251-6727



Impact of topology or surface roughness on contact angle:

The localized contact angle of a liquid water drop on a flat, smooth, homogeneous surface (substrate) is dictated by the surface tension of the liquid and the surface chemistry (surface energy) [17]. However, depending on the surface chemistry of the substrate, an increase in surface roughness may cause a change in the effective contact angle. Raising the surface roughness will usually result in a smaller contact angle if the surface chemistry is hydrophilic. On the other hand, when a surface becomes rougher, its contact angle could increase if its surface chemistry is hydrophobic.

Roughening may result in a decrease in the effective contact angle for a hydrophilic surface and an increase in the effective contact angle for a hydrophobic surface. The first sequence in Figure 5, for example, shows the wicking effect of water in contact with a hydrophilic glass cone spike over time through the illustration of a water drop on the spike. However, if the cone spike's surface chemistry is hydrophobic, as seen by the second sequence in Figure 5, a water drop will be repelled from it. It's crucial to remember that the effective contact angle might vary greatly over time, yet the localized contact angle stays constant. The effective contact angle for a hydrophilic surface can drop to about 0° from about 45° [14], while for a hydrophobic surface, it can rise to almost 180° from about 120° [12]. It is also important to note that when a surface is roughened or its topography is greatly increased, a hydrophilic surface becomes even more hydrophilic and a hydrophobic surface becomes even more hydrophobic.



Figure 5: Water repellency produced by a hydrophobic chemistry on a spikey surface.

1.2 Objectives of research:

- The following are the objectives of the work
- 1. Synthesis of uniformly dispersed TiO₂ nanoparticles using egg albumen as capping agent.

- Preparation of effective hydrophobic solution by mixing various proportions of TiO₂ nanoparticles.
- 3. Fabrication of hydrophobic cotton fabric using hydrophobic solution.
- 4. Analysis of mechanical & handling properties coated cotton fabric.

2. Review of Literature:

Boinovich et. al. (2008) - explores the theoretical foundations for designing hydrophobic materials and superhydrophobic coatings, emphasizing surface characteristics. It discusses the formation of ordered textures, ensuring high contact angles and the thermodynamic stability of heterogeneous wetting regimes. The review outlines current methods for designing materials with specified topology and variable hydrophobicity, addresses issues of aging and degradation in superhydrophobic coatings, and provides examples of successful applications across various industries

Jingjing et. al. (2010)- explain novel bio-templated approach using denatured egg albumen (EA) proteins to synthesize uniform and well-dispersed TiO₂ nanoparticles. The method yielded anatase TiO₂ nanoparticles around 9nm with a narrow size distribution, confirmed by XRD and TEM analysis. Characterization via XPS, FTIR, TG-DTA, and UV-Vis elucidated the synthesis mechanism. This green, costeffective method offers a reproducible route for nanoparticle fabrication under mild conditions.

Anand et. al. (2011)- illustrates review provides a comprehensive overview of self-cleaning coatings, emphasizing prevalent materials in both research and commercial spheres. Focused on hydrophobic and hydrophilic coatings, it delves into fabrication methods, functional aspects (Anti-icing, Electro-wetting, Surface switch ability), and potential applications. The review evaluates characterization techniques and testing feasibility, highlighting untapped potential despite some commercialization in self-cleaning technology.

Yi zhou et. al. (2014) -This literature highlights a novel hydrophobic composite coating incorporating modified TiO_2 nanoparticles and polytetrafluoroethylene (PTFE) micro powders in a fluorocarbon resin matrix. The resulting material demonstrates notable hydrophobicity

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JCHR (2024) 14(3), 518-529 | ISSN:2251-6727



(water contact angle of 133°) and self-cleaning properties. Characterization via scanning electron microscopy and X-Ray photoelectron spectroscopy confirms a micro/nanocomposite structure. Under ultraviolet irradiation, the composite coating efficiently removes oleic acid.

