

Article

Decomposition of Fingerprints on Porous TiO₂ Thin Films

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Abstract: This study investigated the effect of the mixing ratio of TiO₂ nanoparticles (P25) and titanium alkoxide (T-sol) on various properties of TiO₂ films. The specific surface area of the TiO₂ film was determined using BET analysis, while the microstructure and thickness were analyzed by field emission scanning electron microscopy (FE-SEM) and atomic force microscopy (AFM), respectively. Transmittance and pencil hardness tests were conducted to evaluate the transparency and durability of the coating layer, respectively. The results showed that, as the P25 content increased, the specific surface area of the TiO₂ film also increased, but this effect decreased as the ratio of T-sol to P25 increased. Additionally, the thickness and surface roughness (Ra) of the coating layer increased as the P25 content increased, with the thickness increasing from 210 to 950 nm and Ra increasing from 51 to 88 nm. However, the transmittance of the coating layer decreased as the P25 content increased, indicating that the films became less transparent. Furthermore, the pencil hardness of the coating layer decreased as the P25 content increased, indicating that the films became less durable. Finally, the oil contact angle decreased as the P25 content increased, indicating that the films became more hydrophilic.

Keywords: organic decomposition; fingerprints; sol–gel method; porous TiO₂ nanoparticles; photoactive self-cleaning coating



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1. Introduction

Fingerprints are a common problem when it comes to the appearance and functionality of various surfaces, including glass, metals, and electronics. To solve this problem, many studies [1–6] have been conducted to form a surface with a self-clearing function. In general, this technology facilitates foreign substance removal from surfaces via superhydrophobic and superhydrophobic actions, and it has been commercialized for use in anti-fogging mirrors, glasses, and external walls of buildings [7–10]. However, despite numerous studies of surface coatings that allow for “water friendly” removal of dust and organic residue [1–10], few have investigated the degradation/decomposition of oil components. Fingerprint oil on transparent surfaces, such as touchscreens and mobile-phone displays, degrades transmittance and leaves an organic residue of dust, bacteria, etc., that contaminates the surface [11–15]. Most of these fingerprints contain water, but they also include a mixture of organic chemicals such as urea, uric acid, amino acid, and ammonia. To minimize fingerprints, studies have focused on oil-repellent and superoleophobic anti-fingerprint surfaces [15–20]. However, anti-fingerprint coatings often use a nanotextured or coating material, such as polytetrafluoroethylene, which degrades transmittance due to light scattering and/or thermodynamic instability; thus, the repellent performance of these coatings is low. In particular, when the material used for anti-fingerprint coatings relies on structural changes on the surface, it is not possible to change the surface energy, which limits coating

effectiveness. In view of the aforementioned limitations, metal oxide based coatings of ZnO, TiO₂, WO₃, etc. can be a better alternative, as they possess better transmittance and conducting properties, are non-toxic, as well as hydrophilic/hydrophobic, and act as an active photocatalyst in the visible region. Among these materials, TiO₂ coatings offer a better practical solution, as it possesses a superior catalytic nature than do other metal oxides. TiO₂ (titanium dioxide) is a well-known semiconductor material with high photocatalytic activity. When TiO₂ is exposed to light, electrons in the valence band are excited to the conduction band, leaving behind holes in the valence band. These electrons and holes can move freely within the semiconductor and can participate in various chemical reactions. In photocatalysis, the excited electrons and holes can react with water, oxygen, and other environmental molecules to produce reactive oxygen species (ROS), such as hydroxyl radicals, superoxide radicals, and hydrogen peroxide. These ROS are highly reactive and can oxidize and degrade organic compounds, including pollutants, into harmless substances such as carbon dioxide and water [21]. Anatase and rutile are two common crystalline forms of TiO₂, and they have different electronic properties that affect their photocatalytic activity. Anatase has a slightly wider bandgap than rutile, meaning it can absorb light with higher energy (shorter wavelength) than rutile. Anatase has a higher valence band maximum than rutile, which means that its valence band is more oxidizing than the oxidation potential of many environmental molecules. This promotes the transfer of electrons from the adsorbed molecules to the TiO₂ surface, which can enhance photocatalytic activity [22]. The sol–gel chemical process involves dissolving a metal alkoxide in an organic solvent, followed by hydrolysis and polycondensation. The optimized P25-to-T-sol ratio provides an advantage for controlling the surface area, which depends on the extent of the network formed in the thin film. In this respect, the sol–gel method can be a good choice for manufacturing functional coatings with improved surface area. It is also a surface treatment technology with high industrial value, as it is suitable for large-area coatings. Moreover, it can be used to fabricate various porous structures via the sol–gel chemical process.

Moreover, TiO₂ films have recently emerged as a promising material for anti-fingerprint coatings due to their unique hydrophilic character, certainly ticking all the requirement for fingerprint prevention applications. Fingerprint prevention and removal rely on the chemical properties of the physical properties of the coating layer. In this respect, the surface area of the films can play a decisive role because the active participation of the surface of the coating is essential for practical applications. Better surface area allows for the effective wetting of the surface, leading to the decomposition of oils and the removal of fingerprints. This has raised interest in the development and optimization of porous TiO₂ films for use as anti-fingerprint coatings in various applications. The porous TiO₂ solutions can generate an active surface area compared to films with a smoother surface.

In this study, a porous TiO₂ solution was prepared using the sol–gel method, and its physical properties were investigated. The surface area of the thin films was controlled by varying the amount of P25, which affected the physical properties of the films. Here, the TiO₂ nanoparticles were used additionally to generate porous thin films. This suggests that the surface area plays an important role. A study was conducted to simultaneously wet and decompose fingerprints—that is, to prevent fingerprints by utilizing photoactive self-cleaning properties through the decomposition of organic matter through a photocatalytic mechanism. For use in high-value industrial applications, the synthesized porous TiO₂ coating layer was designed to increase the utilization value through material optimization to ensure permeability and durability.

