

Article

Dynamic Synergy of Photocatalysis and Hydrophobicity in PDMS-Grafted TiO₂ Coatings for Long-Lasting Self-Cleaning

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Abstract: A fundamental conflict in self-cleaning coatings lies in the trade-off between photocatalytic activity (requiring hydrophilic TiO₂) and hydrophobic repellency (requiring low-surface-energy polymers). Here, this bottleneck is broken through an in-situ grafting strategy that anchors polydimethylsiloxane (PDMS) chains onto TiO₂ surfaces via robust Si-O-Ti covalent bonds. By controlling the loading amount of TiO₂ in the sol, it enables the adjustment of the water contact angle, light transmittance, as well as self-cleaning performance of the coating. The resultant M-TiO₂ nanocatalyst exhibits excellent dispersion in hydrophobic media and imparts the composite coating with a water contact angle up to 163°. The dynamic photocatalytic process, decomposing organic stains while maintaining surface non-wettability, is systematically elucidated. Remarkably, the optimal coating (0.75 wt.% M-TiO₂) sustains a steady-state degradation rate of rhodamine B under UV light, achieving >90% conversion within 85 h, without compromising its water-repellent function. The covalent interface acts as a bridge between the organic components and the inorganic catalyst, significantly improving the dispersion uniformity and stability of TiO₂ nanoparticles in PDMS, thus endowing the resulting coating with good light transmittance in visible region, excellent self-cleaning performance and satisfied service durability. This work demonstrates that rational interface engineering can transform the inherent photocatalysis vs. hydrophobicity antagonism into a durable, dynamic synergy, providing a versatile platform for next-generation photocatalytic self-cleaning materials.

Keywords: photocatalysis; hydrophobicity; inherent antagonism; dynamic synergy; self-cleaning coating; surface grafting

1. Introduction

With the rapid advancement of modern architecture, glass materials have been widely utilized in both residential and commercial buildings due to their superior optical transmittance, aesthetic appeal, and structural simplicity [1–3]. However, when was exposed to outdoor environments, building glass inevitably accumulates inorganic dust, rainwater stains, bird droppings, oil fumes as well as other contaminants over long-term service [4,5]. Unlike traditional building materials such as cement, building glass require effective maintenance to maintain the appearance and functionality. Traditional manual cleaning not only incurs substantial labor costs and cleaning-agent consumption but also poses significant safety hazards associated with high-altitude operations [6,7].



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Consequently, the development of low-maintenance self-cleaning glass coatings has become an urgent need in the field of functional building materials [8–10].

Currently, super-hydrophobic coatings and photocatalytic coatings are two of the mostly developed self-cleaning coating systems, while both systems still face prominent bottlenecks restricting their large-scale practical application [11–14]. For traditional super-hydrophobic coatings, the self-cleaning efficiency relies on their fragile micro/nano rough structures, which are susceptible to collapse under mechanical abrasion and outdoor sand-wind erosion, leading to irreversible loss of water repellency [15,16]. Meanwhile, the super-hydrophobic coatings are only useful in removing the surface pollutants through passive rolling of water droplets, but helplessness against viscous organic pollutants that tend to clog the surface rough pores [17,18]. As for titanium dioxide (TiO_2) photocatalytic coatings, their inherent super-hydrophilicity result in the spreading of water film and the formation of residual water marks on glass surfaces, which also result in severe fogging and icing problems in high-humidity and low-temperature environments, greatly damages the optical performance and practical usability of building glass [19]. The intrinsic antagonism between the photocatalytic activity of hydrophilic TiO_2 and the water-repellent requirement of hydrophobic coatings represents a critical trade-off that has long constrained the development of durable self-cleaning materials.

Integrating TiO_2 photocatalysis with super-hydrophobic coating is an efficient strategy to overcome the limits of single-functional coatings [20–22]. In the synergistic system, super-hydrophobic surface can remove the loose inorganic dust via rolling water droplets, while the stubborn organic contaminants can be decomposed thoroughly through the photocatalysis of TiO_2 . However, due to the significant differences in their surface properties and the destructive effect of the TiO_2 to organic components, directly mixing the two components is clearly inappropriate [23,24]. On one hand, the hydrophilic TiO_2 will severely agglomerate in the hydrophobic coating, reducing the light transmittance and photocatalytic effect, and results in decreased super-hydrophobic property of the coating. On the other hand, under solar irradiation, the strong oxidizing free radicals generated by TiO_2 will continuously oxidize and etch surrounding hydrophobic polymer substrates, resulting in decreased hydrophobic performance and service durability. It remains a huge challenge to balance three core indicators of building glass coatings: excellent hydrophobicity, high optical transmittance and stable photocatalytic degradation performance synchronously via simple and low-cost preparation methods [25,26].

Surface modification of TiO_2 nanoparticles (NPs) may offer a viable pathway to overcome these inherent conflicts and performance limitations [27–29]. Liang et al. [30] prepared coatings by mixing TiO_2 NPs modified with fluorosilane-free coupling agent KH560 with epoxy resin solution, and found that the modification treatment effectively improved the dispersibility of TiO_2 and the corrosion resistance of the resulting super-hydrophobic coating. Yang et al. [31] modified TiO_2 by using stearic acid, and found that the introduced organic group efficiently decreases the surface energy of TiO_2 NPs and facilitates the in-situ growth of self-cleaning coating on wood. In conclusion, surface-modification provides a simple, mild, and easy-to-operate method to address the mutual constraints between photocatalytic activity and hydrophobic stability.

As a typical silicone elastomer, polydimethylsiloxane (PDMS) has been widely adopted in the preparation of super-hydrophobic coating owing to its unique molecular structure and superior chemical stability [32–34]. The excellent light transmittance makes PDMS highly suitable for the preparation of coatings on the surface of glass building materials [35]. In addition, the flexible Si-O chain and the numerous low-surface-energy methyl (-CH₃) side groups endow PDMS with the ability in reducing the surface free energy of composite coatings, which is essential in the preparation of super-hydrophobic coating [36].

