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Synthesis of titanium dioxide-silica-silver composites using a base catalyst as active antibacterial compound coated on the cotton fabric

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Abstract. Titanium dioxide is a photocatalyst material that can inhibit the growth of microorganisms, bacteria, and viruses. Titanium dioxide can be modified into TiO₂-SiO₂-Ag composites to improve the ability to inhibit the growth of microorganisms due to increased adsorption capability and bactericidal properties. Composites of TiO₂-SiO₂-Ag were synthesized using tetraethylorthosilicate (TEOS) precursor in the presence of NH₄OH base catalyst followed by the addition of TiCl₄ and AgNO₃ precursors. Coating of composite material on the cotton fabric was carried out by eight times dyeing. The coated cotton fabric was tested for antibacterial ability using UV-vis spectrophotometry method and agar disc diffusion method. FTIR data showed a typical functional group of the materials composing the composite. Analysis of XRD and UV-DRS showed that the composite material was amorphous with a bandgap of 2.43 eV. Antibacterial test results showed that the TiO₂-SiO₂-Ag composite material was able to inhibit the growth of *Escherichia coli* gram-negative bacteria in liquid media and solid media by 83.76% and 99%, respectively.

1. Introduction

Cotton is fabric that is widely chosen as a material for various types of clothes, especially by people who live in tropical countries. Cotton fabric is chosen because it has a soft texture, good air circulation, easy to be formed and sewn, and high sweat absorption. However, the cotton fabric also has drawbacks because this fabric is a very good medium for the growth of microorganisms/bacteria so that it creates odor affecting to the user.

Titanium dioxide (TiO₂) is the most commonly used semiconductor as a photocatalyst for photodegradation of various organic materials (Kusnadi et al., 2012). TiO₂ has been studied to inhibit the growth of microorganisms and bacteria (Hoffmann et al., 1995, Linslebigler et al., 1995, Gunlazuardi et al., 2001).

Silver (Ag) material is also reported to have the high antibacterial ability. Panacek et al. (2006) reported that Ag materials have high antibacterial and antimicrobial properties against gram-positive *S. aureus* and gram-negative *E. coli* bacteria. Ag particles are toxic to bacterial cells. Haryono et al. (2010) carried out the preparation of Ag colloidal particles with trisodium citrate reducing agent without stabilizers and coating the cotton fibers so that they had the ability as antibacterial.



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Meanwhile, silica is reported to have the good capability as a photocatalyst material carrier compound and has good ability to bond with fabric fibers. Synthesis of amorphous silica nanoparticles through a facile and economic sol-gel method has been reported by Zawrah et al. (2009). While Helbakar et al. (2011) synthesized silica/TiO₂/Ag composite through a sol-gel method in the absence of a catalyst, the composite material was then coated on cotton fabric, and it can bond with fabric fibers better than without using silica. Also, fiber fabrics coated with composite materials are reported to have self-cleaning and antibacterial activity.

The role of silica as a composite material to give a good binding effect on fabric fibers is important in the manufacture of antibacterial material coated fabrics, hence silica synthesis is something that needs to be considered. The presence of a catalyst is expected to accelerate the synthesis of silica gel. In this article, the synthesis and characterization of silica/TiO₂/Ag composite using the sol-gel method in the presence of ammonium hydroxide base catalyst is studied. Also, a coating of composite materials on cotton fabrics and their ability as antibacterials are also reported.

2. Experimental

2.1. Materials and reagents

Cotton fabric was bought from the general market. Titanium (IV) chloride, ethanol, ammonia (14.5%), tetraethyl orthosilicate, silver nitrate, agar and broth nutrients were Merck p reagents and used as received. Distilled water was used in all experiments.

2.2. Synthesis of silica using a base catalyst

A total of 20 mL of TEOS solution and 25 mL of ethanol were stirred, and then 6.4 mL of distilled water was added. Then NH₄OH was added dropwise until the solution reached pH 9. The mixture was stirred until homogeneous, and the solution was left at room temperature to obtain a solid gel. The solid gel was then heated in an oven at a temperature of 80 °C for 18 hours. The gel obtained was then washed with distilled water until it reached neutral pH and reheated at 100 °C for 2 hours then crushed to a fine size.

