#### Article

# **Optimum Calcination Temperature In Titanium Dioxide (TiO2) Photocatalyst Coating For Stain-Resistant Fabrics**

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Abstract

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The work is licensed under a Creative Commons Attribution-NonCommercial 4.0 International License The utilization of wind energy in Indonesia is still low due to the average wind speed ranging from 3 m/s to 11 m/s. The increasing demand for clothing has driven innovations to address the issue of dirt on clothes in a more practical and environmentally friendly manner. This study explores the use of titanium dioxide (TiO<sub>2</sub>) as an anti-dirt agent on fabric through a photocatalytic coating process. TiO<sub>2</sub> was chosen for its photocatalytic properties, which can oxidize organic compounds into carbon dioxide (CO<sub>2</sub>) and water (H<sub>2</sub>O) when exposed to UV light. The fabric coating was carried out using a modified sol-gel method and tested at various calcination temperatures. The optimal result was obtained at a calcination temperature of 600°C, producing anatase crystals with the best photocatalytic properties. Fabric coated with TiO<sub>2</sub> showed the ability to degrade stains and kill bacteria after nine coating applications, ensuring an even distribution of TiO<sub>2</sub> particles. This study concludes that fabric coated with TiO<sub>2</sub> is effective in self-cleaning under UV exposure, offering an environmentally friendly solution suitable for application in sunlight-rich Indonesia.

Keywords: titanium dioxide (TiO<sub>2</sub>); photocatalytic coating; stain-resistant fabric

#### INTRODUCTION

Clothing is one of the basic needs that humans have. The need for clothes from year to year will increase due to the development of the times and the increasing human population. In the world of clothing, dirt often becomes a problem that is difficult to overcome. Dirt will make clothes look less beautiful and unhealthy. Various innovations have been made to overcome the dirt problem, starting from the manufacture of detergents, washing machines, etc. However, these methods are not practical and economical, so other alternatives are needed to overcome the problem of feces. So far, most people in Indonesia use detergents to clean dirt from clothes. The detergent used to clean clothes is a surfactant that mostly comes from petroleum, a consumable material that is harmful to the environment because it is difficult to decompose [1]. With these various shortcomings, the use of detergent as a laundry agent needs to be reduced because in addition to consuming petroleum, which is an essential material for human life and cannot be renewed, the surfactant can also damage the environment and cause eutrophication so there is a need for an alternative solution to clean clothes that are more practical, economical and more environmentally friendly.

Nature has shown an amazing example by combining chemistry and physics to create a dirt-resistant, super-repellent surface. The anti-fouling effect is commonly called the "taro leaf" effect, taro leaves can repel water and dirt so that the surface is always clean [2]. This effect can be applied in the textile field by utilizing one of the nanotechnologies using titanium dioxide (TiO<sub>2</sub>).

Titanium dioxide is an economically valuable photocatalyst [3]. The working principle of the photocatalyst is that when nano-sized titanium dioxide is exposed to UV light, it will form a superoxide compound that can oxidize various organic compounds into carbon dioxide (CO<sub>2</sub>) and water (H<sub>2</sub>O) [2]. Because titanium dioxide is a catalyst, it will never run out and will continue to experience this reaction.

Nano-sized titanium dioxide can be used as an anti-fouling agent on fabrics by forming a thin layer on the fabric's surface [4]. In this way, all dirt that sticks to the fabric when exposed to UV rays will be oxidized into carbon dioxide (CO<sub>2</sub>) and water (H<sub>2</sub>O) so that the fabric will be free from dirt [5]. In addition, bacteria that stick to clothes will also be oxidized so that clothes free from bacteria become healthier and avoid unpleasant odours [3]. In nature, UV rays are abundant in sunlight. Indonesia is a tropical country rich in sunlight, so this self-cleaning fabric is very appropriate for use in Indonesia.

Titanium dioxide as a fabric upholstery material needs to be tested. This test is intended to determine the influence of each variable, such as calcination temperature and the amount of titanium dioxide coating, on its effectiveness in degrading impurities. If these variables have been studied, self-cleaning fabric with high effectiveness in degrading dirt will be produced. This study aims to test the effect of titanium dioxide calcination temperature and the number of titanium dioxide layers in degrading dye impurities through UV irradiation with a fixed intensity to create a stain-resistant fabric that can be cleaned by itself (self-cleaning fabric) only by irradiation under UV rays or sunlight.