The direct combination of hydrophilic photocatalytic TiO_2 with hydrophobic PDMS has long been considered a logical yet practically unattainable strategy for self-cleaning coatings, owing to two intertwined bottlenecks: the severe aggregation of TiO_2 in non-polar media, and the continuous photo-oxidative erosion of the polymer matrix by TiO_2 -generated reactive radicals. These issues are not merely technical obstacles but reflect a fundamental physicochemical antagonism between photocatalytic activity and hydrophobic stability. In this work, this antagonism is eliminated by introducing an in-situ grafting strategy that constructs a covalent Si-O-Ti interface between TiO_2 and PDMS. This interfacial design simultaneously addresses both bottlenecks: the grafted PDMS shell eliminates TiO_2 agglomeration and contributes to surface hydrophobicity, while the Si-O-Ti linkages suppress polymer degradation through interfacial charge recombination and crosslinking effects. As a result, the optimally formulated coating (0.75 wt.% M- TiO_2) not only exhibits super-hydrophobicity with a water contact angle (WCA) of $>150^\circ$ and robust photocatalytic degradation of organic dyes, but also sustains these functions synergistically under harsh conditions including acid/alkali exposure, mechanical wear, and two-month outdoor weathering. More significantly, our findings reveal that the apparent trade-off between photocatalysis and hydrophobicity can be reconciled via rational interface engineering, transforming it into a dynamic synergy that operates on multiple time scales, rapid physical self-cleaning via water rolling and sustained chemical

decomposition via photocatalysis. We anticipate that this grafting strategy offers a versatile platform for fabricating durable dual-functional coatings, with broad implications for photocatalytic environmental remediation and smart building materials.

2. Experimental Section

2.1. Pretreatment of the Glass Substrate

Firstly, the purchased glass was immersed in 1 mol/L of hydrochloric acid solution for 15 min to remove the inorganic salts and metallic contaminants on the surface. After then, the acid-treated glass was washed by ultrasonic treatment for 30 min respectively in ethanol and deionized water to remove the surface adsorbed acid and the residual organic impurities. Finally, the obtained clean glass was dried in an oven overnight and ready for use.

2.2. Grafting of TiO_2

The grafting of TiO_2 was achieved through a photopolymerization method. Firstly, 1 g of TiO_2 NPs with a particle size of 20 nm were dispersed into 50 mL tetrahydrofuran solution by ultrasonic for 30 min to form suspension. Secondly, 10 g of PDMS was added to the obtained suspension and stir to get the uniform mixture, and then the mixture was exposed under UV-A light (365 nm, 12 W) for 2 h to achieve graft modification of TiO_2 NPs. Subsequently, the resultant solid product was collected by centrifugation, washed by toluene for 5 times to remove residual unreacted PDMS, and then was dried at 100 °C for 24 h. Finally, the dried solid product was ground to obtain hydrophobic modified TiO_2 NPs, namely M- TiO_2 .

The control sample PM- TiO_2 (physical method modified TiO_2) was fabricated through the same process to that of M- TiO_2 apart from the absence of UV-light irradiation.

2.3. Preparation of Coating Sol

A series of coating sols with different M- TiO_2 loadings were prepared as follows. Specifically, M- TiO_2 powder was respectively dispersed into 15 mL of n-hexane (0.659 g/mL) via ultrasonication for 30 min to form homogeneous suspensions. Subsequently, 10.0 g of PDMS and 1.0 g of curing agent were added to the suspension, followed by magnetic stirring at room temperature for 1 h to ensure thorough mixing. The resulting mixture was designated as the coating sol. Through this process, a series of coating sols containing different mass percentages of M- TiO_2 (0.25, 0.5, 0.75, 1.0, and 1.25 wt.%) were prepared.

2.4. Preparation of PDMS/M- TiO_2 Coating

The preparation of PDMS/M- TiO_2 coating is shown in Figure 1. The pre-cleaned glass slides were vertically immersed into the as-prepared coating sol and withdrawn using a dip-coating apparatus at a constant speed of 60 mm/min to obtain a wet film on both sides of the substrate. The coated glass slides were then allowed to cure at 60 °C for 12 h under ambient conditions to ensure complete crosslinking of the PDMS matrix. The resultant coatings were denoted as PDMS/M- TiO_2 -x, where x represents the nominal mass fraction of M- TiO_2 in the coating (x = 0, 0.25, 0.5, 0.75, 1.0, and 1.25 wt.%, respectively). The sample with x = 0 wt.% corresponds to the pure PDMS coating, which served as the control.

2.5. Characterization

The morphology of the samples was observed by the field emission scanning electron microscope (Gemini SEM 500, manufactured by Carl Zeiss (Shanghai, China) Management Co., Ltd., purchased from Shanghai, China) integrated with energy dispersive spectroscopy (EDS). Crystal structure was studied by X-ray diffraction (XRD, Bruker D8 ADVANC, manufactured by Bruker Co., Ltd., purchased from Beijing, China). Fourier transform infrared (FTIR, Bruker VERTEX70, manufactured by Thermo Fisher Scientific, purchased from Waltham, MA, United States) spectroscopy was measured to investigate the functional groups in the samples. X-ray photoelectron spectrometer (Thermo Fisher ESCALAB Xi+, manufactured by Thermo Fisher Scientific, purchased from Waltham, MA, United States) was used to detect the surface chemical state of the samples. The specific surface area of the samples was measured by the Micromeritics Tristar 2000 instrument (manufactured by Anton Paar GmbH, purchased from Graz, Austria) at 77 K and calculated by the Brunauer-Emmett-Teller (BET) method. UV-Visible absorption spectra were recorded through a UV-vis-NIR spectrometer (JASCO, V-670, manufactured by PerkinElmer, Inc., purchased from Waltham, MA, United States) with an integrating sphere by using bare glass as

the blank sample. The water contact angle (WCA, DSA100S, manufactured by KRÜSS GmbH, purchased from Hamburg, Germany) of the coating was measured by an optical contact angle meter at room temperature.