2.3. Synthesis of silica/TiO₂/Ag composite

Synthesis of silica and TiCl₄ precursors with a composition ratio of 0.16 M: 2.28 M were mixed in distilled water for 3 hours and then allowed to stand for 12 hours. The precipitate obtained was washed with distilled water to free Cl⁻ ion. Then AgNO₃ was added to the silica-TiO₂ solids and stirred for 2 hours and dried in an oven with a temperature of 100 °C for 2 hours to form a composite solid. FTIR, XRD, and UV-DRS characterized the resulting composites.

2.4. The composite coating on the cotton fabric

Cotton fabric was cut to the size of 6×3 cm² and 2×2 cm² as much as two pieces each. The cloth was then dipped in a composite solution eight times. Each size was taken half to dry by aeration and the rest to be dried by the oven.

2.5. Antibacterial test against *E. coli* in solid media of nutrient agar

Agar nutrient media were prepared by adding 5.6 g of agar nutrient in 100 mL of distilled water, and the mixture was autoclave sterilized for 20 minutes. The agar nutrient was then poured into four Petri dish and cooled at room temperature. The first Petri dish contained a cotton cloth without any treatment (negative control), the second Petri dish contained a compositely coated cotton cloth with aeration drying, the third Petri dish containing oven-coated composite cotton cloth and the fourth Petri dish containing cotton cloth dipped in tetracycline (as a positive control). *E. coli* bacteria then planted evenly using spider into each Petri dish. Antibacterial activity was measured based on clear zones formed after incubation for 24 hours at 37 °C.

2.6. Antibacterial test against *E. coli* in liquid media of nutrient broth

A total of 0.8 g of nutrient broth was added to 100 mL of distilled water and sterilized in an autoclave for 20 minutes. After that, the nutrient broth media was cooled at room temperature. A total of 10 mL of bacterial stock solution was added to 5 Erlenmeyer containing 100 mL of nutrient broth, and then each mixture was incubated for 24 hours at 37 °C. After that, the initial absorbance measurements were carried out using a UV-Vis spectrophotometer at a wavelength of 600 nm. Then into each of the five erlenmeyers were inserted cotton cloth without composite coated as a negative control (Erlenmeyer 1), compositely coated cotton cloth drying aeration (Erlenmeyer 2), compositely coated cotton cloth drying oven (Erlenmeyer 3) and tetracycline coated cotton cloth as a control positive (Erlenmeyer 4). Furthermore, all erlenmeyers were incubated for 24 hours at 37 °C and the absorbance were measured using a UV-Vis spectrophotometer at a wavelength of 600 nm.

3. Results and discussion

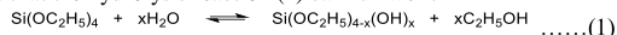
3.1. Synthesis of silica using a base catalyst

This synthesis uses the sol-gel method with TEOS precursor and ammonium hydroxide base catalyst using a mixture of distilled water and ethanol. The process of changing the appearance of silica gel to xerogel with a temperature treatment is shown in Figure 1.



Figure 1. Silica gel (left), silica gel after heating at a temperature of 80 °C (middle) and xerogel product after heating at a temperature of 100 °C (right).

In this synthesis process, ethanol is also used as a solvent with the aim that TEOS can mix well with the solution so that the hydrolysis reaction (1) can run well.



The NH_4OH catalyst was used to accelerate the rate of hydrolysis reaction via the mechanism as shown in Figure 2. After that, a condensation reaction occurs to form silanol as shown in reactions (2) and (3). This silanol will react to form polymers through the Si-O-Si bond forming a sol which will then aggregate to form a gel (Figure 2, left). Heating at a temperature of 80 °C aims to remove ethanol to produce a silica gel solid (Figure 2, middle) which is then continued heating at a temperature of 100 °C to remove water to produce xerogel (Figure 2, right).

