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METHODS

Blocking ultraviolet and anti-staining for cotton fabric 100% using nano titan dioxide

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Nowadays, customers have an increased demand for casual clothes and clothes with additional functions such as anti-staining, anti-ultraviolet (UV), and flame-retardant without losing comfort when wearing them. Some rays from the sun, especially UV rays (i.e., UVA and UVB) are harmful to human health. At high intensity, UV rays can cause sunburn, increasing the possibility of getting cancer. UV radiation damages the skin and destroys skin cells. Fabrics that not only have the ability to block UV rays but also have anti-staining ability are being studied more and have high competitive advantages over fabric items when people pay more and more attention to beauty and health. Using nano TiO_2 to complete UV protection is a suitable method due to its stability, non-toxicity, and wide application technology. Therefore, this research is performed to finish blocking UV, anti-staining treatment on 100% cotton fabric with nano TiO_2 using the popular method in textile finishing of pad-dry-cure aim to attach bonding chemicals to the fabric under the influence of technological parameters as follows: concentration of nano TiO_2, pick-up level, processing temperature, and processing time are different. From there, we can choose the most optimal treatment method for the bifunctional finishing cotton fabric to provide to the textile market.

Keywords: cotton, nano-titan dioxide, self-cleaning, UV transmission

1. Introduction

The Sun emits ultraviolet (UV) rays such as UVA, UVB, and UVC, but because there is absorption of the ozone layer, 99% of UV rays reaching the earth's surface are in the form of UVA rays. The higher the altitude, the more the UV rays, the closer to the equator, the stronger the UV rays. According to the time of day, UV rays influence the highest in the range of 280–400 nm. UVA radiations are also known as long waves or "black light." This ray region is close to the visible light region, with lower energy than the UVB and UVC. UVB rays (315÷280 nm; 3.94÷4.43 eV) or medium wavelength and UVC light (280÷100 nm; 4.43÷12.4 eV) or short wave have coincided. This is the region where UV rays have the highest energy. A small amount of UV light is needed to produce vitamin D, which helps keep bones and muscles strong, and every 15 min of sun exposure in the summer creates higher levels of UV rays that cause skin cancer (1, 2). The majority of skin cancer cases in Australia are caused by exposure to UV rays. In Chile, which is said to have the highest intensity of UV radiation from the sun, the rate of skin cancer in the country has increased by 10.5% over the past 5 years. In our country, skin cancer ranks eighth among the ten most common types of cancer with an average rate of 2.9–4.5 cases per 100,000 population. With the above statistics, many scientists have studied many measures to prevent the impact of UV rays on human skin, for example, cosmetic products, UV protection chemicals with SPF > 15, fashion glasses, car glasses, construction works, and UV-resistant textile products where UV-resistant textiles are a necessary method to protect people from UV rays from the face. Vietnam is located in a geographical area near the equator, so the amount of UV rays received is very high and the demand for UV protection products is very high (3, 4). However, on the market, most of them are imported from abroad with expensive prices because, in fact, our country has not

TABLE 1 | Specific parameters of original cotton fabric 100%.

Fabric	Type	Density warp/weft (yarn count/cm)	Mass (g/m^2)	Width	Original UPF
Cotton 100%	Plain Woven	28/24	110	150 cm	10

studied deeply and applied practically to the production of UV-resistant finishing technology. The finishing achieves protection against UV rays by either reflecting 100% of the UV rays or by absorbing it by the finishing chemical and converting it to a harmless heat-generating ray (5, 6). When choosing UV absorbers in the field of textiles that protect against UV rays, it is necessary to choose products that have the greatest absorption capacity in the range of UV-A and UV-B radiations. Inorganic UV absorbers such as TiO_2 can absorb light with higher energy (7, 8). The presence of this inorganic substance in textiles increases their ability to diffuse, reflect light from the surface, and increase UV resistance. Currently, with nanotechnology, this type of anti-UV product has a size equal to or even smaller than the UV wavelength, which has significantly improved the UV absorption rate. Furthermore, nanomaterials increase their specific surface area and surface energy, facilitate easy incorporation of large molecular substances, reduce diffuse reflection of visible light, increase transparency, and ensure the natural color of textile products. Therefore, this study will perform the best anti-staining treatment sample on the fabric and measure the UV resistance and then select the best anti-staining and anti-UV finishing treatment technology using nano TiO_2 with the popular finishing treatment technology on 100% cellulose natural fabric.