2. Experimental Section

Figure 1 is a schematic diagram illustrating the experimental process. The experiments were carried out in the following order. P25 (TiO₂ nanoparticles, Evonik, Essen, Germany) was used to prepare the coating solutions. The mixing ratio of P25 in the coating solution was controlled from 2 to 10 wt%. Ethanol (1.2 M) and distilled water (0.3 M) were mixed to create a solvent for the P25 coating solution, which was ultrasonicated for 10 min to

disperse the P25 uniformly throughout the solution. The titanium isopropoxide (TTIP; $C_{12}H_{28}O_4Ti$, 99.999%, Sigma-Aldrich, St. Louis, MO, USA) used in the TiO_2 sol (T-sol) source was mixed with 0.3 M and stirred for 24 h at room temperature. Using the prepared coating solution and a dip coating method, a glass substrate slide (soda Lime Glass; DIN ISO 8037/1, Marienfeld Company, Lauda-Königshofen, Germany) was coated with a TiO_2 layer. The dipping time in the coating solution was 20 s, and the pull-out speed was 5 mm/s; this allowed us to obtain a thin, uniform coating. The coated samples were annealed in a furnace at 250 °C for 1 h to reduce the TTIP and necking between the TiO_2 particles. An “artificial fingerprint solution” [23–25] containing 16 wt% oleic acid (99%; Sigma-Aldrich, St. Louis, MO, USA), 12 wt% squalene (98%; Sigma-Aldrich, St. Louis, MO, USA), 25 wt% jojoba oil (Daily Dozen Inc., Seoul, Republic of Korea), and 41 wt% corn oil (Sajo, Seoul, Republic of Korea) was mixed in a 100 mL beaker via stirring for 1 h and used after storage at room temperature for about 24 h.

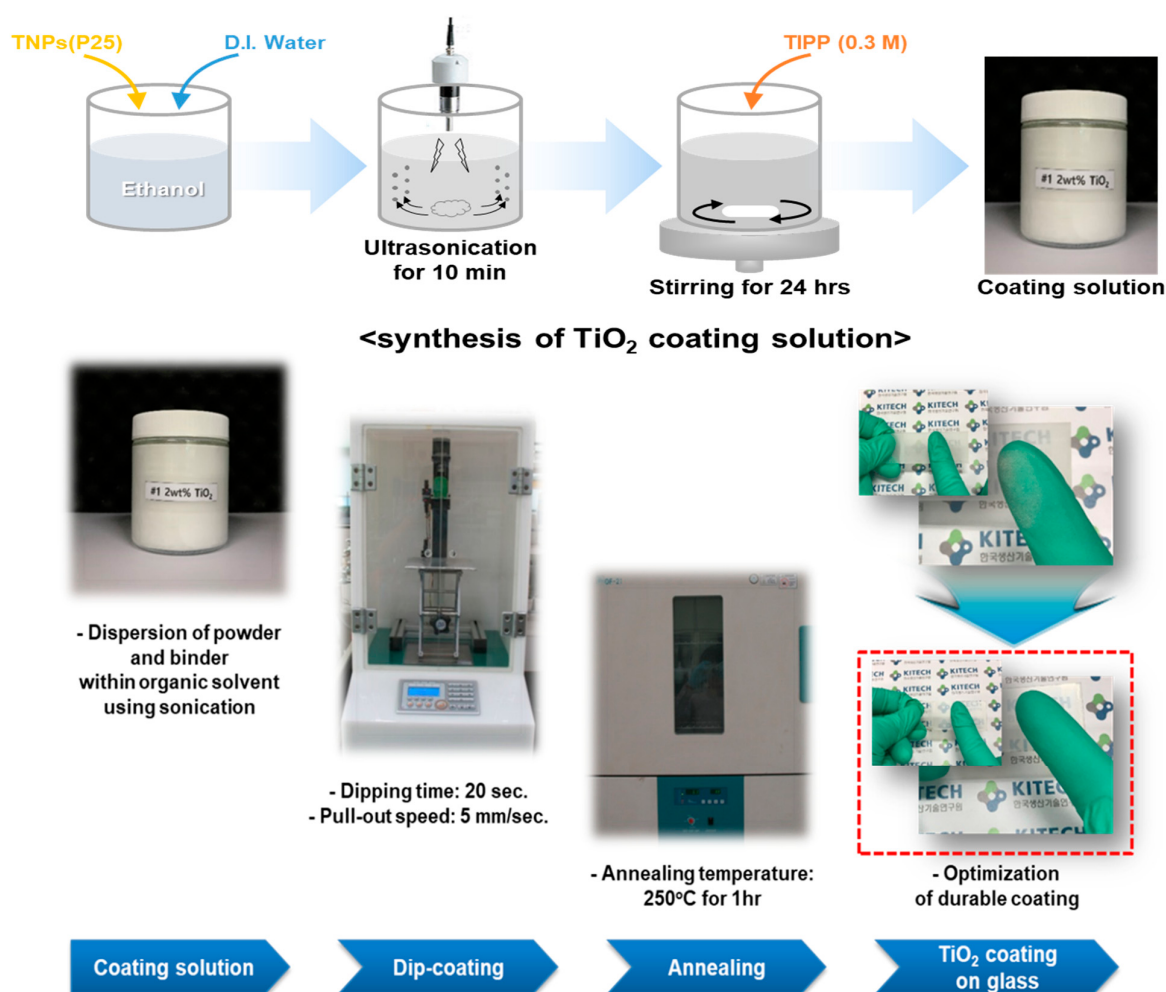


Figure 1. Schematic diagram of TiO_2 coating process by the sol–gel chemical method.