Mertens et. al. (2017) – investigates about the impact of TiO_2 and SiO_2 nanoparticles on atmospheric pressure plasma-synthesized fluorocarbon films in argon and helium atmospheres. Analysis through WCA, XPS, SEM, and profilometry reveal significant effects on film roughness and hydrophobicity. Different influences of nanoparticles are observed with helium and argon, with TiO_2 leading to coatings degradation over time. The study discusses the influence of plasma power and deposition time.

Lihui et. al. (2018) -explains the fabrication of superhydrophobic and UV-blocking cotton fabrics through the incorporation of TiO_2/SiO_2 composite nanoparticles, followed by hydrophobication with hexadecyltrimethoxysilane (HDTMS). Characterization via FTIR, XRD, and SEM confirmed the composite's structure. The resulting fabric exhibited a water contact angle of 159.3°, water shedding angle of 8°, and a high UV protection factor (UPF = 115.42) with UVA transmittance of 3.35%, demonstrating effective superhydrophobicity and UV-shielding properties.

Balraj et. al. (2020) - focuses on achieving superhydrophobicity on cotton fabric for versatile applications. The coating, formed through immersion in TiO_2 nanoparticles and perfluorodecyltriethoxysilane solution, demonstrates a remarkable water static contact angle of 169.3° and excellent tilt angle of 6.3°. Characterized by stability tests, the coating exhibits exceptional mechanical, chemical, and thermal durability. The fabric displays outstanding self-cleaning, stain resistance, rust stain resistance, antiwater absorption, antibacterial properties, and efficiency in oil-water separation, establishing its multifunctional utility with high reusability.

Darliane et. al. (2022) - introduces hydrophobic cellulose-based fabrics with embedded mesoporous TiO_2 particles for potential use in healthcare and contaminated settings. Applying sol-gel hydrosols to

cotton and non-woven fabrics exhibits significant virucidal effects on Murine Coronavirus (MHV-3) and Human Adenovirus (HAdV-5) under indoor light irradiation. The results highlight the efficacy of TiO₂coated fabrics in preventing virus contamination, particularly beneficial for healthcare indoor environments.

3. Experimental Details:

3.1. Materials:

Anhydrous ethanol (AE) and titanium iso-propoxide (TTIP) [02] were purchased from Sigma-Aldrich. Fresh eggs were acquired from a local market. Egg yolks were carefully separated using tweezers, and the chalazae cords were removed to extract egg albumen (EA) [04]. Fluorocarbon (FC) (98% purity) was sourced from Indian Mart. Throughout the entire process, only deionized water (DW) [05] was utilized.

3.2. Preparation of EA-Templated TiO2 Nanoparticles:

In a standard procedure, 3 mL of egg albumen (EA) [04] was combined with a solution containing 150 mL of Ethanol and 3.75 mL of de-ionized water (DW) & solution was stirred 25min. Then, the mixture was filtered to eliminate the floc, which consists of several thin filaments likely corresponding to the insoluble ovomucin. Then 9 mL of titanium isopropoxide (TTIP) was added dropwise to the solution under vigorous stirring. The resulting mixture was stirred further for 4 hours at a temperature of 85°C and then allowed to cool to room temperature (26°C) for 45 minutes [19]. Subsequently, the solution was sealed in an airtight container using silver foil and subjected to sonication for 1 hour, resulting in the controlling aggregation of solid particles. The solid product was isolated via centrifugation at 4000 RPM for 45min so the solution & TiO₂ nanoparticles(A) were separated and separated particles were thoroughly separated using filter paper was kept dried under the room temperature $(26^{\circ}C)$ [17].

www.jchr.org

JCHR (2024) 14(3), 518-529 | ISSN:2251-6727





Figure 6: Separation of egg albumen



Figure 7: Titration of mixing egg albumen

3.3 preparation of hydrophobic solution:

The 2ml of fluorocarbon (FC) was added dropwise into the 24ml of Deionized water (DW) under vigorous stirring for 30min [04]. Then 05 mg of synthesised TiO_2 nanoparticles(A) were added and kept stirred for another 30min [01]. Later stirred solution were filled in the container and kept for sonication for 1 hour to enhance the proper mix between the nanoparticles & the solution. the prepared solution was stored in a container.