2. Research elaborations

2.1. Raw samples and chemicals

The raw samples were scoured and bleached, provided by Viet Thang Corporation. The parameters of the fabric are given in **Table 1**:

Nano TiO_2 (<100 nm, China); distilled water; and survey stains including coffee and tea.

2.2. Methods

Carry out investigation experiments: Concentration, pick-up level, processing temperature, and treatment time to the fabric's anti-staining and UV resistance.

Investigate the concentration of nano TiO_2 on the anti-UV, anti-staining performances of samples with: TiO_2 levels: 6, 5, 4, 3, and 2 (g/L) corresponding to the name of the fabric samples ND5, ND4, ND3, ND2, and ND1; pick-up level: 70%; processing temperature: 130°C; and processing time: 90 s.

Investigate the pressure (pick-up level) to UV resistance, anti-staining performances of samples with titan dioxide: chemical's concentration: 4 g/L; pick-up level: 60, 70, and 80 (%) corresponding to the name of the fabric sample is F1, F2, and F3, respectively; processing temperature: 130°C; and processing time: 90 s.

Investigation of treatment temperature: TiO_2 concentration: 4 g/L; pick-up level: 70%; treatment temperature: 110, 120, 130, 140, and 150°C corresponding to the name of the fabric samples are S1, S2, S3, S4, and S5, respectively; and processing time: 90 s.

Investigation of the treatment time to anti-UV, anti-staining performances of samples: nano titan dioxide concentration: 4 g/L; pick-up level 70%; processing temperature: 130°C; processing time: 50, 70, 90, 110, and 130 s corresponding to the name of the fabric samples TG1, TG2, TG3, TG4, and TG5, respectively.

2.3. Treatment processes

The pad-dry-cure method is used.

Step 1: Prepare the fabric sample. Cut fabric samples with dimensions of 20 cm × 20 cm.
Step 2: Prepare the complete solution with the volume of each fabric sample of 300 mL. Chemicals with different concentrations are used according to the experimental requirements.
Step 3: Perform padding (on the experimental padding machine).

The impregnation solution is mixed and stirred evenly according to the technology application (the concentration of the finishing agent as above). Then the fabric sample is placed into the chemically mixed glass bottle, and the fabric sample is allowed to absorb evenly and continue to pad fabric. The pressure level is set on the parameters through the padding machine: with a speed of 14 m/min and a pressure of 3 bar.

Step 4: Dry the fabric samples at 80°C for 1 min.
Step 5: Perform curing (on the laboratory dryer). The processing temperature and time are different for each sample.
Step 6: Store samples in nylon bags.
Step 7: Anti-staining survey with two basic stains: coffee and green tea.

- Drop a drop of coffee and a drop of tea equivalent to 0.05 mL onto the coated fabric surface at a height of 10 ± 1 cm.
- When the water drop has just come into contact with the fabric surface, take a picture of the water drop with a camera with the highest resolution in the cross-section and vertical section.

- The fabric samples are then dried in the sun. Every 4 h, fabric samples were observed and photographed to assess the ability of stains to fade on the fabric surface.

2.4. Structure and morphology characterization

In this investigation, the structure and morphology of the fabric before and after coating were characterized based on electron microscopes of Hitachi Company. The structures of the samples were specifically characterized based on Fourier IR spectra (Bruker).

2.5. Assessing of sample's anti- staining ability

The anti-staining ability of the sample was identified by the loss of color of a coffee and tea dirty mark, when it was applied on the surface of the raw fabric and finishing fabric, after 0, 30, 240, 480, and 1,200 min in natural light.