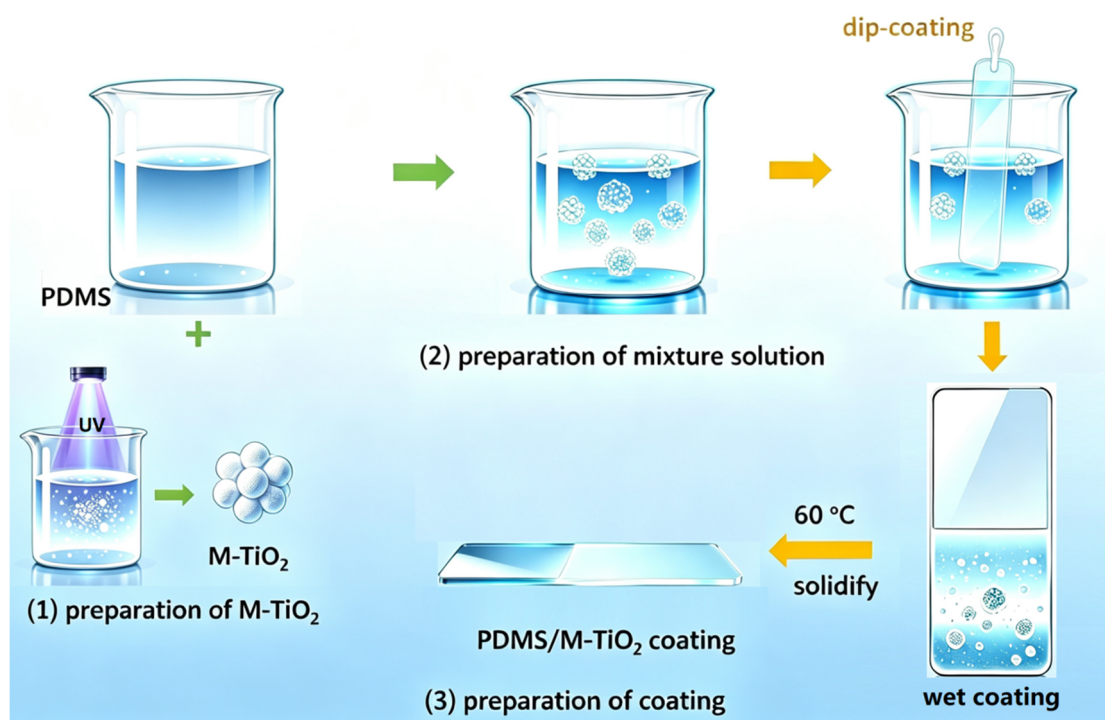


Figure 1. Schematic diagram of the preparation process of PDMS/M-TiO₂ self-cleaning coating.

2.6. Superhydrophilicity Performance Test

The superhydrophobic self-cleaning effect of the coating was tested on an inclined glass plate. Specifically, the glass with and without coating were placed with the slant angle of 30°, and a certain amount of liquid was dropped onto the upper end of the glass through a dropper. The rolling and remaining conditions of the droplets on the glass surface were observed to evaluate the superhydrophobic behavior. Furthermore, a certain amount of dust was scattered on the glass slides in the same slant angle, and liquid droplets were added drop by drop. The rolling of droplets on the glass surface and the removal of dust was observed to evaluate the effectiveness of dust pollution removal.

The long-term hydrophilicity of PDMS/M-TiO₂ coated glass and bare glass was studied by placing the glass outdoors for 2 months, after then the water contact angle and the degradation efficiency to Rhodamine B (RhB) were studied.

2.7. Photocatalytic Performance Test

RhB, Sudan III and methylene blue (MB) solutions were used to simulate organic pollutants. The photocatalytic ability of the coatings was investigated by monitoring the degradation efficiency of organic dyes after irradiated under UV light (12 W, 254 nm) for different time. The glass with the coatings area of 2.5 cm × 2.5 cm was placed in a culture dish with a radius of 6 cm with the coated side facing upwards. After then, 40 mL of 5 mg/L RhB solution was added into the culture dish, after stored in the dark for 30 min to achieve adsorption-desorption equilibrium, the test sample was irradiated under 12 W of UV light for photocatalytic reaction. The solution was taken out in every 10 h and measured through a spectrophotometer.

2.8. Stability and Long-Term Service Tests

In the acid and alkali corrosion resistance test, HCl and NaOH were utilized separately in preparing the acid and alkali solution with different pH values. The coating was soaked in each solution for 8 h, and the WCA of the coating surface was tested after removing the solution.

In the wear test, the PDMS/M-TiO₂ coated glass was placed below the 1000-grit SiC sandpaper face up, and a 200 g of counterweight was placed on the sandpaper. In each cycle, the counterweight was dragged from the left

side to the right side of the glass and then returned to the beginning position. After then, the water contact angle of the coating was measured to evaluated the durability.

3. Results and Discussion

3.1. Effect of Graft on the Morphology, Surface State, and Wetting Properties of TiO₂

Fourier-transform infrared (FT-IR) transmittance spectra were firstly carried out to investigate the effect of surface modification on the exposed functional groups of pristine TiO₂. As shown in Figure 2a, bare TiO₂ displayed four peaks in the measured region. The broad weak absorption peak centered at 3400 cm⁻¹ and the sharp peak centered at 1637 cm⁻¹ are respectively the O-H stretching vibration and H-O-H bending vibration of surface-adsorbed water molecules. The characteristic peak at 793 cm⁻¹ and 434 cm⁻¹ are respectively the stretching vibration of Ti-O bonds and Ti-O-Ti bonds, which are the fingerprint signal of TiO₂ [11,15]. No organic absorption bands are observed for pure TiO₂, confirming the absence of organosilicon impurities in the raw material. For pure PDMS, two distinct peaks at 2966 cm⁻¹ and 2910 cm⁻¹ correspond to the asymmetric and symmetric C-H stretching vibrations of methyl groups (-CH₃) in Si-CH₃ moieties. The peaks at 1413 cm⁻¹ and 1260 cm⁻¹ are the C-H bending and symmetric deformation vibrations of -Si(CH₃)₂ groups. The strong double peaks at 1069 cm⁻¹ and 1012 cm⁻¹ arise from the asymmetric stretching vibration of Si-O-Si backbones. The absorption at 790 cm⁻¹ is characteristic of Si-C stretching vibration from methylsilane groups [3,34,37]. All characteristic bands perfectly match the standard FT-IR signatures of polydimethylsiloxane, verifying the intact molecular structure of PDMS. In M-TiO₂, the coexistence of characteristic peaks belonging to both TiO₂ and PDMS can be observed, demonstrating the successful grafting of PDMS on TiO₂ NPs. Compared to PDMS, the intensity of the peaks at 1069 and 1012 cm⁻¹ in M-TiO₂ undergone a reversal, which may suggest the change of local environment in Si-O bonds after grafting PDMS onto TiO₂. In order to provide more evidence for the graft of PDMS, the magnified FTIR spectrum was displayed in Figure S1a (Supporting Information). Compared with pure PDMS, the signal peaks corresponding to the Si-O-Si and Si-CH₃ vibration of PDMS shift to higher wave numbers in M-TiO₂, indicating that there is a strong interaction between PDMS and TiO₂, rather than a simply physical mixture. Moreover, from Figure S1b, PM-TiO₂ depicted similar FTIR signal to that of pure TiO₂, indicating that the physically mixed PDMS can be totally removed by n-hexane. By contrast, for M-TiO₂, although the signal of TiO₂ was much higher, the characteristic vibrations of PDMS could still be observed. These results fully demonstrate the successful grafting of PDMS on the surface of TiO₂.