The specific surface area of suspensions with different TiO_2 particle contents was measured using the Brunauer–Emmett–Teller (BET) method and a gas adsorption analyzer (Autosorb 1-1; Quantachrome, Boynton, FL, USA). The structural phase was investigated by X-ray diffraction (XRD, Philips X’pert MRD diffractometer, Philips, Amsterdam, The Netherlands). The microstructure and thickness of the TiO_2 coatings were characterized using scanning electron microscopy (SEM; Mira 3; Tescan, Brno, Czechia). Surface roughness (R_a) changes with TiO_2 particle content were measured using atomic force microscopy (AFM; Nano-R; Pacific Nanotechnology, Santa Clara, CA, USA). In addition, the optical

properties of the film were measured using an ultraviolet (UV) spectrophotometer (VS650; Jasco International, Ltd., Tokyo, Japan) over the wavelength range of 350–800 nm. The durability of the coating layer was measured using a pencil hardness test (CT-PC; Core Tech Co., Ltd., Ulsan, Republic of Korea).

In order to compare the surface characteristics of the artificial fingerprint liquid and water, the contact angle according to the TiO_2 particle content was measured using contact angle measurement (DSA 100, Krüss, Hamburg, Germany).

The fingerprint decomposition of the coating layer was characterized using optical microscopy (BX 51 M; Olympus, Tokyo, Japan) and Fourier-transform infrared spectroscopy (FT-IR; IR-Prestige-21; Shimadzu, Kyoto, Japan) after UV irradiation for 20 min.

Finally, the photocatalytic activity of the TiO_2 coatings was measured by evaluating the degradation of organic matter using a 0.01-mM methylene blue (MB) dye solution (Sigma-Aldrich, St. Louis, MO, USA) under sunlight irradiation. During irradiation, 2–3 mL of solution were collected per hour. The light absorbance of an aliquot was measured using a UV spectrophotometer (VS650; Jasco International, Ltd.).

3. Results and Discussions

The representative XRD patterns obtained from the P25 and TiO_2 films are shown in Figure 2. The thin film prepared comprised an anatase and rutile TiO_2 phase, respectively, with all the peaks matching the Joint Committee on Powder Diffraction Standards; JCPDS-ICDD Card No. 894,921 (anatase) and JCPDS-ICDD Card No. 894,920 (rutile), confirming the successful formation of both phases. The XRD pattern for all prepared TiO_2 films was similar, and is not included in the figure.

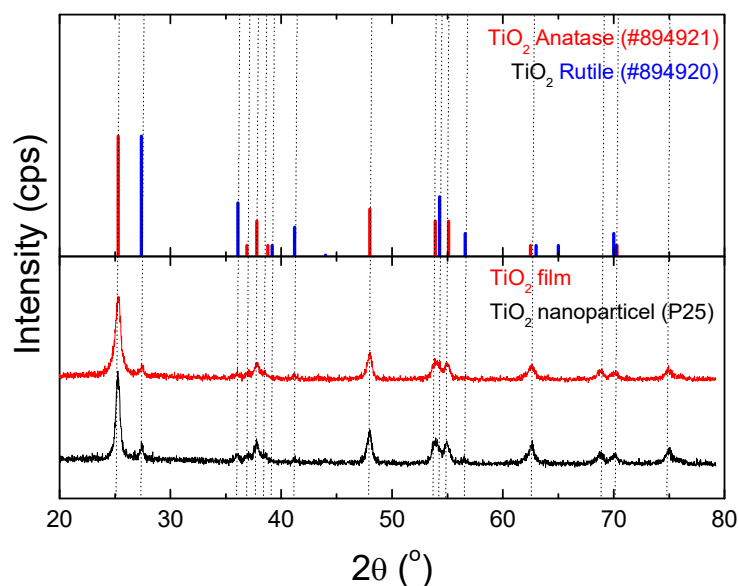


Figure 2. XRD pattern of P25 and TiO_2 film.

The increase in the surface area of the TiO_2 films can be understood by considering the porosity of the T-sol network formed on the film. The TTIP precursor of the sol–gel undergoes a hydrolysis process that yields H_2O . Hydrolyzed T-sol forms a network between nanoparticles to limit aggregation. The formation of extended networks between the P25 determines the porosity of the TiO_2 film. Such network formation prevents P25 aggregation, forming a porous TiO_2 film. TiO_2 networks maintain an integrated P25 construct and increase the specific surface area of the film by forming porous networks. Figure 3 shows the BET analysis results of the specific surface area with respect to the P25 content.

