Antibacterial and ultraviolet protective neodymium-doped TiO₂ film coated on polypropylene nonwoven fabric via a sputtering method

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Abstract
A neodymium-doped titanium dioxide film was coated on polypropylene nonwoven fabric via a magnetron sputtering method to prepare multifunctional textiles with antibacterial and ultraviolet protective properties. The antibacterial property of the single-layer titanium dioxide film coated sample was better than that of the single-layer neodymium film coated sample. And that of the three-layer film coated samples was better than that of the double-layer film coated samples, which the latter also was superior to that of the single-layer film coated samples. A longer deposition time of the titanium dioxide film increased the antibacterial rate. The hydrophilicity of the coated samples was improved by ultraviolet irradiation, which improves the catalytic antibacterial ability. The ultraviolet protective factor of the sample that absorbs more ultraviolet is higher, and the water contact angle after ultraviolet irradiation decreases. This improves antibacterial performance. A titanium dioxide film doped with neodymium was improved without any significant change to the morphology and luster of the samples. This can lead to further applications of the coated polypropylene nonwoven fabric with good antibacterial properties.

Keywords
TiO₂ film, magnetron sputtering, Nd film, PP nonwoven fabric, antibacterial property

Introduction
Magnetron sputtering is a physical vapor deposition method. It can deposit thin films at room temperature and is suitable for surface modification of textiles that are not resistant to high temperatures or for coating with thin films to prepare functional textiles.¹,² We have conducted many studies in previous research on the preparation of functional textiles by coating thin films on fabrics surface via magnetron sputtering, which proves that this method is feasible and reliable.³–⁸ Titanium dioxide (TiO₂) has a high refractive index and extinction coefficient. It is widely used in optical thin films, solar energy, and semiconductor photocatalytic antibacterial agents.
due to its non-toxicity and durable antibacterial properties. Coating TiO2 film on textiles can obtain structural coloring, electrical, antibacterial, ultraviolet (UV) protective, photocatalytic, and other properties. TiO2 can be even used to improve the adhesion of nanoparticles on the fabric surface.

TiO2 grains contain a band structure with a band gap between the empty high-energy conduction band (CB) and the electron-filled low-energy valence band (VB). The bandgap energy of anatase TiO2 is 3.2 eV. When TiO2 is exposed to light, the absorbed photon energy, that is, near-ultraviolet light with a wavelength of less than 387.5 nm, is equal to or greater than the band gap energy, electrons in the valence band are excited under the action of an electric field to cross the forbidden band and transition to the conduction band. Negatively charged highly active electrons (e−) are then formed in the conduction band, and positively charged holes (h+) are generated in the valence band at the same time (Figure 1). Electrons and holes migrate to different positions on the surface of TiO2 under the action of an electric field or separated by diffusion to transfer charges to substances absorbed on the surface and interact with dissolved O2 and H2O molecules to cause redox reactions. Electrons (e−) reduce O2 to superoxide radicals −O2−, and holes (h+) oxidize H2O molecules to highly active hydroxyl radicals −OH−, −OOH+, etc. These free radicals can oxidize most organic matter and inorganic pollutants because of their strong oxidizing ability. They can penetrate the cell membrane and destroy the membrane structure, then decompose cells, viruses, and molds, and convert them into small inorganic molecules, that is, CO2, N2, and H2O and other harmless substances. This leads to antibacterial properties and sterilization.

Electrons (e−) and holes (h+) may recombine directly on the surface or inside of the film or be trapped by surface lattice defects thereby reducing their photocatalytic efficiency. The position of the conductive band, the edge of the valence band (band gap), and the redox potential of the adsorbate will affect the rate and probability of electron-hole charge transfer. Therefore, inhibiting or delaying the electron-hole recombination process, decreasing the structural defects associated with oxygen vacancies, and reducing the band gap can improve the photocatalytic performance of TiO2 films. TiO2 has a low light utilization rate due to its narrow photo-response range: It only absorbs near ultraviolet light with a wavelength of less than 387.5 nm. Therefore, broadening the absorption spectrum of TiO2 is better to improving its photocatalytic performance. In addition, increasing the crystallinity of TiO2 is conducive to improving its catalytic performance.

In order to find a better doping amount of Nd, it is necessary to further study the effect of the combined effect of TiO2 and Nd on the antibacterial properties. In this study, polypropylene (PP) nonwoven fabrics were coated as the substrate with films of TiO2, Nd, TiO2/Nd and TiO2/Nd/TiO2 via a magnetron sputtering method. Nd was doped into the TiO2 film in different amount, the photocatalytic activity and antibacterial performance of the Nd-doped TiO2 film were examined. PP nonwoven fabrics are widely used and are commonly used for masks to prevent infection of COVID-19. This research will help expand the application range of PP non-woven fabrics and provide a reference for antibacterial textiles.