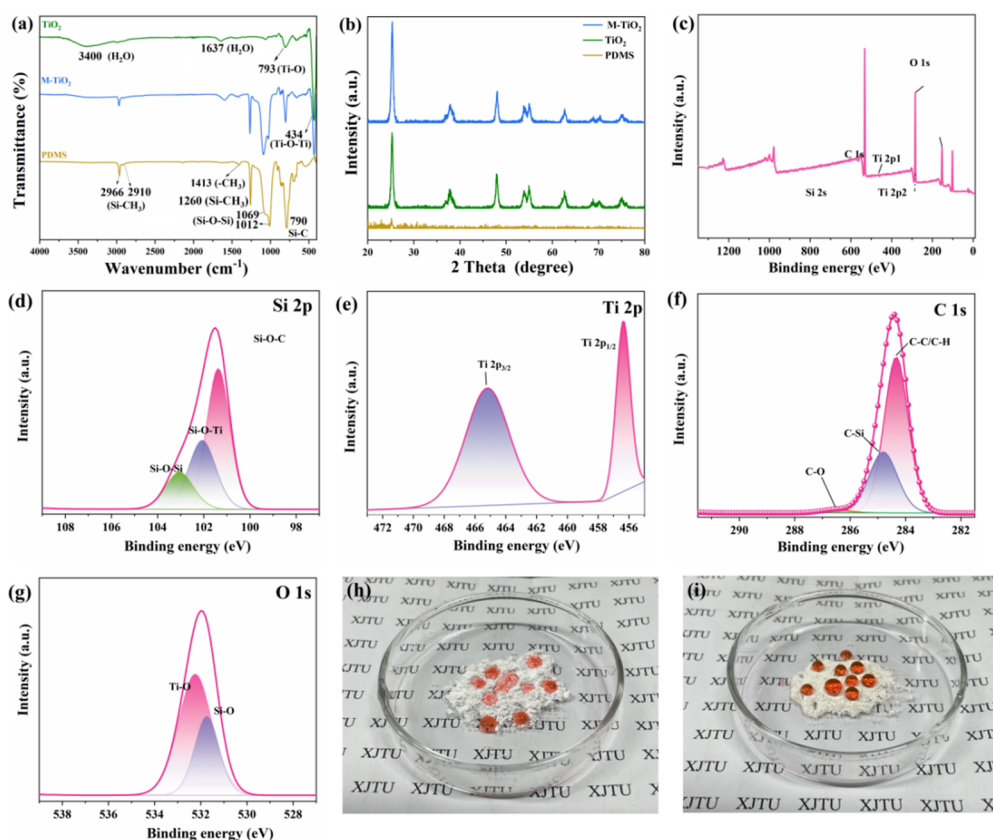


Figure 2. (a) FTIR spectra and (b) XRD patterns of TiO₂, M-TiO₂ and PDMS. (c–g) are the XPS spectra of the PDMS/M-TiO₂ mixture. (h,i) are the digital photos of the wetting behavior of Rhodamine B on TiO₂ and M-TiO₂ respectively.

Figure 2b displayed the XRD spectra of the samples. Pure TiO_2 displayed the characteristic signal of anatase TiO_2 [4], M- TiO_2 displayed similar diffraction signals to that of pure TiO_2 , indicating that the surface graft has barely effect on the crystal structure of TiO_2 . From Figure 2c–g, the coexistence of C, O, Si and Ti elements identified the modification of PDMS on TiO_2 . The peak belonging to the Si-O-Ti bond can be observed from the Si 2p signal from PDMS/M- TiO_2 (Figure 2d) and M- TiO_2 (Figure S2), confirming the formation of chemical bonding at the interface [1,3]. Moreover, the percentage of Si-O-Ti in M- TiO_2 is much higher than that in PDMS/M- TiO_2 , which can be ascribed to the higher proportion of grafted PDMS because the remove of free PDMS from M- TiO_2 . The formation of Si-O-Ti covalent bonds at the interface not only confirms successful PDMS grafting but also serves as the chemical foundation for the dynamic synergy between photocatalysis and hydrophobicity, as this robust linkage prevents phase separation and mitigates photo-oxidative degradation of the polymer matrix. From Figure 2h,i, it can be observed that the surface grafting modification efficiently improves the surface hydrophobicity of TiO_2 NPs. As a result, the M- TiO_2 displayed good dispersion stability in n-hexane (Figure S3) and facilitated the uniform formation of coating on the surface of glass (Figure S4).