#### METHODS

Anti-fouling fabrics are realized by coating obtained by the process of photocatalystizing titanium dioxide (TiO<sub>2</sub>). Titanium dioxide (TiO<sub>2</sub>) is chosen as a photocatalyst over other agents such as ZnO, ZnS, CdS, Fe<sub>2</sub>O3 because of its excellent photocatalytic ability with maximum quantum yields, especially the anatase crystal structure of TiO<sub>2</sub> [1]. In addition, the price of TiO<sub>2</sub> is relatively lower, the photocatalyst reaction is quite fast even under excellent operating conditions (room, temperature, atmospheric pressure), has a broad spectrum of organic contaminants that can be converted into water and CO<sub>2</sub>, does not require chemical reagents, and does not produce side reactions [2]

The process of photocatalyzing TiO<sub>2</sub> occurs through UV irradiation by a UV reactor, which is a representation of sunlight. If this substance is illuminated by light with an energy higher than its gap band, electrons from TiO<sub>2</sub> will jump from the valence band to the conduction band, with electron pairs ( $e^-$ ) and electric holes (h+) forming on the photocatalyst's surface. Negatively charged electrons and oxygen will combine to form radical O<sub>2</sub> ions, while positively charged electric holes and water will produce OH- hydroxyl radicals. Because the two products are chemically unstable, when organic compounds/dye impurities adhere to the surface of the photocatalyst, the compounds will combine with O<sub>2</sub><sup>-</sup> and OH<sup>-</sup> and become carbon dioxide (CO<sub>2</sub>) and water (H<sub>2</sub>O) [3]. The process can be explained through Figure 1.

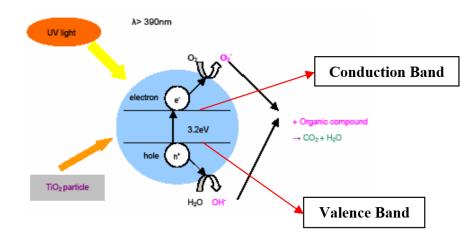


Figure 1. Mechanism of titanium dioxide photocatalyst [3]

#### TiO<sub>2</sub> Synthesis by Modified Sol-Gel Method

To obtain (synthesize)  $TiO_2$ , the sol-gel method is used using an organic metal precursor in an organic solvent (Isopropanol) to produce a gel which is then calcined into  $TiO_2$  [8]. TiCl4 (4 ml) is dissolved in Isopropanol (80 ml) slowly in a stirrer at room temperature to form a precursor solution (Equation 1). Add NH3 until

the solution becomes thick (gel) (Equation 2), then add another 20 ml of Isopropanol to control viscosity and ensure a homogeneous mixture (Equation 3).

Wash the gel with aquades (distilled water) until the chloride ions (Cl) are gone. The washing process is carried out using the AgNO3 indicator. When Cl is undetected (no AgCl deposits are formed), leaching is considered complete (Equation 4). The resulting colloids are dried at 105°C for 5 hours (Equation 5). This drying process aims to remove the solvent and dry the gel into a dry powder. The TiO<sub>2</sub> produced from the drying process is then calcined using *a furnace* at various temperature variations (150°C, 300°C, 450°C, and 600°C). This calcination process aims to increase the crystallinity of TiO<sub>2</sub> and convert it into a stable crystalline form [4]. The change of TiO<sub>2</sub> from gel to crystal is shown in Figure 2.



**(a)** 

**(b)** 



**Figure 2.** Colloidal TiO<sub>2</sub> (a); dry powder of TiO<sub>2</sub> after drying (b); crystalline TiO<sub>2</sub> after calcination (c)

The reaction equation in the process of  $TiO_2$  synthesis is shown in Equation (1-5) below.

 $TiCl_4 + 4H_2O \rightarrow Ti(OH)_4 + 4HCl$ (1)

$$TiCl_4 + 4NH_3 + 4H_2O \rightarrow Ti(OH)_4 + 4NH_4Cl$$
<sup>(2)</sup>

$$Ti(OH)_4 \xrightarrow{aging} TiO_2 \cdot xH_2O$$
(3)

$$NH_4Cl + AgNO_3 \rightarrow AgCl(s) + NH_4NO_3$$
(4)

$$Ti(OH)_4 \rightarrow TiO_2 , xH_2O + (4 - x) H_2O$$
 (5)

#### Fabric coating with TiO<sub>2</sub> crystals

Coating fabrics with TiO<sub>2</sub> is carried out through the dye-press method to improve the photocatalytic properties of the fabric. In this procedure, the cloth is dipped in a solution containing TiO<sub>2</sub> for one minute. After dyeing, the fabric is pressurized at 1.25 kg/cm<sup>2</sup> for five minutes to ensure an even distribution of TiO<sub>2</sub> across the fabric fibres. This process helps to improve the adhesion of TiO<sub>2</sub> particles to the fabric's surface and maximize the contact area. After the pressing stage, the coated fabric is heated in an oven at 60°C to evaporate the solvent and dry the TiO<sub>2</sub> layer. This step is followed by heating at 100°C for five minutes to increase the bond between TiO<sub>2</sub> and the fabric fibres [5].

#### **Degradation Test**

The degradation test method was carried out by soiling the fabric using Remazol Black B stains, and then the cloth was placed in water for 12 hours to evaluate the photocatalytic ability of  $TiO_2$  in degrading the stain. This degradation test was conducted using a UV reactor to expose fabrics coated with  $TiO_2$  to ultraviolet light. Exposure to UV light activates the photocatalytic properties of  $TiO_2$ , which then breaks down and removes Remazol Black B stains from fabrics. This procedure demonstrates the effectiveness of  $TiO_2$  coating in imparting self-cleaning properties to fabrics. The results of degradation testing can provide information on how well  $TiO_2$  coated on the fabric can break down and remove stains under certain lighting conditions, which is an essential indicator in photocatalytic applications. The degradation test in this study is shown in Figure 3.