2.6. UPF measurement

To evaluate the anti-UV ability on fabrics, we measure the index UPF. The UPF value criteria of the cotton fabric specimens were tested on the reliable international standard: AS/NZS 4399: 1996. UV-VIS Spectronic Camspec M550 is the device used to measure the absorbance of a solution or the transmittance of a fabric:

$$UPF = \frac{\sum_{800}^{400} E_\lambda.S_\lambda.\Delta_\lambda}{\sum_{800}^{400} E_\lambda.S_\lambda.T_\lambda\Delta_\lambda}$$

E_λ: Relative erythrocyte spectral effect;
S_λ: Solar irradiance [see solar irradiance table ($W/cm^2/nm$) for wavelength (nm)];
T_λ: Mean transparency of the test specimen (measured);
Δ_λ: Wavelength value (nm).

3. Results

3.1. Evaluation of the stain resistance of fabric samples

The water absorption ability of the fabric samples was investigated after 0 h and 30 min in the sun and then the ability of stains to fade after 0, 4, 8, and 20 h was investigated.

Hydrophilic and hydrophobic ability in Tables 2, 3: When both stains are dripped onto the untreated cotton fabric, the

TABLE 2 | Investigation of the water absorption and anti-staining ability of the fabric samples for coffee stains.

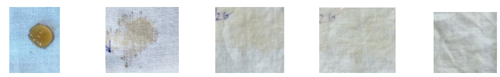
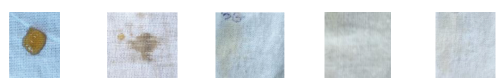
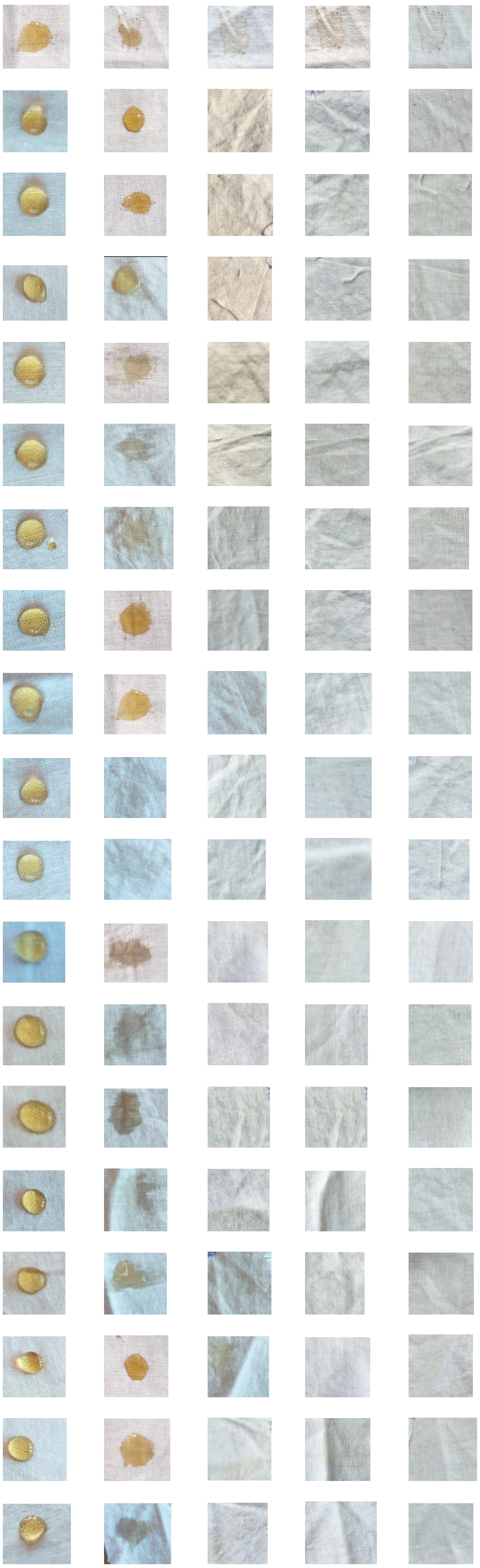
Sample code	0 h	0.5 h	4 h	8 h	20 h
C0					
NĐ1					
NĐ2					
NĐ3					
NĐ4					
NĐ5					
F1					
F2					
F3					
S1					
S2					
S3					
S4					
S5					
TG1					
TG2					
TG3					
TG4					
TG5					

TABLE 3 | Investigation of the water absorption and anti-staining ability of the fabric samples for tea stains.



