Research article

Pseudomonas aeruginosa antibacterial textile cotton fiber construction based on ZnO–TiO₂ nanorods template

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A B S T R A C T

An alternative method of synthesizing ZnO–TiO₂ nanorods is through route precipitation and sintering at 600 °C. In this study, the introduction of Ti into Zn in the molar ratio Ti:Zn (1:3) produced a composite ZnO-Low TiO₂ (ZnO-LTiO₂) while 1:1 produced ZnO-High TiO₂ (ZnO-HTiO₂). The effect of the Ti introduced on the antibacterial properties of ZnO–TiO₂ nanorods was investigated with the product structure characterized by XRD and the optimal intensity at 2θ: 31.72°, 34.37°, 36.19° showed a Wurzite structure and a crystal size of 35.8–41.5 nm. The average pore diameters for ZnO-LTiO₂ and ZnO-HTiO₂ were around 5.159 nm and 6.828 nm while the surface areas were 15.692 m²/g and 15.421 m²/g respectively. The anti-bacterial textile fiber construction was prepared using dip-spin coating with the application of an adipic acid crosslinker for 6 h and stable coating up to 10 times washing. The improvement of Pseudomonas aeruginosa (Pa) antibacterial properties in the textiles with coating had an inhibition zone of 20.5–25.0 mm and 16.2 mm without the coating. The elements of the cotton fiber construction include C at 54.60%, O at 40.89%, Ti at 0.81% and Zn at 2.60% while the TG-DTA analysis conducted showed an increase in the heat stability of the textile fibers to a temperature of 400 °C, after which the textiles were modified by coating ZnO–TiO₂ nanorods. The findings of this research could be successfully applied to improve the antibacterial properties of textiles.

1. Introduction

Textiles are used as body armor, both indoors and outdoors, and are very important to protect direct body contact with hot and cold air. However, in order to produce contamination-free and healthy textiles for human skin, researchers have collaborated with industries to create textile fibers with superior physical, chemical, and biological properties, and which can be used in several aspects of life (Saba et al., 2014; Shabbir et al., 2017; Harifi and Montazer., 2017; Patra et al., 2013; Rilda et al., 2015). Moreover, the application of textiles in fashion, decoration, medical sector, military, sports, and hospitals has also led to the study of its anti-microbial preparation by several researchers in recent years (Das et al., 2019; Pandey and Verma., 2019; Sadeghi-Kiakhani et al., 2018; Shahid et al., 2019; Sharma et al., 2019; Rilda et al., 2016). It is also important to note that some other textiles are being equipped with anti-stain and anti-heat functions for more effectiveness and applicability (Rilda et al., 2015; 2019; Wang et al., 2011).

Textile cotton fibers contain natural fibers with high humidity and ability to adjust the body temperature and this increases their vulnerability to microbial contamination. Therefore, there is the need to improve the quality of the fibers by modifying them to overcome the communal problem of infection associated with some heat-resistant pathogenic bacteria. As a result, metal oxide nanoparticles with sizes ranging from 1 to 100 nm have been recommended as additives to modify the antibacterial functions of textiles and an example of this is the TiO₂ nano-materials which have been widely tested for superiority in several applications (Tang et al., 2016; Yu et al., 2016). The use of these compounds as additives is very appropriate because they are photocatalysts and have contributed to microbial clearance with 90% efficiency (Rilda et al., 2019).

The use of zinc oxide (ZnO) as an additive to modify the construction of textile fibers has several advantages considering its higher photocatalyst activity in visible areas, commercial availability, non-toxicity, and environmental friendliness (Karthik et al., 2017; Noorian et al., 2019; ZnO).

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2019; Roman et al., 2019). It also has the potential to provide specificity in modifying textile cotton fibers due to its unique rod pattern (Aladpoosh and Montazer, 2015). Therefore, this study modified the structure of the ZnO by forming composites through the hybridization of doped TiO2 and Chitosan to optimize its ability to improve the physical, chemical and mechanical properties of textile cotton (Deepthi et al., 2016; Morlando et al., 2018; Shabbir et al., 2017; Thirumalraj et al., 2017; Xu and Cai, 2008). The main focus was on the improvement of the antibacterial properties of the textiles.

2. Experiment

2.1. Material

All the chemicals (Table 1) were used as received without further purification while all the water used was distilled.