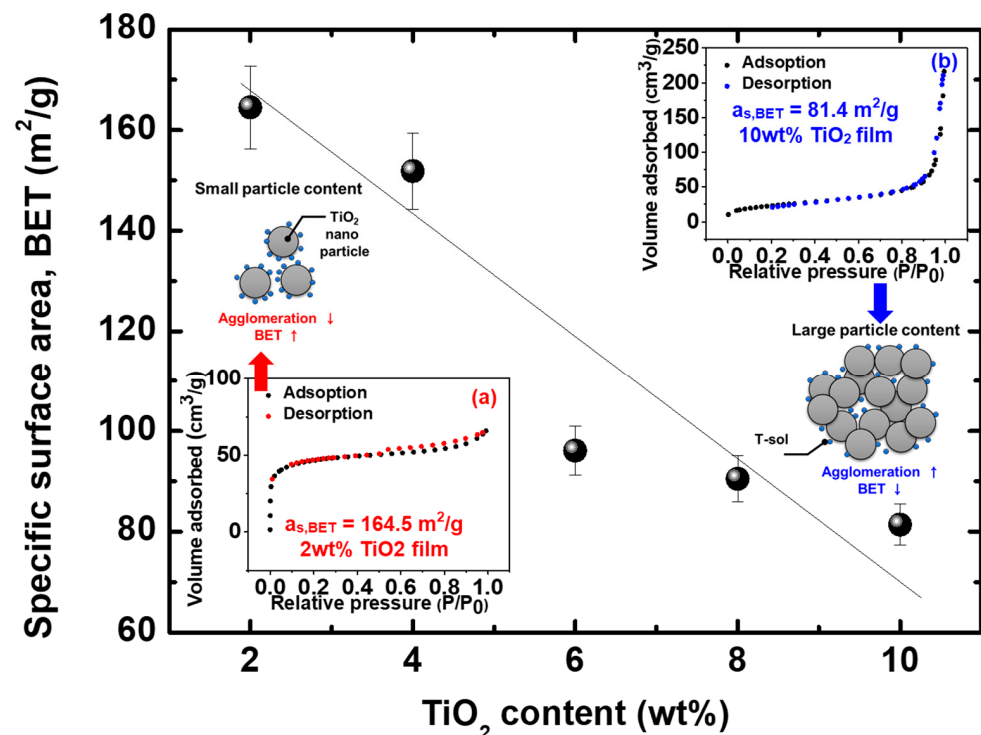


Figure 3. BET analysis image of TiO_2 films. The inset figures and schematic images in (a) and (b) show representative BET graphs and synthesis structure diagrams of 2 and 10 wt% TiO_2 films, respectively.

As can be seen in the graph, for the smallest mixing amount of P25 of 2 wt%, the specific surface area was $164.5 \text{ m}^2/\text{g}$. A sharp decrease in the specific surface area was observed for a P25 concentration of 6 wt%, and a minimum specific surface area of $81.4 \text{ m}^2/\text{g}$ was obtained for a concentration of 10 wt%. As the ratio of T-sol to P25 content increased, the specific surface area decreased. Thus, P25 aggregation increased with the P25 content, with same amount of T-sol, resulting in a smaller specific surface area. The microstructure of the coating layer prepared by the deep coating method, as well as its thickness and surface characteristics, were analyzed to determine the optimal coating conditions, according to the P25 content. Figure 4 shows the changes in the microstructure of the coating layers observed using field emission scanning electron microscopy (FE-SEM). Using the 2 wt% mixing ratios of P25, i.e., the smallest amount, the P25 microstructure and coating layer formed by the T-sol could be clearly distinguished in the SEM images. The coating layer formed by the T-sol had a relatively dense microstructure (Figure 4a). In contrast, as the content increased to 4 wt% (Figure 4b), a large number of P25 particles was visible in the coating layer. At ≥ 6 wt%, a porous structure was observed (Figure 4c–e). When the T-sol content is constant, the amount that allows bonding between P25 determines the porosity of the resulting coating structure. Ultimately, the porous structure of the coating layer is created by P25 aggregation as the mixing ratio of the P25 increases. The thickness and Ra of the coating layer were analyzed using AFM, according to the microstructure characteristics of the coating layer and the P25 content (Figure 5). The thickness of the TiO_2 coating layer increased from 210 to 950 nm as the P25 content increased. The Ra of the coating layer was measured at 51–88 nm, and the thickness showed the same tendency. As shown in the FE-SEM image of Figure 4, the Ra value increased sharply with the mixing amount of 6 wt% and exceeded 25 nm due to the agglomeration of P25 and the resulting porous structure.

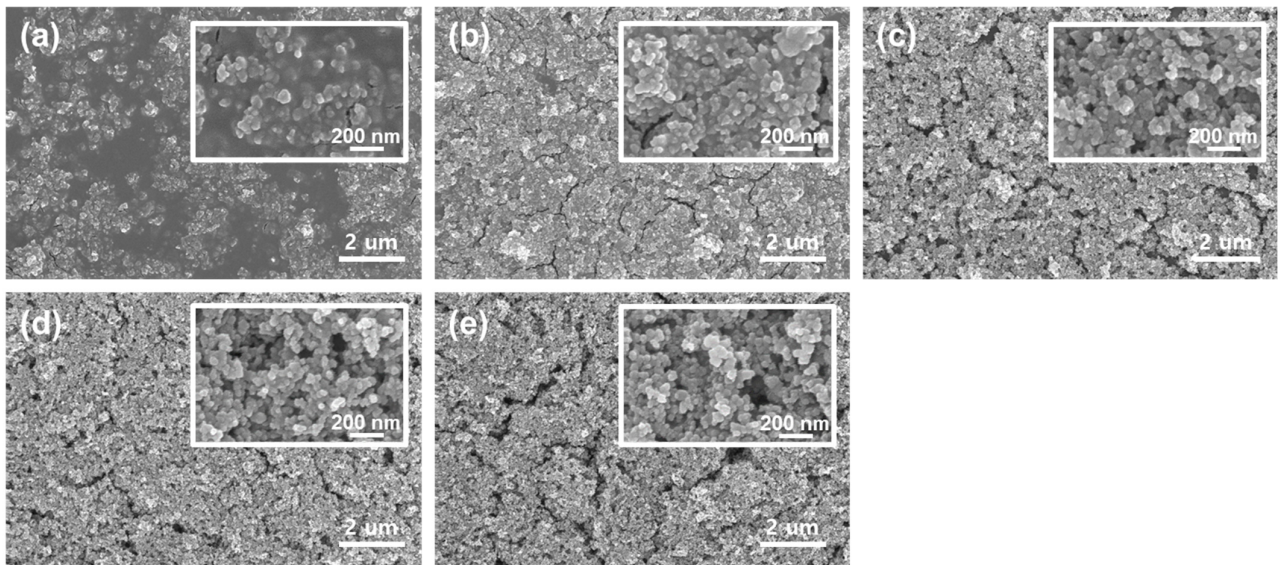


Figure 4. FE-SEM images of TiO_2 coating layer prepared with different TiO_2 particle contents of (a) 2 wt%, (b) 4 wt%, (c) 6 wt%, (d) 8 wt%, and (e) 10 wt%.