Sample code	0 h	0.5 h	4 h	8 h	20 h
C0					
NĐ1					
NĐ2					
NĐ3					
NĐ4					
NĐ5					
F1					
F2					
F3					
S1					
S2					
S3					
S4					
S5					
TG1					
TG2					
TG3					
TG4					
TG5					

TABLE 4 | Evaluation of the stain resistance of fabric samples according to the classification level.

Sample / Stain	C0	NĐ1	NĐ2	NĐ3	NĐ4	NĐ5	E1	E2	E3
Coffee	4	3	3	2	2	1	2	2	3
Tea	4	3	3	2	2	1	2	2	2

TABLE 5 | Evaluation of the stain resistance of fabric samples according to the classification level.

Sample / Stain	T1	T2	T3	T4	T5	TG1	TG2	TG3	TG4	TG5
Coffee	1	1	1	2	2	3	3	2	2	2
Tea	1	1	1	2	2	2	2	1	2	2

water droplet shows signs of spreading to the fabric surface immediately, and the water droplet is almost completely in contact with the fabric surface. For fabrics that have been treated with nano TiO_2 when dripping water, droplets are created to locate on the background of the specimen. Those are consistent with the theoretical basis presented above, and the fabric surface after being coated with TiO_2 film will become a hydrophobic surface. When the specimen was lighted with UV radiation in 30 min, the surface of that unfinished fabric was completely absorbed by the dirt, and the surface of the finished fabric sample became hydrophilic because the photocatalytic properties of nano TiO_2 cause water droplets to spread to the fabric surface.

Regarding the anti-staining ability of the fabric: Fabrics have not been completely treated, and the dirt remains the same after the time of sun exposure. For fabrics finished with nano TiO_2, stains fade over time with sunlight exposure, indicating that the fabric samples are resistant to staining. The stain resistance of tea stains is better than that of coffee because, after 20 h, the coffee stain fabric sample has a slightly yellowish color, while the tea stain fabric sample is as white as the original sample. To evaluate the fabric's ability to resist stains, an assessment of the ability to fade after 20 h is conducted with level creasing as follows: 1: excellent, 2: good; 3: pretty; and 4: no resistance to staining. The results are shown in Tables 4, 5.

For fabric samples when surveying concentrations: ND1 and ND2 (2 and 3 g/L) are resistant to stains but do not completely clean stains; ND3 and ND4 (4.5 g/L) are all rated as 2 (good), and ND5 (6 g/L) is rated as very good at level 1.

Regarding the pressure (pick-up level): The anti-staining ability of the fabric samples is at level 2 (good), so the selected pressing level is in the range of 60–80%, all effective.

Regarding the processing temperature: The processing temperature of the fabric samples T1 (110°C), T2 (120°C),

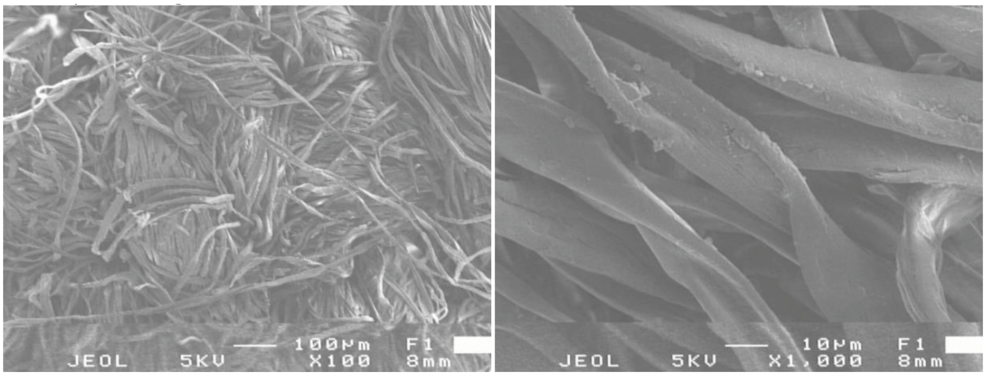


FIGURE 1 | SEM images of the original cotton fabric.