**Experimental**

**Materials**

PP hot rolled nonwoven fabric (white, 80 g/m²) was used as the substrate. The fabrics were cut into a circle with a diameter of 5 cm, cleaned with absolute ethanol in an ultrasonic cleaner, and then dried in a hot air-drying box. The targets of titanium dioxide (TiO2) and rare-earth neodymium (Nd) are supplied by China Shenzhen Zhongchengda Target Material Co., Ltd., with a circle of 75 mm, a thickness of 4 mm, and a purity of 99.99%.

**Film preparation**

A magnetron sputtering instrument was used for coating (W500, China Shenyang Scientific Instrument Co., Ltd.). The film was formed by sputtering upward with the substrate fixed on the top and the target installed on the bottom (Figure 2(a)). A single-layer film of TiO2, Nd, a two-layer film of TiO2/Nd, Nd/TiO2, and a three-layer film of TiO2/Nd/TiO2, Nd/TiO2/Nd were coated on PP nonwoven fabrics (Figure 2(b)–(d), respectively). The sputtering time of each single-layer film is shown in Table 1. The base vacuum was set to 5 × 10⁻³ Pa. Argon (Ar) was used to provide a plasma environment.
for protective gas and oxygen (O$_2$) for reactive gas in TiO$_2$ film coating with TiO$_2$ target. The gas flow ratio of Ar and O$_2$ was 35 and 20 ml/min, respectively. In TiO$_2$ film coatings, the sputtering working pressure was 0.5 Pa, and the sputtering power was 100 W in direct current (DC) sputtering. The sputtering power was 80 W and the sputtering working pressure was 1 Pa for a Nd film coating in radiofrequency sputtering. The antibacterial properties and UV protection properties of polypropylene nonwoven fabrics after coating were examined by changing the structure and the relative content of TiO$_2$ and Nd film.

**Testing and characterization**

PP nonwoven fabric is often used for disposable medical and sanitary products. The antibacterial rate of the samples is measured by a quantitative shaking flask method referencing *Sanitary Criteria for Disposable Sanitary Products* (GB15979-2002, China). The test samples were shaken in a speed of 100 r/min within 6 h. The antibacterial rate $R$ (%) was calculated by the bacterial reduction percentage according to formula (1) list below\(^\text{26}\) where $N_A$ and $N_B$ (cfu/ml) were the number of colonies of the sample before and after contacted with the antibacterial agent, respectively.

$$R = \frac{N_A - N_B}{N_A} \times 100\%.$$  

**Figure 2.** Schematic diagram of magnetron sputtering (a) and three kinds of stepped film with single-layer (b), double layer (c), and three-layer (d).

**Table 1.** Film structure and coating time of the samples.

| Samples            | Sputtering time of each layer/s | Sputtering time of TiO$_2$ and Nd/s | Ratio of sputtering time of TiO$_2$ and Nd |
|--------------------|---------------------------------|------------------------------------|------------------------------------------|
| Nd(248)            | 248                             | 0:248                              | –                                        |
| TiO$_2$(248)       | 248                             | 248:0                              | –                                        |
| TiO$_2$(288)       | 288                             | 288:0                              | –                                        |
| TiO$_2$/Nd(30:1)   | 240/8                           | 240:8                              | 30:1                                     |
| TiO$_2$/Nd(5:1)    | 240/48                          | 240:48                             | 5:1                                      |
| Nd/TiO$_2$(30:1)   | 8/240                           | 240:8                              | 30:1                                     |
| Nd/TiO$_2$(5:1)    | 48/240                          | 240:48                             | 5:1                                      |
| TiO$_2$/Nd/TiO$_2$(30:1) | 120/8/120                        | 240:8                              | 30:1                                     |
| TiO$_2$/Nd/TiO$_2$(5:1)| 120/48/120                     | 240:48                             | 5:1                                      |
| Nd/TiO$_2$/Nd(30:1) | 4/240/4                         | 240:8                              | 30:1                                     |
| Nd/TiO$_2$/Nd(5:1) | 24/240/4                        | 240:48                             | 5:1                                      |

**Figure 3.** XRD diffraction spectrum of the reference samples.
(XRD, Rigaku 9000, Japan) was used to analyze the crystalline state of the film on the surface of the samples. An ultraviolet transmission and protection performance tester (NF021, China Ningbo Textile Instrument Co., Ltd.) was used to test the UV absorption and transmission properties of the samples in accordance with the criteria of Assessment of Anti-ultraviolet Performance of Textiles (GB/T18830-2009, China). The radiation of ultraviolet in wavelength A (UV A), wavelength B (UVB), and ultraviolet protection factor (UPF) values were also attained.