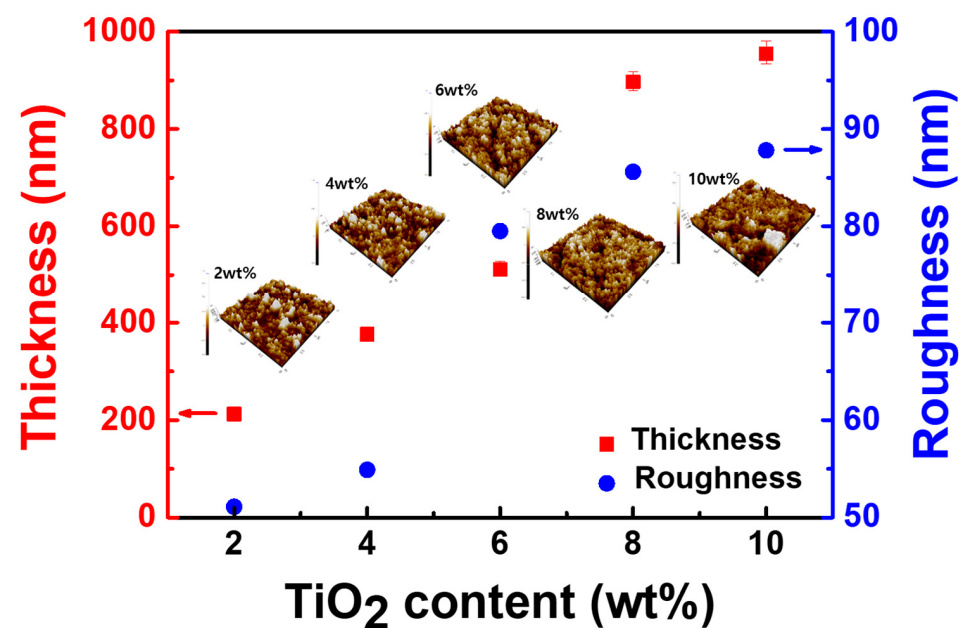


Figure 5. Thickness and roughness of the coating layers with different TiO_2 particle contents.

The transparency changes and resilience of the coating layer according to the P25 content were confirmed by transmittance and pencil hardness tests performed on the coating (Figure 6). The transmittance of the coating layer containing 2 wt% P25 was >85% in the visible light range (300–900 nm) and tended to decrease as the P25 content increased (Figure 6a). In particular, in samples with a P25 content of 6 wt%, the transmittance decreased sharply because the thickness and Ra of the coating layer increased with the P25 content; this promoted scattering from the surface microstructure of the coating layer and reduced the transmittance of incident light (Figures 4 and 5).

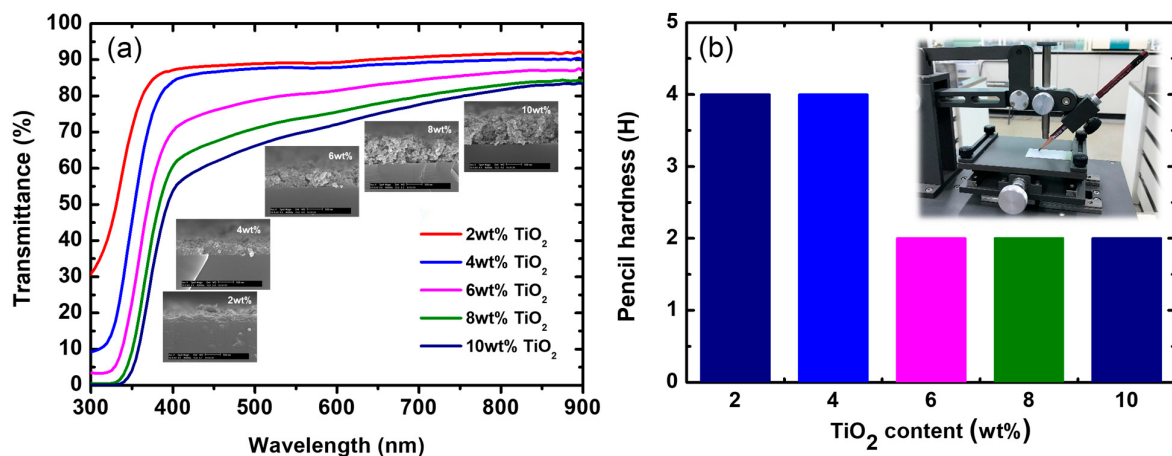


Figure 6. Characterization of TiO₂ coating layer with different particle contents—(a) transmittance and (b) pencil hardness. The inset of (a) is an inclined cross-section image observed by SEM. The inset of (b) is the result of the pencil hardness test.

In previous studies, the transmittance of the coating layer prepared using an alkoxide solution was described based on the ratio of light scattering and the precursor content of the pore structure formed in the coating layer [26–28]. In another study by Chrysicopoulou et al. [2], the optical properties of the coating layer with a thickness < 100 nm depended on the surface Ra, as shown by AFM and UV analyses. Therefore, the results of the current study are consistent with those of prior studies that observed changes in the transmittance of the coating layer according to the coating thickness and surface Ra. Among the many methods used to evaluate coating resistance, the pencil hardness test is commonly used in industry to measure the scratch hardness of substrate coatings [29,30]. In this study, with 2 and 4 wt% P25 content, the pencil hardness was 4H, as shown in Figure 6b. Above 6 wt%, the pencil hardness was 2H, i.e., half the value of that with 2 and 4 wt% P25 content. Thus, good transmittance and durability of coating layers with a 2 to 4 wt% P25 content were confirmed, consistent with our BET, FE-SEM, thickness, and surface Ra analyses (Figures 3–5).