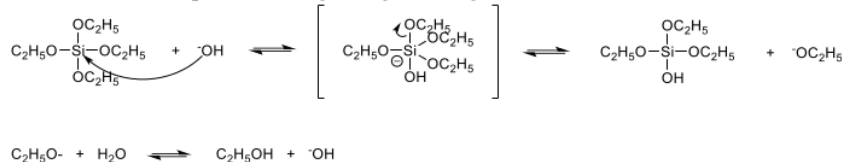


Figure 2. Mechanism of hydrolysis reaction in the presence of a base catalyst.

3.2. Synthesis of silica/TiO₂/Ag composite

Titanium (IV) chloride (TiCl_4) was used as a precursor in the formation of TiO_2 . Mixing silica powder and TiCl_4 with a ratio of 0.16 M and 2.28 M produced a white suspension. The resulting suspension was washed with distilled water until it is free of chloride ions (Cl^-) tested by using AgNO_3 . The

resulting silica/TiO₂ composite is white (Figure 3, left). Silica/TiO₂/Ag composite was obtained through the addition of AgNO₃ to silica/TiO₂ composite until it became homogeneous which then the mixture was dried using an oven at 100 °C to form black solid (Figure 3, right).

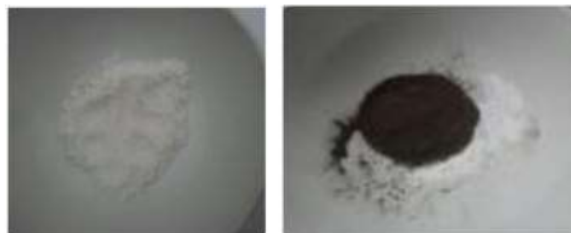


Figure 3. Appearance of silica/TiO₂ (left) and silica/TiO₂/Ag (right) composites.

Silica/TiO₂/Ag composites were characterized using FTIR to determine functional groups found in composite materials. Characterization results using FTIR are shown in Figure 4(a). FTIR data shows that there are absorptions at the wavenumber of 1103.28 cm⁻¹ which is the Si-O stretching vibration of Si-O-Si and at 1200 cm⁻¹ which is the vibration character of SiO₄ which shows that there has been a polymerization process. The absorption band at 3448.72 cm⁻¹ is the vibration absorption of -OH groups derived from Si-OH. The appearance of absorption at wavenumbers 1408.18 cm⁻¹ and 2337.72 cm⁻¹ which are bending vibrations of Si-O-Ti and Ti-O-Ti, respectively, indicates that TiO₂ has entered the silica polymer matrix. Meanwhile, the appearance of absorption at wavenumber 550 cm⁻¹ shows the stretching vibration of Ag-O-Ag which proves that Ag has formed on the matrix of composite material.

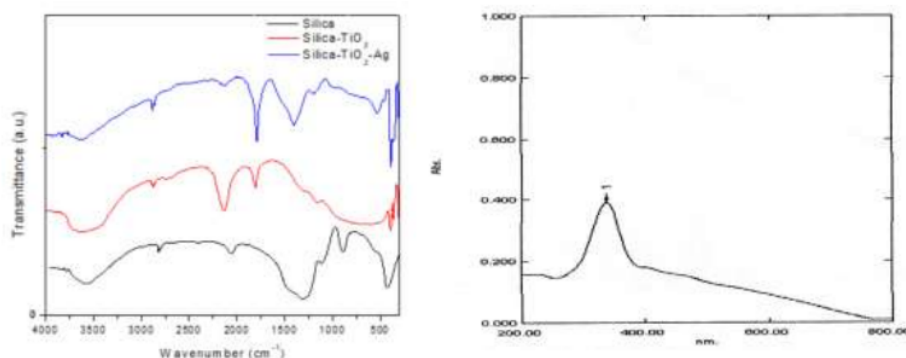


Figure 4. (a) FTIR spectra of silica (black line), silica/TiO₂ composite (red line) and Silica/TiO₂/Ag composite (blue line) and (b) DRUV spectra of Silica/TiO₂/Ag composite.

Characterization using DRUV aims to determine the energy gap (E_g) of the composite material. DRUV spectra data is processed with the Kubelka-Munk equation to obtain the energy gap of 2.4 eV. This energy gap is lower than the original TiO₂ energy gap (3.2 eV) which is caused by the Ag dopant which causes a new band to appear between the conduction band and the valence band so that the energy gap becomes smaller.