In the above-mentioned process, 5 more hydrophobic solutions (A= 10mg, 15mg, 20mg, 25mg & 30mg) were prepared simultaneously.



Figure 8: Synthesised Tio₂ NP's

3.4 Synthesis of hydrophobic coating:

The cotton fabric was maintained at room temperature, approximately 27°C. An 8 M NH₄OH solution [24] was consistently utilized to create adherent cotton fabric films. Before dip-coating, the hydrophobic agent solutions were shaken for a minimum of ten minutes to re-mix the sedimented nanoparticles. The resulting hydrophobic solution was then transferred into glass jar tubes with an inner diameter of 30 mm and a height of 85 mm. Cotton fabric Substrates were vertically dipped in hydrophobic solutions and withdrawn at a rate of 3 mm/sec before gelation occurred. Then, again cotton fabric substrate was immersed in the solution for 20 minutes in the glass dish. The samples were coated and subsequently dried at room ambient temperature (approximately 27°C) [16]. After drying the samples were used for experimental and characterization purposes.



Figure 9: Immersion of fabric into the hydrophobic solution

3.5. EXPERIMENTAL SECTION:

3.5.1 Experimentation on changes in mechanical properties of the fabric

The coated samples were regularly washed with a variety of solvent solutions and detergent water, using the sonication method for an hour, to guarantee mechanical stability [20]. The coated samples were then dried, and a new evaluation of their wettability was conducted which is resulted in up to 8 washes and the performance of the fabric was shown to decrease.

In addition, an abrasion test comprised rubbing the coated fabric against sandpaper while holding a 20 g weight that showed white powder coming out from the

www.jchr.org

JCHR (2024) 14(3), 518-529 | ISSN:2251-6727



surface of the fabric, and a high-speed water jet spray was used to assess the mechanical durability of the fabric, in the jet spray initially it was opposed [13] the water up to 3.5seconds after that wetting phenomenon will be initiated and the tap spray was resisted 26 seconds. The coated samples were periodically submerged in solutions ranging in pH from 5 to 11 [24], simulating severe corrosive environmental conditions, and their wettability was evaluated following the results. Additionally, the coated fabric's stability at high temperatures was tested by annealing it for an hour at temperatures between 50 and 300°C, cooling it to room temperature, and looked for any visible physical changes and fabric was start black coloured formation from 110°C after the time span of 335seconds and no powder will be coming off the surface.

By striking water droplets at different velocities on the created superhydrophobic cotton fabric, the dynamics of droplet behaviour were studied. The droplet behaviour was observed and captured on video with an IQOO 9se mobile camera (480 FPS, Exmor, Sony). In addition, to analyze the impact dynamics, the Weber number (We = $\rho Dv^2/\sigma$, which represents the ratio of kinetic energy to surface energy) and Reynolds number (Re = $\rho Dv/\mu$, which represents the ratio of inertial force to viscous force) were calculated. Here, ρ , D, μ , and σ stand for the liquid's density, droplet diameter, dynamic viscosity, and surface tension, respectively [13].

3.5.2 Applications of Superhydrophobic Fabric:

The coated sample's ability to clean itself was tested by first applying dust particles to its surface and then spraying water on it to see if any dust was removed. A colored water drop was applied to the sample, allowed to evaporate over many hours, and the stain it left behind was quantified to determine the stain resistance. To further evaluate the fabric's stain resistance qualities [10], hanging it from a metal wire caused rust to discolour the fabric.

In the oil-water separation experiment, a coated cloth sample was placed on a funnel and covered with a 1:1 mixture of coconut oil and distilled water. The filtrate was then collected in a beaker. This procedure was repeated for an oil-in-water emulsion without surfactants that was made by combining coconut oil and distilled water in a 1:1 ratio and stirring continuously for an hour [12].