2.2. Preparation of ZnO–TiO2

The Zn(NO3)2·4H2O sample used was measured to be 2.72 g, dissolved in 100 ml aqua dest, and homogenized for 15 min at 60°C while the pH of the solution was adjusted to 11.0 using 20 ml NaOH 2M. This was followed by the addition of methylene compound as nanorods template and later homogenized for 3 h at 60°C. Moreover, 0.81 g of ZnO seed suspension was added and everything was stirred for 2 h while the TiO2 suspension, as a doped material, was prepared from the Titanium Isopropoxide (TIP). After this, each suspension was mixed with a variation of the TiO2 composition to the precursor ZnO with a concentration of 0.03M at 1:3 and 0.10M at 1:1 and marked with the symbol LTiO2 (Low TiO2) and HTiO2 (High TiO2) respectively (Rilda et al., 2019). Furthermore, Chitosan and CTAB were used as a template controller for porosity and nanorod growth with the mixture homogeneous for 6 h (Rilda et al., 2019). ZnO–TiO2 powder was obtained after calcination at 600°C for 6 h and characterized by XRD, SEM-EDX, BET/BJH, and TG-DTA analyses.

2.3. Coating of ZnO–TiO2

A piece of textile measured to be 8 × 8 cm was dewaxed with a solution of 3.7 × 10−3M a2CO3 and dried at 80°C for 15 min. Furthermore, cross-linkers were conducted on the textile cotton with 1.5M adipic acid for 6 h at room temperature with the addition of NaH2PO4 as a catalyst in a molar ratio of 5:3. The textile cotton was dried at 80°C for 15 min and this was followed by the ZnO–TiO2 coating process.

2.4. Characterization

The powder X-ray diffraction (XRD) using an XPERT MPD-PRO diffractometer (Cu Kr radiation) accelerating at 45 Kv and applied current of 40 mA for the characterization of crystal structures. The Bragg law \( \lambda = 1.5406 \) Å and Fourier Transform Infrared (FT-IR) spectra as KBR (JASCO 4100 type), wavenumber range (600–4000 cm⁻¹) Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-Ray Spectroscopy (EDX) were used for the morphological determination and elemental analysis of the prepared nanoparticles, combined with Brunauer-Emmett-Teller/Barrett-Joyner-Halenda (BET/BJH), and Thermal Gravimetric - Differential Thermo Analysis (TG-DTA).

2.5. Antibacterial test

The antibacterial textile test was qualitatively conducted using the diffusion method based on the measurement of the clear zone, the ability of the tested bacteria to grow. The cotton textile was coated at 0.6 cm diameter, positioned on a Petri dish containing sterile NA media, and inoculated with the Pa bacterium. The Petri dish plates were incubated in a UV/Vis aerobic incubator for 24 h and the intensity was monitored using a Blue Light Safety Detector UV detector with an intensity of 536 Lux from a distance of 15 cm at a vertical position above the Petri dish surface. Moreover, the zone of inhibition around the textile cotton disk was measured at 2 cross-sectional points and the average size was evaluated using the diameter of the inhibition zone in millimeters to describe the ability of ZnO–TiO2 to inhibit the bacteria.
Figure 2. $\text{N}_2$ adsorption-desorption isotherm of the ZnO-LTiO$_2$ (a), ZnO-HTiO$_2$ (b). Inset the corresponding pore size distribution of ZnO-TiO$_2$. 
on the calculation from Debye-Scherer’s equation, ZnO crystal size was found to be 35.6 nm while ZnO-HTiO2 and ZnO-HTiO2 had a crystal size of 35.8 nm and 41.5 nm respectively. This means the presence of TiO2 oxide in ZnO–TiO2 composites was able to increase the size of the crystals.

3.2. Analysis of BET/BJH

BET/BJH analysis was used to evaluate the surface area and pore volume size of ZnO–TiO2 nanomaterials as shown in Figure 2. Based on the adsorption-desorption nitrogen gas, the absorption pattern was at 77°K temperature on the surface of the material, measured as surface area and pore size distribution with BJH of ZnO-LTiO2 (Figure 2a) and ZnO–HTiO2 (Figure 2b) respectively. According to the IUPAC classification, the loop observed is considered a hysteresis loop with type H3 and this indicates the presence of excessive pores (Pandimurugan and Thambidurai, 2017, 2014).

The average pore diameters for ZnO-LTiO2 and ZnO-HTiO2 were estimated to be 5.159 nm and 6.828 nm while the surface areas were 15.692 m²/g and 15.421 m²/g respectively. The single-phase crystal and porous properties of ZnO–TiO2 allowed this material to provide multifunctional properties in the textile fibers.

3.3. Analysis of TG-DTA

Thermal analysis of uncoated and coated cotton fabrics was conducted under a nitrogen atmosphere to study the effect of nanoparticles on their thermal properties as shown in Figure 6.