The composite material was also characterized using XRD to determine its crystal structure. The XRD characterization result is shown in Figure 5. The diffractogram data shows amorphous characters appearing from the obtained silica. It is clear that the amorphous characters of silica cover the crystal peaks of TiO₂ and Ag. The same results were also reported by Zawrah et al. (2009).

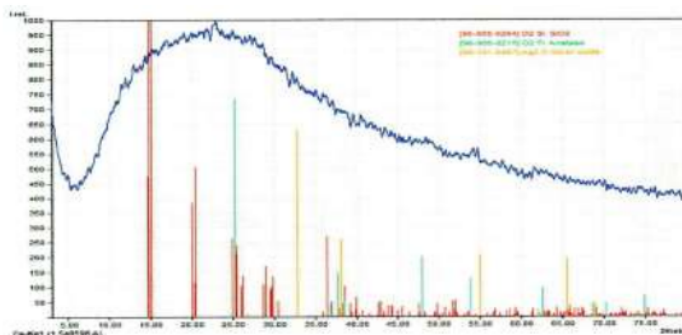


Figure 5. Diffractogram of Silica/TiO₂/Ag composite.

The composite coating on cotton fabric and its antibacterial test against E. coli in liquid media nutrient broth and solid media nutrient agar

A cotton cloth dipped in a composite solution that is dried by aeration and by oven have a similar appearance that changes from white (original cotton fabric) to black (Figure 6). This color change indicates that the fabric has been coated with composite material.

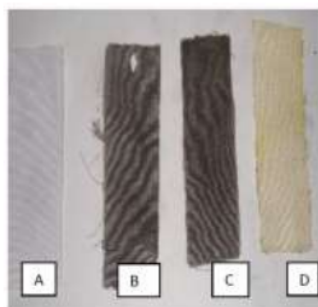


Figure 6. The appearance of (a) non coated fabric (negative control), (b) coated fabric dried by aeration, (c) coated fabric dried by the oven, and (d) fabric coated with positive tetracycline control).

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Figure 7 shows the antibacterial activity of coated cotton fabrics with composite materials on nutrient broth media and nutrient agar. Data shows that the coated cotton fabric of composite material has the ability to inhibit the growth of *E. coli* bacteria. The difference in drying method, both through aeration and oven, does not give a difference to the inhibitory ability in liquid media. While the solid media (nutrient agar) obtained data that drying with aeration provides greater inhibitory power than oven drying. Antibacterial test results showed that the cotton fabrics coated with TiO₂-SiO₂-Ag composite material were able to inhibit the growth of *E. coli* gram-negative bacteria in liquid media and in solid media by 83.76% and 99%, respectively.

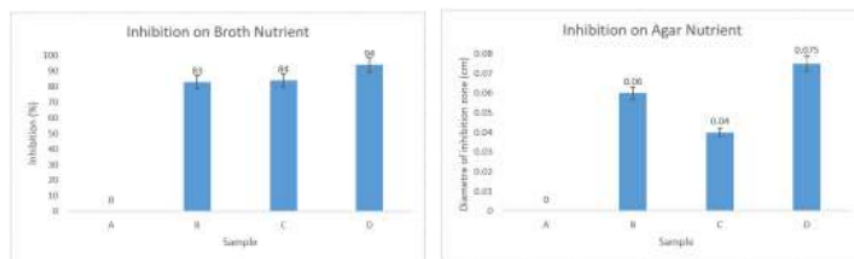


Figure 7. Inhibition on broth nutrient (left) and agar nutrient (right), where: (a) non coated fabric (negative control), (b) coated fabric dried by aeration, (c) coated fabric dried by the oven, dan (d) fabric coated with tetracycline (positive control)

4. Conclusion

Silica/TiO₂/Ag composite has been successfully synthesized by a sol-gel method, and the presence of base catalyst produces black and amorphous solid and has a bandgap powder material of 2.43 eV. Antibacterial test results showed that the cotton fabrics coated with TiO₂-SiO₂-Ag composite material were able to inhibit the growth of *E. coli* gram-negative bacteria in liquid media and solid media by 83.76% and 99%, respectively.

Acknowledgment

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