Figure 7 shows the contact angle measurement results. The oil contact angle, when using an artificial fingerprint solution, was found to be 4.5 to 19.4 degrees, which is a comparably low contact angle. It was confirmed that a coating layer having oleophilic properties compared to the oil component was formed, and the contact angle result tended to decrease according to the increase in P25 content. Generally, it was reported that if the coating layer has oleophilic properties, it has a hydrophobic character in terms of the water component [31,32]. In the same way, our result is consistent with those of previous studies. Moreover, when the P25 content was higher than 6 wt%, it was found that the oil contact angle was maintained at less than 6 degrees. As shown in the microstructure (Figure 4) and roughness (Figure 5) results, the surface characteristics tended to change suddenly, depending on the microstructure and surface roughness of the films. These phenomena could be described by capillary imbibition, where the wetting and dispersion of droplets are enhanced on a rough surface [17]. Hence, we could confirm that a coating layer with a P25 content of 6 to 10% is suitable for the rapid absorption and large contact of fingerprints within the coating layer, demonstrating good decomposition properties. However, concerning the microstructure, thickness, transmittance, and pencil hardness results overall, we determined that the optimum sample for decomposition was the coating layer containing 2 wt% P25, which has good transmittance and durability with a thinner thickness than the other samples.

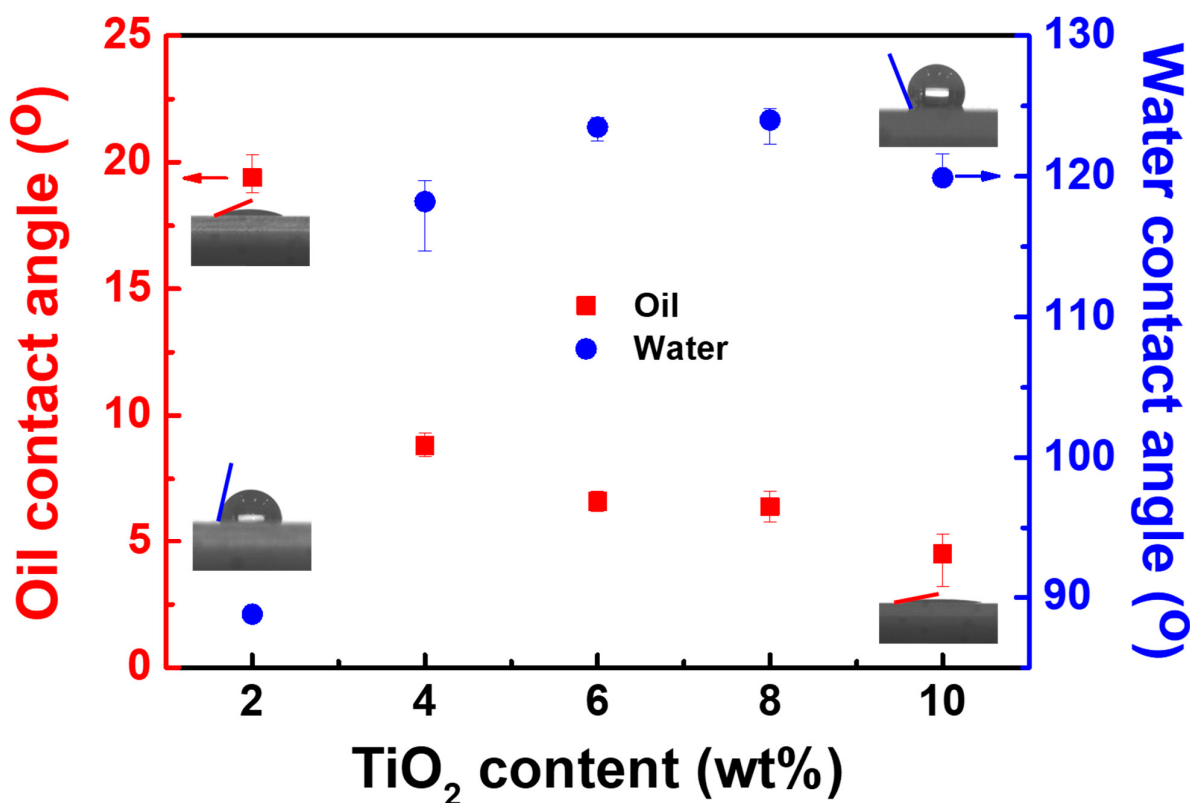


Figure 7. Water and oil contact angle measurement of coating layer with varying TiO₂ content.

Figure 8 compares fingerprint degradation due to UV exposure between the 2 and 10 wt% coating solutions on a glass substrate. UV light ($\lambda = 365$ nm) was irradiated at a distance of 2.2 mm from the photocatalytic film, and the average light intensity was 1.0 mW/cm². In the case of glass, as shown in Figure 8a, exposure for up to 60 min partially dried the coating via the heat generated by UV irradiation. However, even after drying, traces of fingerprints (white arrow in Figure 8) indicating an organic residue remained. In contrast, for TiO₂ coating layers using 2 and 10 wt% P25 contents, the fingerprints degraded gradually over a few minutes. For the smallest content, 2 wt% P25, it took 16 min for fingerprints to completely deteriorate (Figure 8b). On the other hand, the decomposition time of a coating layer with a P25 content of 10 wt% was reduced to 12 min or less because the wettability of the oil was relatively high (Figure 8c). The coating layer with a P25 content of 10 wt% provided the optimal conditions for fingerprint removal; however, the coating layer with a P25 of 2 wt% provided the best transmittance and durability. These parameters should be suitable for commercial applications. As can be seen in the previous results, the average particle size is composed of the same P25 in all samples, and the specific surface area, permeability, pencil hardness, and oil contact angle are affected by the increasing content of P25. Moreover, the P25 size affects the specific surface area and, at the same time, is a decisive factor for the photocatalytic reaction [33,34].