Figure 10: Hydrophobic solution coated fabric sample

3.6 Characterizations:

Using a microscope running at 200 kV, standard and scanning electron microscopy (SEM) measurements were made [02]. Before analysis, all samples were ultrasonically dispersed in ethanol, placed on the surface of a cloth that was supported by a copper grid, and allowed to dry naturally. Using a KBr pellet method, Fourier transform infrared (FT-IR) spectra were acquired with a Bruker Vector 22 FT-IR spectrophotometer [08]. Using the Quanta chrome ChemBET 3000 chemisorption analyzer [09], BET surface areas were calculated. AFM (atomic force microscopy) analysis was performed on the B-2 AFM using AFM Control software [11].

4. Results and Discussion

4.1. Surface Morphological Studies:

SEM micrographs were used to analyze the twodimensional surface morphology of the unmodified (A = 0) and TiO2-modified (A = 15 mg) cotton films [03]. SEM pictures of the original and TiO₂-modified cotton films at 1000x magnification are shown in Figures (a and b). The unaltered cotton film in Figure (a) has a less dense, network-like surface topology. On the other hand, the TiO₂-modified cotton film (Figure b) has a very porous and dense surface morphology.

www.jchr.org

JCHR (2024) 14(3), 518-529 | ISSN:2251-6727





Figure 11 (a): SEM image of the unmodified cotton film





The hydrophobic cotton film (12 ml) modified by FC is shown in Figures (c and d) at two different magnifications in SEM pictures. The presence of small and dense pores within the film is confirmed by the low magnification image (c) [17]. A high magnification image of TiO₂ nanoparticles (d) was taken to allow for a more in-depth analysis of these pores and to provide information on their diameter. The film's high magnification SEM micrograph displays [21] a microporous surface morphology, with randomly scattered pores that are normally 60 to 70 nm in diameter on the substrate.



Figure 11 (c): SEM image of the FC modified (12ml) cotton film with 5,000X magnification.



Figure 11 (d): SEM image of the TiO2Nano particles with 5000X magnification.

4.2 Atomic Force Microscopy (AFM):

A surface's wettability is influenced by both its topography and chemical makeup. The threedimensional AFM pictures of the as-deposited, TiO₂modified, FC-modified (12 ml) [07], and TIO₂ NP'Smodified (15 mg) cotton films on the cotton substrates are shown in Figure 3.2 (a-d) [08]. In contact mode, the pictures were taken at a planar size of $1 \times 1 \mu m^{-2}$. The films' surface displays a large number of scattered islands spread out over it. Using AFM, the root-meansquare (RMS) roughness value of every film was examined. The RMS roughness values of the asdeposited (A = 0), TiO₂ modified, FC modified (12 ml), and TIO₂ NP's modified (15 mg) cotton films.



Figure 12 (a): AFM image of the unmodified (A = 0) cotton film.



Figure 12 (b): AFM image of the FC cotton film.

www.jchr.org

JCHR (2024) 14(3), 518-529 | ISSN:2251-6727





Figure 12 (c): AFM image of the FC & TiO_2 modified (A = 15 mg) modified cotton film.



Figure 12 (d): AFM image of the TiO₂ NP's.

4.3 Fourier Transform Infrared Studies:

Using FT-IR spectroscopy and the KBr technique in transmission mode, the chemical makeup of the films formed on the cotton substrate was examined. Numerous distinct absorption peaks were seen in the 3336.13 cm⁻¹ range, indicating the existence of organic groups in the material [06].

The FT-IR spectra of the unmodified cotton film and the TiO₂-modified cotton film, with a molar ratio of (0.579×10⁻⁵) OF 10 mg, are shown in Figure 3.3 (a-c), respectively. The stretching and bending of C-H bonds responsible for absorption bands located is approximately at 3336.13 cm⁻¹ and 1400 cm⁻¹ [07]. The presence of C=C alkene groups is indicated by peaks at about 1636 cm⁻¹ and a broad absorption band at 3336 cm⁻¹. Less intense C-Br absorption peaks at 694 cm⁻¹ are seen in the case of the unmodified (A = 0) film, while larger C-Cl peaks at 806.9 cm⁻¹ indicate the hydrophobic character of the cotton film.