Figure 3 exhibited the micro-morphology of different coatings. Pure PDMS coating displayed relative smooth surface (Figure 3a). In comparison, the addition of TiO_2 NPs resulted in increased roughness of the coating (Figure 3b). However, due to the poor compatibility of hydrophilic TiO_2 in PDMS solution, only a small amount of surface protrusions can be observed in the sample. After TiO_2 was grafted with PDMS, the uniform agglomerated structure formed due to the adhesive effect of PDMS (Figure 3c,d), which significantly increasing the surface roughness of the material [1,8,13]. From Figure 3e–g, a large amount of agglomerated TiO_2 with the size of about 200 nm dispersed uniformly in the PMDS substrate, which lays the foundation for enhancing the super-hydrophobicity of the coating. From Figures 3h and S5, the average thickness of the coatings gradually rises as the increased loading amount of TiO_2 . When the loading percentage of TiO_2 is 0.75 wt.%, the thickness of the coating is about 2.1 μm with rough surface. From Figure 3i,j, a large number of small NPs with the size of ~20 nm can be observed, the recognized lattice spacing was about 0.352 nm, corresponding to the (101) crystal plane of anatase TiO_2 . This is consistent with the results observed by XRD. PDMS can barely found from the TEM images, this may be due to the low content of PDMS grafted on the surface of TiO_2 . In addition, in the EDS mapping images (Figure 3k), the existence of PDMS in the M- TiO_2 can be confirmed by the detected Si and O elements, confirming the successfully graft of PDMS on the surface of TiO_2 .

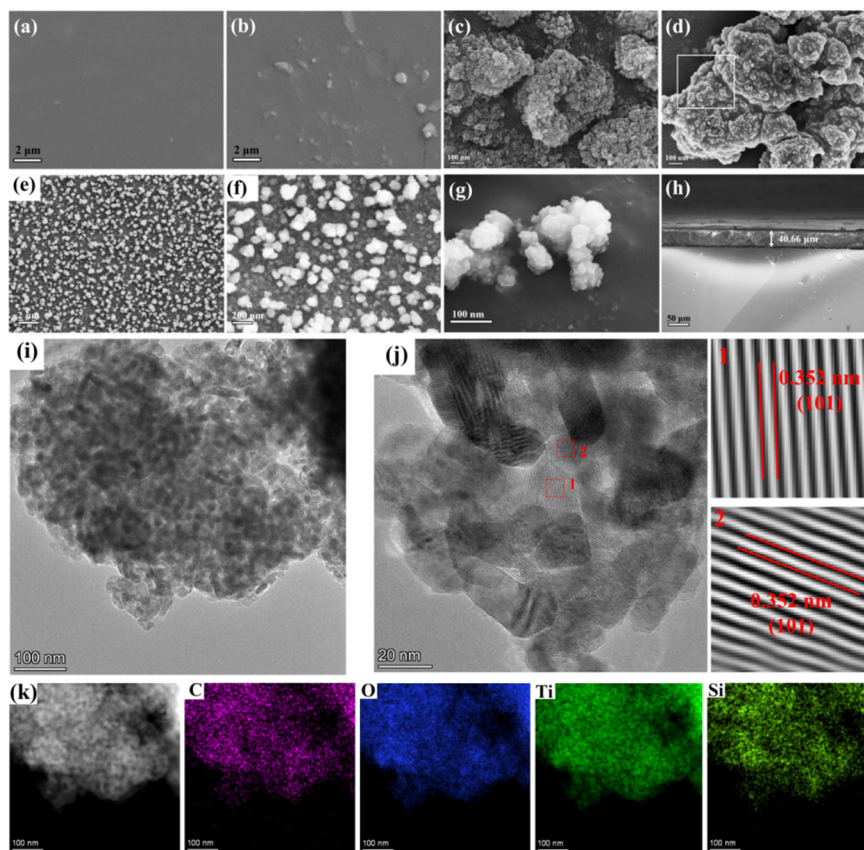


Figure 3. (a,b) are respectively the SEM images of PDMS coating and PDMS/ TiO_2 coating, (c,d) are the SEM images of M- TiO_2 under different magnifications, (e–g) are SEM images of PDMS/M- TiO_2 coating under different

magnifications. (h) is the SEM image in the oblique angle of the glass that reflecting the thickness of the coating. (i,j) are the TEM images of the PDMS/M-TiO₂ coating obtained. (k) is the HADDF and corresponding element dispersion mappings of C, O, Ti, and Si elements.

3.2. Water Contact Angles and Light Transmittance Properties of the Coating

The effect of added TiO₂ on the WCA of PDMS was studied by adding different amount of TiO₂ (or M-TiO₂) in the preparation of self-cleaning coating. As shown in Figure 4a, pure PDMS coating displayed hydrophobicity with the WCA of about 111.71°. With the increased addition amount of pristine TiO₂, the WCA of the coating increases firstly, and then gradually decreases. When a small amount of TiO₂ was introduced, the particles agglomerate on the surface of the coating and form bulges, which increases the hydrophobicity of the coating. When the amount of TiO₂ increased to 0.25–0.5 wt.%, the hydrophobic performance of the coating gradually increased to 113.65°. When the content of TiO₂ exceeds 0.5 wt.%, the hydrophilicity of TiO₂ comes into play, which counteracts the hydrophobicity of PDMS and reduces the hydrophobicity of the coating. The WCA of the coating decreased to 89.40° with the percentage of TiO₂ increased to 1.25%, indicating that the hydrophobicity of PDMS was gradually broken by the TiO₂. By contrast, when the M-TiO₂ was added into PDMS solution, a gradually increased WCA from 125.85° to 163.01° was observed with the percentage of M-TiO₂ increased from 0.25% to 1.25%. The grafting of PDMS alters the surface wettability characteristics of TiO₂, minimum the loss of hydrophobicity of the coating during the addition of TiO₂. Simultaneously, during the grafting process, the adhesive effect of PDMS induces the agglomeration of TiO₂ NPs into larger-sized aggregates with enhanced roughness, thereby improving the hydrophobic performance of the coating. This contrasting trend clearly illustrates how surface grafting resolves the wettability conflict: the hydrophobic PDMS shell on M-TiO₂ ensures that increasing the photocatalyst loading contributes positively to surface repellency, rather than deteriorating it, a key prerequisite for achieving dynamic synergy in the composite system. Figure 4b–e displayed the contact angle photos of water droplets on different surface. When the water was directly dropped on the bare glass, the WCA was about 56.54°, while the WCA of pristine TiO₂ was about 26.38°. The introduction of coating significantly changes the surface wetting state of the glass, and the PDMS/M-TiO₂ coating resulted in the much larger WCA than that of PDMS coating, indicating the efficiency of surface grafted TiO₂ in improving the hydrophobicity of the coating.