On the TG-DTA curve, the reduction in mass of the textile fiber was estimated to be 5% when the temperature was lower than 200 °C mainly due to the free water volatilization binding water to cotton fibers. Moreover, the decomposition temperature of cellulose has been reported to be 380–580 °C (Lin et al., 2018; Nabil et al., 2018). The contaminants in cellulose (Figure 6x) and 7.2% reduction in mass observed with a degraded carbon residue at 580 °C. Simultaneously, the initial decomposition temperature of the coated cotton cloth was 334.6 °C while the carbon residue at 580 °C was 15.8%. These results showed ZnO-LTiO2 coated cotton fiber has a lower

Figure 3. FT-IR spectra of cotton fabrics (a), crosslink (citric acid) coated cotton fabrics (b), citric acid and ZnO-LTiO2 (c), citric acid and ZnO-HTiO2 coated cotton fabrics (d).

Figure 4. The FT-IR spectra of ZnO (a), ZnO–TiO2 (3:1) (b), and ZnO–TiO2 (1:1) (c).

3. Result and discussion

3.1. Analysis of XRD (X-ray diffraction)

Figure 1 shows the XRD pattern of ZnO and ZnO–TiO2 with the analysis conducted using standards (JCPDS no. 36–1451). Moreover, the structure of the ZnO nanorods was wurtzite as indicated by nine main peaks at (100) (002) (101) (110) (103) (200) (112) and (201), and categorized as hexagonal geometry (Yu et al., 2016).

In this study, the ZnO–TiO2 was synthesized with different Zn and Ti molar ratios to observe the effect of each composition in shaping antibacterial textile construction. ZnO was found to have a higher crystallinity level at 2θ while none was observed for TiO2. Furthermore, the TiO2 nanoparticles doped on ZnO were assumed to have formed composites and combined with the ZnO–TiO2 intensity. The doping effect of TiO2 was observed to have the ability to reduce the corrosive nature of ZnO due to its usual negative impact on fiber properties. Moreover, based

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The average pore diameters for ZnO-LTiO2 and ZnO-HTiO2 were estimated to be 5.159 nm and 6.828 nm while the surface areas were 15.692 m²/g and 15.421 m²/g respectively. The single-phase crystal and porous properties of ZnO–TiO2 allowed this material to provide multifunctional properties in the textile fibers.

3.3. Analysis of FT-IR

The textile fiber construction interaction was characterized by FT-IR while the vibrational spectrum showed the specific peak of intramolecular interactions. FT-IR spectrum analysis clearly revealed the absorption peak with strong intensity at 3318 cm⁻¹ corresponded to the vibration stretching –OH in cotton fiber cellulose (Figure 3).

The weaker peaks at 2902 cm⁻¹ and 1371 cm⁻¹ were associated with vibrations of C–H stretching and C–H bending of cellulose respectively while weak peak at around 1600-1700 cm⁻¹ was associated with C=O asymmetric stretching vibrations of cellulose (Yu et al., 2019). Furthermore, Figure 3 showed 3a cotton fabric, 3b change in the intensity of the C=O peak of coated cross-linker (citrac acid) coated textiles and (c) coated citric acid and ZnO-LTiO2 and (d) coated citric acid and ZnO-HTiO2 experienced changes in intensity and shifted in smaller wavenumbers. A significant increase in intensity occurred in textiles coated with Adipic Acid crosslinker due to the interaction observed between the hydroxyl groups-adipic acid and nanorods after the coating. These indications were marked by a drastic decrease in intensity but controlled after the nanorods were coated leading to significant intensity shift with the peak towards the smaller wavenumbers called the redshift. Therefore, it can be said that ZnO–TiO2 nanoparticle-coated cotton fibers were formed. Figure 4 shows the cotton fibers coated with different compositions, namely ZnO-LTiO2 and ZnO-HTiO2, significantly not showing changes in the C=O peak. An understanding of the mechanism of covalent ester interaction between functional groups shown in Figure 5.

3.4. Analysis of TG-DTA

Thermal analysis of uncoated and coated cotton fabrics was conducted under a nitrogen atmosphere to study the effect of nanoparticles on their thermal properties as shown in Figure 6.

On the TG-DTA curve, the reduction in mass of the textile fiber was estimated to be 5% when the temperature was lower than 200 °C mainly due to the free water volatilization binding water to cotton fibers. Moreover, the decomposition temperature of cellulose has been reported to be 300–380 °C (Lin et al., 2018; Nabil et al., 2018). The contaminants and temperatures were found to be 338.3 °C as shown in (Figure 6x) and 7.2% reduction in mass observed with a degraded carbon residue at 580 °C. Simultaneously, the initial decomposition temperature of the coated cotton cloth was 334.6 °C while the carbon residue at 580 °C was 15.8%. These results showed ZnO-LTiO2 coated cotton fiber has a lower
initial decomposition temperature and a higher carbon residue (Gao et al., 2018). Moreover, the slight change in the maximum degradative peak temperature observed with the increase in residual percent also confirms the composite coating did not affect the degradation mechanism nor interfere with the thermal stability of nanoparticle-coated cotton fabrics (Zhang et al., 2018). However, the DTA curve shows the rate of decomposition of uncoated (Figure 6a) and coated cotton increased sharply between \(370^\circ C\) and \(428^\circ C\). This means there is a drastic decrease for both materials but the cotton-coated fabric was lower and constant.