Next, using FT-IR, we evaluated the fingerprint removal performance of the coating layer after UV exposure. Figure 9 shows the FT-IR analysis results. Artificial fingerprints made of oleic acid, squalene, jojoba oil, and corn oil consist of organic compounds. A carbon compound such as C=O, C=C, and CO bonds was suggested in previous studies [35,36]. In the case of the 2 wt% P25 coating layer, the characteristic peak representing the FT-IR spectrum was reduced after exposure to UV light for 20 min, indicating that CH (2674, 2925, 3006 cm⁻¹), C=O (1743 cm⁻¹), C=C (1655 cm⁻¹), and CO (1170 cm⁻¹) bonds decomposed under UV exposure. In contrast, in the case of glass, no change in the spectrum was seen after exposure to UV light for the same amount of time.

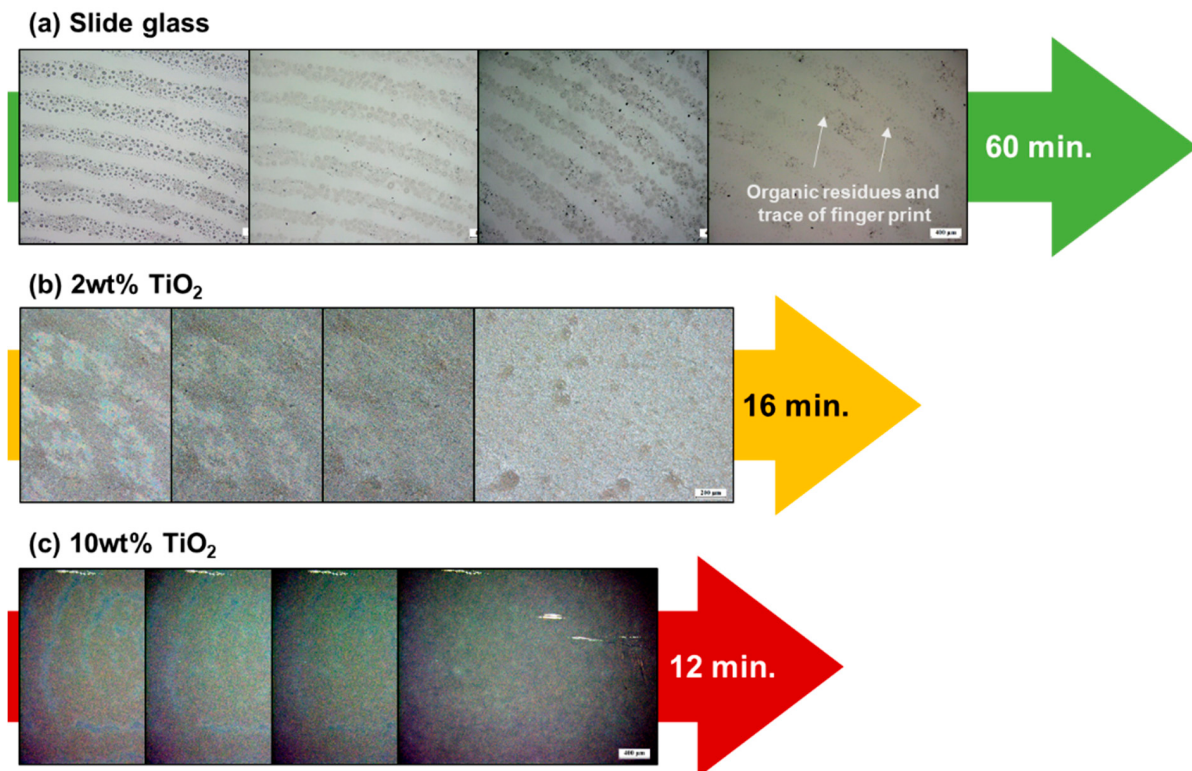


Figure 8. Comparison of degradation under UV irradiation of fingerprints on (a) glass slide, (b) 2 wt% TiO_2 , and (c) 10 wt% TiO_2 .