A notable fraction of silanol groups is present on the film surface of the as-deposited cotton films, as indicated by a prominent peak at 3336.13 cm^{-1} , which decreases as the A value increases. Compared to films created with A values of 10 mg. while larger C-Cl peaks at 806.9 cm⁻¹ indicate the hydrophobic character of the cotton film

Frequency (cm ⁻¹)	Functional Groups		
3336.13	=C-H stretch		
1636.14	C=C alkene		
694.30	C-Br		
806.90	C-Cl		
855.89	C-Cl		



Figure 13: FTIR spectra of the FC & TIO₂ NP's (A=15 mg) modified cotton film.

www.jchr.org JCHR (2024) 14(3), 518-529 | ISSN:2251-6727



4.4 Water contact angle measurements:

The contact angle of a water droplet on a surface is typically used to determine the hydrophobic nature surface. As the molar ratio of FC/TIO₂ NP'S increases [6]. The water droplet was first positioned on a horizontal surface, then the film surface was progressively tilted until the droplet began to slide. This allowed to measure the water droplet's sliding angle.



Figure 14: contact angle of the droplet over the cotton fabric substrate

With an increase in A value, it was found that the static water contact angle increased and the water sliding angle decreased [23]. In particular, an A value of 5 mg has shown the lowest contact angle of 1250& highest in sliding angle 12⁰, also at 15mg shown the optimum results on contact angle of 140° &sliding angle of 8° . Water droplets rolled off the film surface from all directions, leaving no discernible water trace behind. Van der Waals forces produced by the liquid-solid interface between the coated substrate and the water droplet are the main source of adhesion. The unaltered cotton films. showed a strong adherence to the water droplet and at the A value of 30mg needed gravity to push the droplet off the surface [07] with a sliding 63.37 µN of maximal frictional force and a sliding angle of 12^{0} . The adherence of the water droplet to the film surface, however, decreased for the 15 mg FC/TIO2 NP'S modified cotton films 12]. As a result, the water droplet with a lowest frictional force of 27.01 µN could effortlessly roll off the slanted substrate ($SA = 8^{\circ}$).



Figure 15: measure mentation of Sliding angle

Table 3.4: Maximum frictional force and static and

 dynamic water contact angle values change as A values

 rise.

S.no.	TiO ₂ nano particles (mg)	Water contact angle (θ)	Water sliding angle (0)	Maximum frictional force fmax (µN)
1	5	1250	120	58.97
2	10	1290	110	42.96
3	15	1400	80	27.01
4	20	1350	100	35.12
5	25	1320	110	50.47
6	30	1330	100	63.37



Figure 14 (a) : graphical representation of contact angle data

www.jchr.org

JCHR (2024) 14(3), 518-529 | ISSN:2251-6727





Figure 14 (b): graphical representation of sliding angle data



Figure 14 (c): graphical representation of maximum frictional force (f_{max}) data

5. Conclusions

Using dip-coting method, optimized proportions involving in 1ml of FC in 12ml of DW along with the 15mg of TiO₂ nanoparticles shown best production of superhydrophobic cotton fabric. That coated fabric showed $140 \pm 2.1^{\circ}$ for static water contact angle and 10 $\pm 2.0^{\circ}$ as sliding angle. Thorough testing in a range of settings validated the fabric's wetting stability and showed encouraging mechanical, chemical, and thermal stability of the coating.

The cotton fabric that was produced an exceptional qualities such as reduced water absorption, stain resistance, and tenacity against rust stains. It was also very self-cleaning. Furthermore, the cotton fabric that is superhydrophobic exhibited dependable antibacterial properties and worked well in oil-water separation procedures, attaining a high separation efficiency [20]. Because of this, the prepared coated cloth exhibits adaptability for use in both home and industrial settings.

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