The light transmittance of architectural glass is a crucial issue that must be considered in the preparation of coating. To this end, the impact of different types and additions amount of TiO₂ on the light transmittance of the coating was investigated. As shown in Figure 4f,g, the light transmittance of PDMS/M-TiO₂ coating and PDMS/TiO₂ coating both decrease with the increased percentage of TiO₂, while the light transmittance of PDMS/M-TiO₂ coating is slightly better than that of PDMS/TiO₂ coating, this maybe because the surface graft of TiO₂ benefits to decrease the light scattering caused by interface defects and pores between TiO₂ and PDMS. It should be noted that, in Figure 4f, the light transmittance decreased to 75% when the loading percentage of TiO₂ in PDMS-M-TiO₂ coating was 0.75%, which is the average transmittance across the entire wavelength range from ultraviolet to visible light. However, from Figure S6, the most significant light transmittance is concentrated in the ultraviolet wavelength range before 400 nm. Such reduced transmittance originates from strong UV absorption by TiO₂ nanoparticles, which enables effective excitation of the photocatalyst and subsequent photocatalytic reactions. In contrast, only a slight transmittance reduction to 87% is observed within the visible region (420–780 nm), suggesting negligible loss in the visual transparency of the glass. This optical behavior agrees with the digital photographs in Figure 4g, in which the attenuation of light absorption remains visually acceptable when the addition percentage of TiO₂ is no higher than 0.75 wt.%.

Based on above results, it can be concluded that the construction of Si-O-Ti bonding between PDMS and TiO₂ efficiently improves the dispersion of TiO₂ in the sol, enhances the roughness of the film surface and thereby facilitates the formation of an ultra-hydrophobic surface, as well as reducing the scattering loss of light at the interface due to the improved interfacial compatibility.

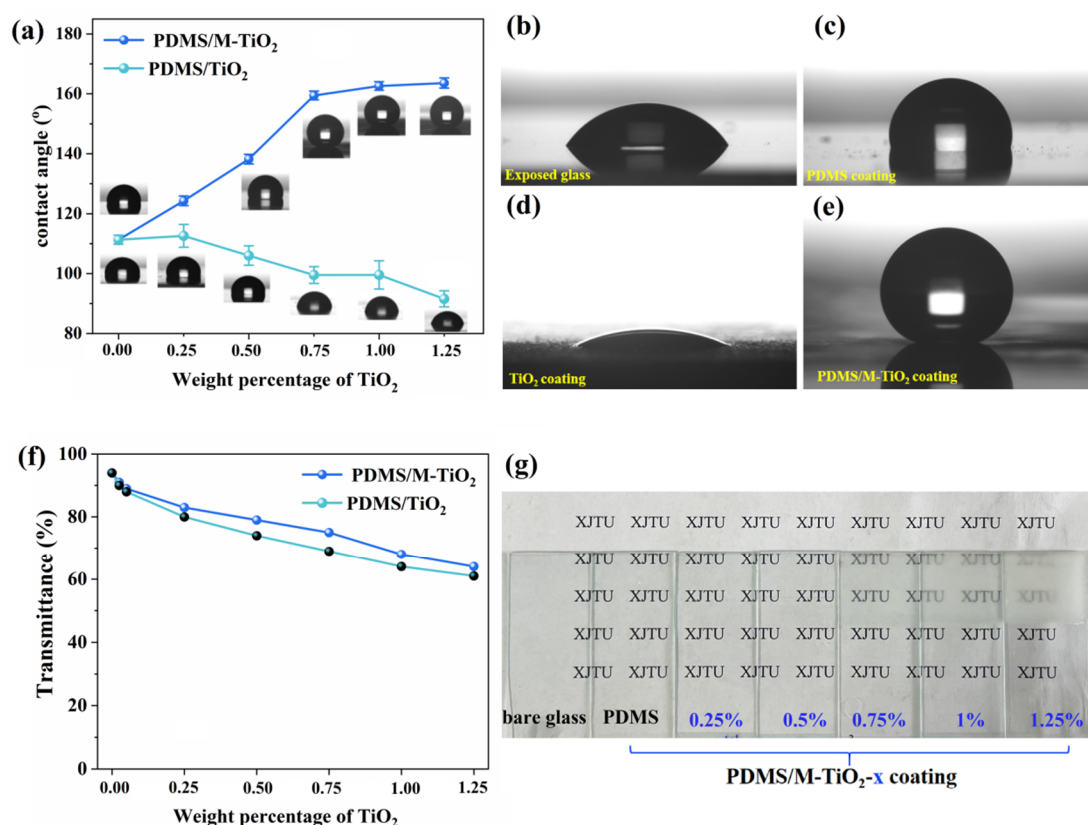


Figure 4. (a) WCA of PDMS/TiO₂ and PDMS/M-TiO₂ coatings with different adding percentage of TiO₂ in the mixture (The error bars in all figures represent standard deviation (SD) of several independent parallel coating samples prepared with same parameter). (b–e) are the contact angle photos of water droplets on different surface. (f) light transmittance of PDMS/TiO₂ and PDMS/M-TiO₂ coatings with different adding percentage of TiO₂, (g) digital photos of the bare glass and the PDMS/M-TiO₂ coated glass with different adding percentage of TiO₂.

3.3. Self-Cleaning Properties of the Coating

Taking the requirements of glass building materials for the WCA and its light transmittance of the coating into account, the coating with M-TiO₂ addition percentage of 0.75 wt.% was utilized in all the subsequent research. Based on the investigation to the WCA of the coatings, the self-cleaning effect to water and dust were tested over the bare glass and the PDMS/M-TiO₂ coated glass. All the glass was putted with a slant angle of 30°. In order to enhance the visualization of the experiment, methylene blue (MB) solution was utilized to investigate the adhesion effect of water droplets on the bare glass and the PDMS/M-TiO₂ coating. MB was dripped on the surfaces of two clean glass sheets with different surface. It can be seen that (Figure 5a), when the MB was dropped on bare glass, the droplet slowly slid down to the bottom of the glass, with a part of liquid adhere on the glass. By contrast, on the surface of PDMS/M-TiO₂ coating (Figure 5b), the droplets slid down to the bottom and eventually settled on the glass petri dish, and no liquid residue or water print was observed on the coating, reflecting super self-cleaning effect of the hydrophobic coating.