Results and discussion

XRD characterization

XRD spectra (22°–50°) show that the samples coated with the outermost layer of TiO₂ has a weak anatase crystal state diffraction peak at 38.3° in line with PDF#89–4921. It confirms that TiO₂ film with a certain crystallinity was successfully coated on the PP nonwoven fabric. The TiO₂ pattern indicating amorphousness because it is very thin by coating. However, the Nd diffraction peak was not obtained, which may be due to its unobvious crystal state.

Antibacterial properties

The antibacterial rates of the pristine samples and the coated samples are shown in Figure 4. The pristine uncoated PP nonwoven fabric has no antibacterial ability. Samples coated with a single layer of Nd or TiO₂ film have a certain antibacterial property. A longer sputtering time of TiO₂ films led to a higher antibacterial rate. The antibacterial rate of sample TiO₂(288) is 19.27% higher than that of sample TiO₂(248), while the sputtering deposition time only increases by 40 s. The average antibacterial rate of sample TiO₂(248) and TiO₂(288) is 28.73%, which is much higher than that of the Nd-coated sample. In other words, the TiO₂-coated samples have better antibacterial properties than the Nd-coated samples.

The antibacterial rate of sample TiO₂/Nd (30:1) is the highest (47.25%) among the two-layer film structure. The average antibacterial rate of sample TiO₂/Nd(30:1) and TiO₂/Nd(5:1) is 38% while the average antibacterial rate of sample Nd/TiO₂(30:1) and Nd/TiO₂(5:1) is 32.5%. In general, the antibacterial properties of the two-layer film coated samples are on average higher than those coated with single-layer film. Similarly, a longer sputtering time of TiO₂ is beneficial to improve the antibacterial properties of the sample. This means that an increase in the proportion of TiO₂ increases the antibacterial effect.

The antibacterial rate is significantly improved after the sample is coated with a three-layer film. The average antibacterial rate of the samples TiO₂/Nd/TiO₂(30:1) and TiO₂/Nd/TiO₂(5:1) is 94.75%, and that of sample Nd/TiO₂/Nd(30:1) and Nd/TiO₂/Nd(5:1) is 42.81%. The results indicate that the antibacterial property of the three-layer composite film is better than that of the two-layer film. After the TiO₂ film is doped with Nd, the Ti⁴⁺ ions may be...
replaced due to the difference in radius between Nd and Ti\(^{4+}\) ions resulting in TiO\(_2\) lattice distortion and an unbalanced charge distribution.\(^{32}\) However, the outer valence band electrons of Nd will capture photo-generated electrons and hinder the recombination of electrons and holes to strengthen the effective separation of electron-hole pairs.\(^{33}\) This in turn improves the antibacterial photocatalytic activity of the composite film.\(^{34}\)

**Hydrophilicity and UV transmission performance**

The pristine uncoated PP fabric does not contain hydrophilic polar groups,\(^{35}\) which implies good water repellency and poor hydrophilicity. A water contact angle of 122° of the pristine uncoated PP fabrics was obtained. Figure 6 shows that the water contact angle of the coated samples (before irradiation) is about 120°–123°. Generally, a contact angle less than 65° implies a hydrophilic surface angle while a contact angle greater than 65° is hydrophobic.\(^{36}\) A smaller water contact angle leads to better hydrophilicity and wettability. Hydrophilic materials generally have strong anti-pollution ability, and bacteria cannot easily adhere to them. Therefore, improving the hydrophilicity of PP fabric samples is beneficial to decrease the protein adsorption on material surface and thus improve the antibacterial performance.\(^{37}\) Studies have shown that the hydrophilicity of TiO\(_2\) films increase after being irradiated with ultraviolet light.\(^{38}\) An ultraviolet lamp was added to the water contact angle testing for UV irradiation in this research (Figure 5). The hydrophilicity and UV transmission performance of the samples was also examined.

The water contact angle of the samples after UV irradiation is reduced by 10° or more except for sample Nd(248) (Figure 6(a)). For reference, sample TiO\(_2\)(248) attained a water contact angle of 117.8° and 107.8° before and after UV irradiation, respectively (Figure 6(b) and (c)). Sample Nd/TiO\(_2\)(30:1) after UV irradiation had a minimum water contact angle of 96.8°. The hydrophilicity of the coated samples improved by UV irradiation, which will improve the catalytic antibacterial ability.