3.5. Analysis of SEM-EDX

The coatings of the ZnO–TiO\(_2\) nanorods for the textile cotton fibers were homogeneously and evenly distributed using adipic acid cross-linkers. This process interacted in the covalent esters between textile cellulose fibers functional groups - adipic acid cross-linkers and ZnO–TiO\(_2\) to form a textile construction with superior physicochemical properties. The coatings were optimized as indicated by the increase in the mass of the textile dip-spin coating method. Furthermore, SEM-EDX analysis showed the morphology of the fibers with an even distribution of nanoparticles while the EDX proved the ZnO-LTIO\(_2\) coating process based on the composition detected as shown in Figure 7. It is important to note that the compound contains four main elements and they include Carbon (C) at 54.60\%, Oxygen (O) at 40.89\%, Ti at 0.81\%, and Zn at 2.60\% (Zhong et al., 2017). The surface morphology of the coated and uncoated cotton fibers observed with the SEM are shown in Figure 8 and ZnO–TiO\(_2\) particles were discovered to be scattered throughout the cotton fibers (Figure 9).

3.6. Antibacterial activity

The efficiency of ZnO–TiO\(_2\) nanorods used in the construction of Pa antibacterial textile fibers was measured by assessing the inhibition zones 24 h after the UV/Vis incubation. Figure 10 shows an increase in the difference of inhibition zones between coated and uncoated textile fibers. Furthermore, the presence of ZnO–TiO\(_2\) nanorods was able to optimize the antibacterial properties of the fiber due to an increase in photocatalytic activity in the composites which can be related to the amount of OH radicals formed by the photocatalysts. It was also reported
that ROS species and OH radicals had high oxidizing potential to destroy bacterial cells more effectively (Sethi and Sakthivel, 2017).

The increased photocatalytic activity of the mixed nanoparticles, ZnO-TiO$_2$, was also attributed to the interface reaction produced between ZnO and TiO$_2$ to provide a more active side in the photocatalysis process (Qi et al., 2017). Moreover, Zhong et al. (2017) reported the improved interface structure in hybrid nanostructures facilitated the process of charge transfer and optimized spatial separation of photogeneration charge carriers to increase the photocatalytic activity. Moreover, in the hybrid semiconductor structure, ZnO-TiO$_2$, heterojunction assisted in transferring electrons from the conduction band (CB) of ZnO to CB of TiO$_2$ and hole of the valence band (VB) of TiO$_2$ to VB of ZnO. The data in Table 2 shows the differences in the uncoated textile cotton did not produce inhibition zones while the ones coated with ZnO-LTiO$_2$ and ZnO-HTiO$_2$ had an antibacterial activity with inhibition zones of 20.5 and 20.0 mm respectively as well as a positive control of 22.0 mm.
4. Conclusion

The physicochemical characterization results showed the crystalline structure, surface area, and morphological properties of the ZnO–TiO$_2$ were strongly influenced by precipitation preparation method as well as low and high TiO$_2$. The XRD analysis found the Zinc Oxide has a nano-crystal with wurtzite hexagonal structures. ZnO–TiO$_2$ was coated on cotton cloth using a dip-spin method while the surface morphology and chemical compositions were confirmed using SEM and EDX analyses. The inhibition zones were observed not to be very different from the positive control 22.0 mm. Therefore, the coated cotton fabric has an antibacterial ability against gram (+) bacteria, namely *Pseudomonas aeruginosa* (Pa), which is pathogenic, where the ZnO-LTiO$_2$ inhibitory ability is greater than ZnO-HTiO$_2$, had an antibacterial activity with inhibition zones of 20.5 and 20.0 mm respectively, as well as a positive control of 22.0 mm.

### Table 2. Zone of inhibition (mm) of each sample against PA.

| Sample                               | Inhibition Zone (mm) |
|--------------------------------------|----------------------|
| Textile Cotton Fiber without treatment | -                    |
| Positive control of Clarithromycin    | 22.0                 |
| ZnO                                  | 16.2                 |
| ZnO-LTiO$_2$                         | 20.5                 |
| ZnO-HTiO$_2$                         | 20.0                 |

### Declarations

#### Author contribution statement

Yetria Rilda: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Doni Damara: Analyzed and interpreted the data.

Anthoni Agustien: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data.

Refinel Refinel, Yulia Eka Putri: Contributed reagents, materials, analysis tools or data.

Hilfi Pardi: Analyzed and interpreted the data; Wrote the paper.

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#### Competing interest statement

The authors declare no conflict of interest.

#### Additional information

No additional information is available for this paper.
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