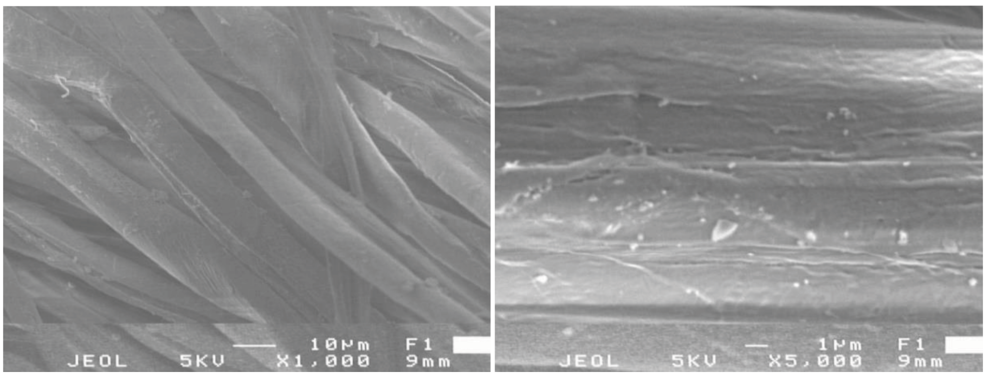


FIGURE 2 | SEM photos of the treated fabric.

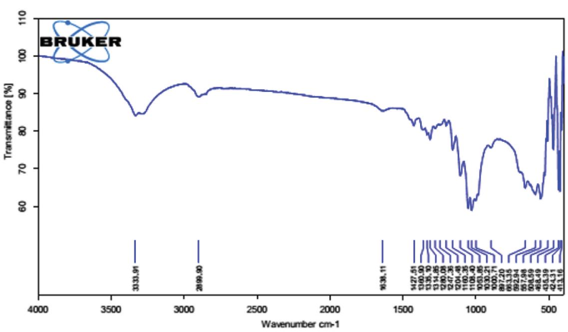


FIGURE 3 | FTIR spectrum of the original cotton fabric.

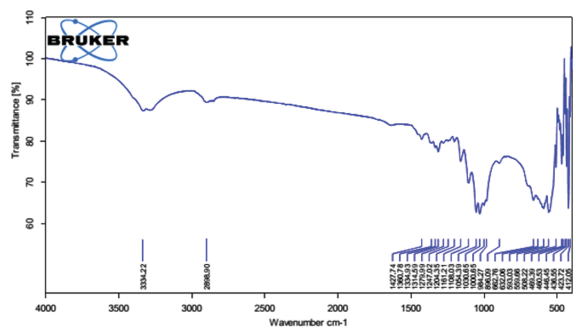


FIGURE 4 | FTIR spectrum of the treated cotton fabric.

and T3 (130°C) is rated at level 1 (very good), so the processing temperature is selected from 110 to 130°C.

Regarding the heat treatment time: Fabric samples TG3, TG4, and TG5 were rated at approximately the same level of 2 (good), but TG3 samples were heat treated for 90 s with better efficiency. Among the above samples, T1 fabric has the best anti-staining ability.

3.2. Evaluating surface morphology of fabric

To clearly identify the fabric structure before and after finishing the treatment with nano TiO_2, the SEM imaging method was used with magnifications of 100×, 1,000×, and 5,000× with resolutions of 100, 10, and 1 µm in Figures 1, 2.

The above photos of the untreated cotton fabric show a clean lint surface. For the SEM images of cotton fabrics that have been finished with nano TiO_2 treatment, there are small nanoparticles that adhere to the fiber surface but do not make the fiber surface to become rough.