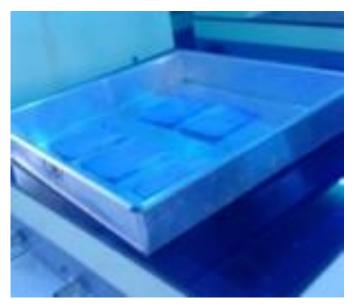


Figure 3. Stain degradation test with Remazol Black B impurities

## **RESULT AND DISCUSSION**

The synthesis of TiO<sub>2</sub> nanoparticles by reacting TiCl4 with Isopropanol and NH3 is quite effective, as evidenced by the number of TiO<sub>2</sub> nanocrystals and the results of the XRD analysis. Week 1 produced 1.84 gr of TiO<sub>2</sub> crystals for a calcination temperature of 150°C, 1.64 gr for a calcination temperature of 300°C, 1.58 gr for a calcination temperature of 450°C and 1.54 gr for a calcination temperature of 600°C. The best results were obtained at calcination temperatures of 600°C as shown in the XRD analysis in Figure 4.

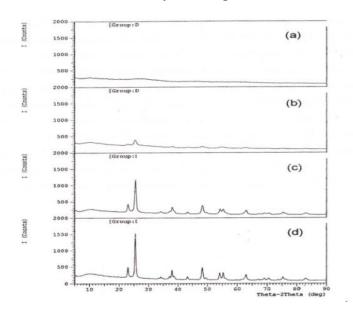
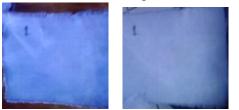


Figure 4. X-ray diffraction (XRD) graphics

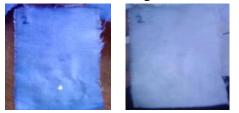
From the image, it appears that the typical diffraction shows the characteristics of titanium dioxide with an antase phase. At  $600^{\circ}$ C, the peak of TiO<sub>2</sub> with the crystalline anatase phase appears, which is the form with the best photocatalyst effect. After getting the best results, TiO<sub>2</sub> synthesis is carried out with a calcination temperature of  $600^{\circ}$ C. The results obtained from the synthesis were 1.03 grams and 5.69 grams.

Furthermore, in the process of coating the fabric with a  $TiO_2$  photocatalyst, the fabric will be coated with  $TiO_2$  by soaking. After the fabric is coated with  $TiO_2$  using the written method, a color degradation test is carried out on the Remazol Black B stain to determine the photocatalyst effect of the fabric coated with  $TiO_2$ . From the results of  $TiO_2$  coating of 1x, 3x, 6x, 9x, and 12x and irradiation for 24 hours, the results of color observation due to photocatalyst reaction shown in Figure 5 were obtained.

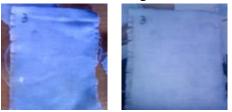
# 1x coating



3x coating



6x coating



9x coating



12x coating

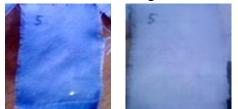


Figure 5. Degradation of Remazol Black B stain color before reaction (left) to after photocatalyst reaction (right)

From the results of the color observation of the study, it can be concluded that the fabric that has been coated with  $TiO_2$  has a sufficient photocatalyst effect. So, it can be concluded in this experiment that the fabric coated with  $TiO_2$  has a

photocatalyst effect that makes the fabric anti-dirty, anti-odor, and anti-bacterial due to the photocatalyst effect.

Chakhtouna et al. [6] highlighted the improved photocatalytic performance and anti-bacterial properties of Ag/TiO<sub>2</sub> photocatalysts. Doping TiO<sub>2</sub> with silver nanoparticles (Ag) extends photocatalytic activity to visible light and improves photocatalysis efficiency by forming Schottky inhibitors that reduce electron/hole pair recombination. In addition, Ag nanoparticles increase the bacterial removal capacity, making Ag/TiO<sub>2</sub> potentially useful for water treatment and surface cleaning applications. These findings support my research on fabric coating with TiO<sub>2</sub> catalyst and a small amount of AgCl precipitate, aiming to develop anti-fouling and anti-bacterial textiles with high photocatalytic and anti-bacterial capabilities.

# CONCLUSION

The maximum calcination temperature required to form  $TiO_2$  in the anatase phase is 600°C. At this temperature, the anatase crystals form well, providing optimal structure and photocatalytic properties for  $TiO_2$  materials. In addition,  $TiO_2$ coating on fabrics using an immersion system achieves maximum effectiveness after nine coats. This iterative process ensures optimal distribution and adhesion of  $TiO_2$ particles on the fabric's surface, thereby improving the photocatalytic properties and self-cleaning of the fabric.

#### Acknowledgment

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