Furthermore, dust was thrown on the surface of the glass to investigate the cleaning effect of the coating on powdered solid contaminants. Water was slowly dripped onto the glass through a rubber dropper to simulate the washing effect of rain on glass buildings. As shown in Figure 5c,d, after the droplets roll off the surface of PDMS/M-TiO₂ coating, all the dust on the coating is taken away and slides away from the glass sheet. However, on the bare glass, the added water and dust adhere and spread out into a film on the surface on the glass, which cannot be completely removed even under the rinsing of a large number of water droplets. All the tests demonstrated that the introduction of PDMS/M-TiO₂ coating efficiently protect the glass from the pollution of water and solid dusts (Video S1, Supporting Information).

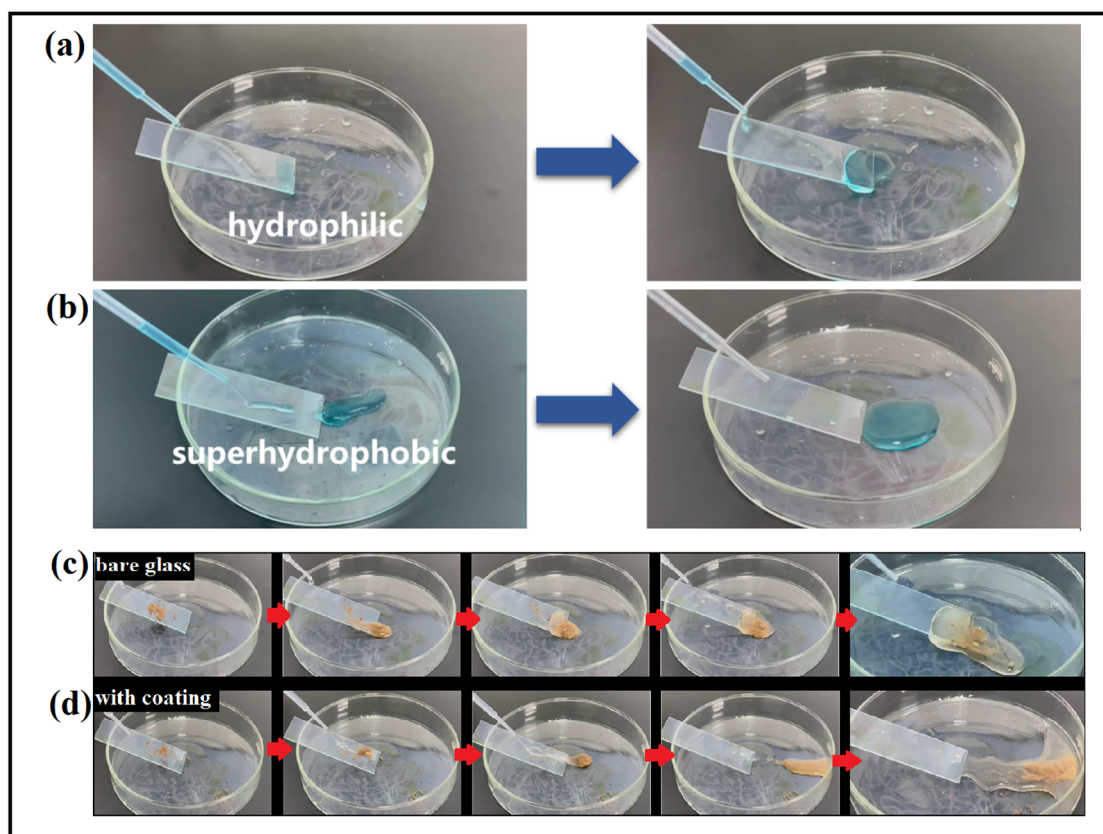


Figure 5. (a,b) are respectively the rolling-off behavior of droplets from hydrophilic and hydrophobic glass surfaces. (c,d) are respectively the removal behavior of dust from the exposed glass and the coated glass surfaces under the simulated effect of rainwater erosion.

In order to verify the photocatalytic performance of PDMS/M-TiO₂ coating, 5 ppm of RhB solution was utilized as an indicator and the tests were carried out on the PDMS/M-TiO₂ coating with different adding percentage of TiO₂. A 12 W ultraviolet lamp was utilized to provide light irradiation. As shown in Figure 6a, nearly no degradation of RhB was observed during 85 h of irradiation, indicating that the RhB cannot be denigrated by the PDMS coating. With the addition percentage of TiO₂ increased from 0.25% to 1.25% in the coating, an increased degradation rate of the RhB was observed. In addition, from the digital pictures of RhB solution shown in Figure 6b, a gradually fading of the solution was observed with prolonging of the irradiation time, indicating that the addition of M-TiO₂ in the self-cleaning coating facilitates the degradation of pollution through the photocatalytic process. After 85 h of irradiation, about 92% of RhB solution was degraded (Figure 6c). The slow degradation behavior is due to the low content of TiO₂ and the low power of ultraviolet lamp. The removal of organic pollution is mainly driven by the photocatalytic effect of TiO₂ nanoparticles. Upon ultraviolet irradiation, TiO₂ nanoparticles absorb photon energy to generate photogenerated electron-hole pairs. The separated electrons and holes migrate to the catalyst surface and subsequently react with adsorbed water and atmospheric oxygen to produce abundant reactive oxygen species (ROS), including hydroxyl radicals ($\cdot\text{OH}$) and superoxide radicals ($\cdot\text{O}_2^-$). These highly oxidative free radicals can continuously break the chemical bonds of organic contaminants adhered to the coating surface, gradually decomposing macromolecular pollutants into small intermediates, and finally mineralizing them into carbon dioxide and water.