3.3. FTIR characterization

Figures 3, 4 show the IR spectrum of original and processed specimens. Both graphs have the same contour. They are

TABLE 6 | Result of measurement of the sun protection index on the fabric (UPF).

No.	Method		Result		
				Sample 1	Sample 2
			First	14.7	17.2
			Second	10.2	19.0
1	UV protection Assessment and classification	AS/NZS 4399: 1996	Third	15.6	18.9
			Fourth	9.5	19.3
			UPF mean	12.5	18.6
			UPF assessed	10	15–20
			UPF classification	–	15
			UVA medium transmittance (%)	6.9	4.9
			UVB medium transmittance (%)	8.0	5.6

TABLE 7 | Tensile durability of the specimen before and after finishing the treatment.

Parameter	Method	Unit	M1	M2
Tensile durability	Nike	kgf	8	7.5

located from 3,000 to 3,600 cm^{-1} corresponding to the oxygen–hydrogen ion oscillation network. The spikes 1,000–1,500 cm^{-1} and the spikes 2,899 cm^{-1} correspond to the C–H ion lattice oscillations. Observing **Figure 4**, we see the presence of Ti–O–Ti bonding ion network corresponding to the wavelength range from 450 to 700 cm^{-1}. The disappearance at peak 1638 cm^{-1} proves that the carbonyl group existing in the original fabric has been bonded to the titanium atom.

3.4. Evaluating the UV resistance of the cotton fabric

With 100% untreated cotton fabric sample (sample 1) and sample T1 (sample 2) having the best anti-staining ability with a size of 15 × 15 cm, the UPF value was measured. The UPF measurement results on the fabric samples are shown in **Table 6**.

From **Table 6**, we observe that sample 1 has not been completely treated with nano TiO_2 with a UPF index of 10, so it has no sun protection, but sample 2 is completely treated with nano TiO_2 with a UPF index of 15–20. rated at a good level for the fabric to be good UV resistance. The difference in the UPF value of the total four measurements is very small, indicating that the chemical is evenly absorbed into the fabric.

3.5. Evaluation of the durability of the specimen

As the fabric must be treated at high temperatures and has a bonding agent, the fabric after the treatment has a lightly reduced mechanical strength in **Table 7**, but the reduction is not significant.

4. Conclusion

This research has found that the quality requirements of technical criteria such as fabric with anti-staining ability and fabric with UV resistance at UPF $\geq$ 15 can be found by analyzing influencing factors, comparing technological factors, and finding the optimal technological parameters for the treatment of anti-UV and anti-staining fabrics by the most common finishing method at factories (dip-pad-dry-cure treatment method). The resistance to UV and staining of fabrics depends on the following four factors investigated: TiO_2 chemical concentration, pick-up level, processing temperature, and treatment time. Among them, the concentration of TiO_2 has a great influence on the results, the second is the processing temperature, the third is the processing time, and finally the pick-up level. When the chemical concentration increases, the anti-staining ability of the fabric increases, and when the treatment time is long enough for the chemical to adhere to the fabric, it is appropriate to increase the concentration to a certain value. If the amount continues to increase, the amount of chemicals on the fabric is close to saturation and only wastes chemicals. When the setting temperature rises, the processing time can be decreased. In addition, reducing the time to keep the fabric in the machine also increases the capacity of the machine and the machine operator and saves energy as the energy to raise the temperature is easily compensated for by the shortening of the time. However, increasing the temperature is also not good for cotton fabrics because above 150°C is the temperature zone that begins to affect the physical and mechanical properties and color of cotton fabrics. Therefore, the processing temperature is selected from 110 to 130°C. The average value of the heat treatment time is to ensure the chemical adhesion to the fabric when processing, so the treatment time should be selected at 90–130 s. The pick-up level of 60–80% gives good results, so the average value is 70%.

Author contributions

HK has written all the content of this manuscript. QT has checked again all formats. Both authors contributed to the manuscript and approved the submitted version.

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