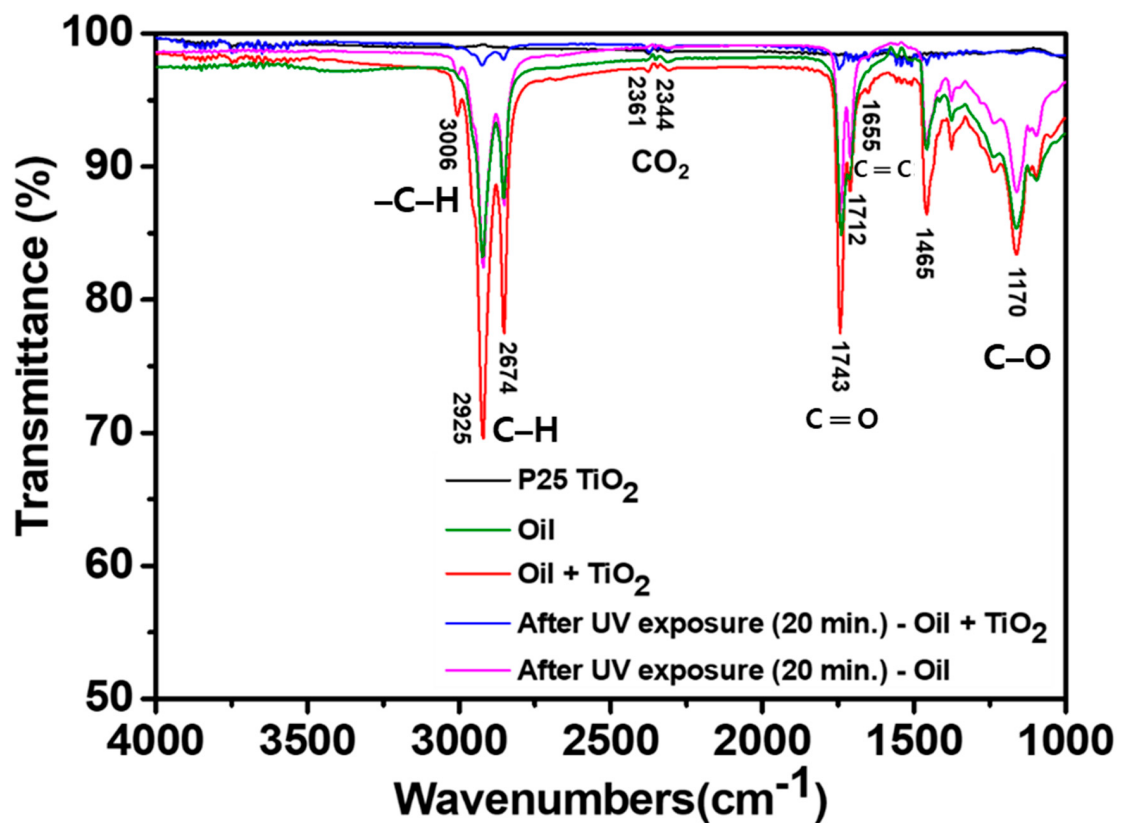


Figure 9. Comparison of Fourier-transform infrared (FT-IR) spectra after the fingerprint degradation process on 2 wt% of TiO_2 coating layer and glass.

We wanted to ensure that fingerprint oil was naturally decomposed by the heat or light associated with UV light exposure. Changes in artificial fingerprints on glass were observed after UV exposure, but there were few or no changes in the FT-IR spectrum or characteristic peaks. Therefore, successful fingerprint removal from the TiO_2 coating layer was confirmed. The TiO_2 coating layer produced through this study confirmed whether oil ingredients and various organic matters could be decomposed. This test was carried out using a 2 wt% P25 coating sample in the methylene blue dye solution most commonly used for photocatalyst disassembly. Figure 10a shows the almost complete decomposition of MB within 6 h, without peeling or damage to the coating layer. With an increasing irradiation time, a gradual decrease in the MB peak at 600–680 nm occurred, clearly indicating degradation of the MB dye molecules. Figure 10b shows almost complete degradation of MB within 6 h, without peeling or damage to the coating layer. Since the volume of the MB dye solution (100 cm^3) ratio to the TiO_2 coating layer volume ($3.125 \times 10^{-4} \text{ cm}^3$) is very small, this result seems to have taken longer than when using powders. We expected that the rapid decomposition of organic compounds would be possible within a few minutes, when a large number of TiO_2 films were used.

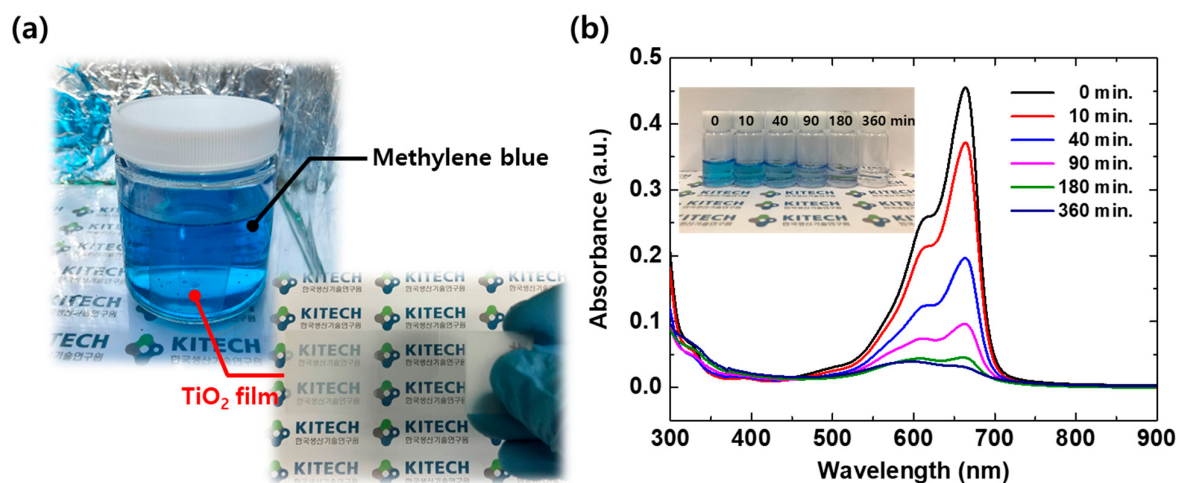


Figure 10. (a) Optical images of photocatalytic evaluation process, and (b) absorbance spectra of methylene blue dye after sunlight irradiation.

4. Conclusions

In summary, we synthesized a porous TiO_2 multifunctional coating material for protection against fingerprints through wetting and decomposition using the sol–gel method. The effect of the P25 content on prepared porous TiO_2 thin films was specifically investigated, including the porosity, microstructure, thickness, surface roughness, transmittance, and contact angle. T-sol increased the specific surface area of the film by forming a porous network with the P25. These properties were significantly influenced by the addition of P25 from 2 to 10 wt%. It can be seen that the specific surface area of the TiO_2 film decreases from 164 to $81.4 \text{ m}^2/\text{g}$ as the ratio of T-sol/P25 decreases, and the content of P25 increases from 2 to 10 wt%. The porous thin film with highest surface area showed better transmittance and pencil hardness test stability compared to the TiO_2 film with higher content of P25, respectively. The thickness and R_a of the coating layer increased with increasing P25 content, while the transmittance and pencil hardness decreased. Furthermore, this study confirmed that the oil contact angle decreased as the P25 content increased, and the optimum mixing ratio of P25 was between 2 and 4 wt%, which was used to obtain good transmittance and durability of the coating layers. The results suggest that optimizing the mixing ratio of P25 is crucial to control the physical and chemical properties of TiO_2 films. The films with a suitable porous surface, exhibiting a better surface area, allows for the effective wetting of the surface, leading to the decomposition of oils and the removal of fingerprints.

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