**Figure 5.** UV irradiation during the water contact angle testing.

**Figure 6.** UV transmission performance and water contact angle (a), and sample TiO\(_2\)(248) attained a water contact angle of 107.8° with UV irradiation (b) and 117.8° without UV irradiation (c).
The results also show that the pristine uncoated PP nonwoven fabric has a weak UV protective performance with a UPF of 9.84 as well as a UVA and UVB transmission of 13.12% and 10.7%, respectively. The UV transmission of the coated samples—whether coated with a single-layer, double-layer, or three-layer film—is lower than that of the pristine uncoated sample. It is reflected in the increase in UPF and the decrease in UVA and UVB values. Sample Nd/TiO₂(30:1) has the smallest UV transmission and the largest UPF of 26.83, which means that the film has the largest UV absorption. The hydrophilicity of this sample is most obviously improved after UV irradiation, which may be related to the maximum film thickness of the TiO₂ film coated on the outermost layer of this sample. TiO₂ has been proved a strong absorption and high refraction to UV. After TiO₂ is doped with Nd, the average UPF of the two-layer film coated samples is 19.34, and that of the three-layer film coated samples is 18.7. Nd itself does not have photocatalytic activity. Doping with Nd ions can improve the photocatalysts performance of TiO₂ mainly due to the changes in the surface structure of TiO₂ caused by doping atoms, and its photocatalytic activity varies with the doping amount of Nd within a certain range. Regardless of the influence of film thickness, the UV protection performance of the coated samples are all at a good level (Table 2).

**Surface topography and appearance color**

Coating films with magnetron sputtering might have a bad impact on the surface topography and appearance of the samples. However, the color and surface morphology of the pristine uncoated PP nonwoven fabric and coated samples are quite similar (Figure 7).

Coloring depth (or dyeing depth) is an important indicator to evaluate the dyeing performance of the fabric in textile industry. It is expressed by a K/S value used for color measuring and color matching. The K/S value is simplified by the Kubelka–Munk expression and expressed as follows:

\[ \frac{K}{S} = \frac{A^2}{2(1-A)} = \frac{(1-R)^2}{2R} \]  

Here, \( K \) is the absorption coefficient; \( S \) is the scattering coefficient; \( A \) is the absorption (%); and \( R \) is the reflectivity (%). A larger K/S value implies a darker color. On the contrary, a smaller K/S value implies a lighter color. Therefore, the K/S value is used to express the color depth of the fabric surface because there is a linear relationship between K/S and gloss. Equation (2) also shows that a

**Table 2.** Evaluation criteria for anti-UV performance of textiles.

| UPF range | UVA transmittance/% | Protection level |
|-----------|---------------------|------------------|
| 15–24     | 6.7–4.2             | Good protection  |
| 25–39     | 4.1–2.6             | Very good protection |
| 40–50+    | \( \leq 2.5 \)      | Excellent protection |

Figure 7. Color, topography of reference samples, K/S value, and the relationship curve between K/S and absorptance A or reflectance R: (a) pristine sample, (b) Nd, (c) TiO₂(248), (d) TiO₂/Nd/TiO₂(5:1), (e) K/S, (f) K/S-A, and (g) K/S-R.
larger reflectivity $R$ means a smaller K/S but a smaller $R$ leads to a larger K/S value (Figure 7(g)). Correspondingly, a greater absorption leads to a greater K/S value. Sample TiO$_2$/Nd(30:1) has the largest absorption of ultraviolet light and visible light with the highest K/S and UPF value, which is in line with conclusions drawn from previous analysis.

It can be seen that the $L^*a^*b^*$ values of the pristine sample and the coated samples are relatively concentrated which show in Figure 8, and even difficult to distinguish, indicating that they are very close to each other in terms of color hue and K/S value. Nd-doped TiO$_2$ films were prepared via a sputtering method and were improved without any significant change to the appearance and luster of the substrate textile; thus, this PP non-woven fabric with good antibacterial properties can be smoothly applied for subsequent products.

**Conclusion**

Nd-doped TiO$_2$ films prepared via a magnetron sputtering method had good antibacterial performance. The antibacterial property of the single-layer TiO$_2$ film coated sample is better than that of the single-layer Nd film coated sample. The antibacterial rate of the two-layer film-coated sample is higher than the single-layer film coated sample, while that of the three-layer film coated sample is better than that of the two-layer film coated samples. The relative content of TiO$_2$ and Nd impacts antibacterial performance. An increase in the proportion of TiO$_2$ increases the antibacterial effect. The hydrophilicity of the coated samples improved with UV irradiation, which can improve the catalytic antibacterial ability, which will improve the antibacterial performance. There will be a further possibility of application for PP non-woven fabric with good antibacterial properties by coating Nd-doped TiO$_2$ film.

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