Figure 6d displayed the pseudo-first-order kinetic fitting curves for photocatalytic degradation of RhB over PDMS/M-TiO₂ coatings with different loading percentage of M-TiO₂. As observed, the slope of kinetic fitting lines increases markedly when the loading percentage of M-TiO₂ rises from 0.25 wt.% to 0.5 wt.%, demonstrating an obvious enhanced photocatalytic degradation activity. When the loading percentage of M-TiO₂ exceeds 1.0 wt.%, a continuous decreased photocatalytic kinetics was observed, which is consistent with the light transmittance test results of the coatings. When the loading amount of M-TiO₂ was higher than 0.5 wt.%, the light transmittance of the coatings decreases drastically. Partial of the M-TiO₂ NPs embedded deeply inside the coating cannot be sufficiently excited to participate the photo-degradation reaction, thereby resulting in decreased apparent reaction kinetics. The catalytic kinetics results are directly linked to the light transmission performance of the coatings, which provides mechanistic evidence that the degradation of RhB on the PDMS/M-TiO₂ coated glass is a

photocatalytic self-cleaning process dominated by M-TiO₂ in the coating, rather than the spontaneous decomposition of the pollutant exposed under light. The volcano-shaped trend of apparent reaction kinetics with increasing M-TiO₂ loading reflects a dynamic balance between photocatalyst availability and light penetration.

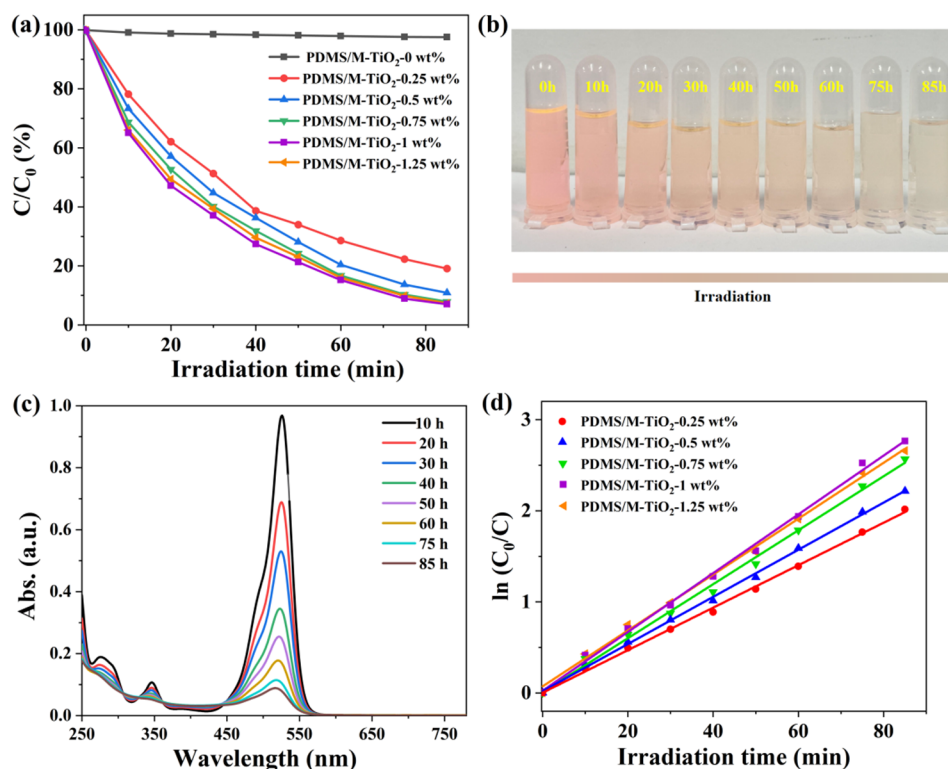


Figure 6. (a) the time related degradation of RhB over different coatings. (b,c) are respectively the digital picture and absorption curves of RhB after different irradiation time. (d) RhB degradation kinetics over different coatings.

In order to further investigate the accessibility of the pollution solution to the photocatalyst surface, the variations in the WCA of the coating under different conditions were monitored. As shown in Figure 7, under 365 nm of UV-light irradiation, the WCA of the coating gradually decreases with prolonged illumination, leading to the transformation of the coating from superhydrophobic to hydrophilic state. Numerous studies have demonstrated that the reduction of WCA originates from the Ti⁴⁺-to-Ti³⁺ valence transition of TiO₂ triggered by UV excitation, which facilitates the dissociation of surface water molecules to hydrophilic -OH groups [38,39]. As a result, the decreased WCA ensures sufficient contact between the photocatalyst within the coating and organic dyes, and finally contributing to the photocatalytic self-cleaning reaction. After then, when exposed in dark environment, the WCA gradually recovered to a superhydrophobic state, this reversible surface change indicating the dynamic balance between the superhydrophobic self-cleaning behavior and the photocatalytic self-cleaning behavior of the coating.

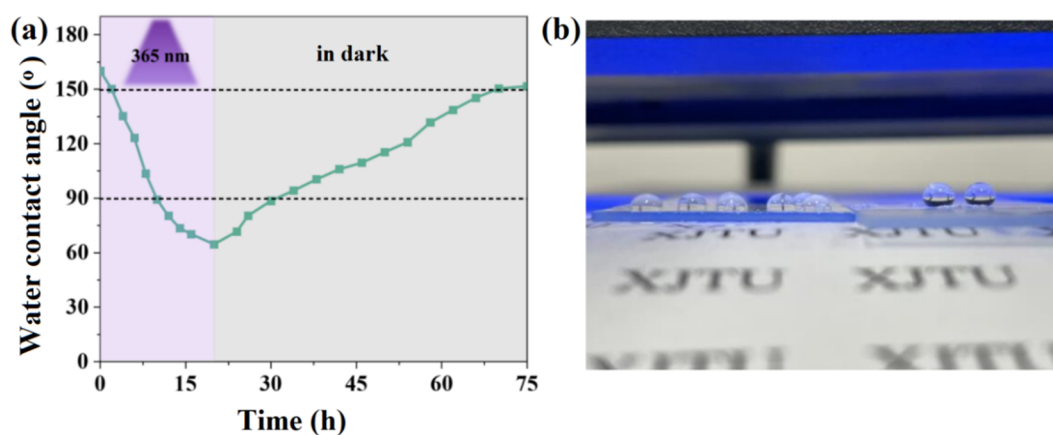


Figure 7. (a) WAC of the coating under different conditions, (b) Digital photos of the surface wetting state of the coating under light (left) and dark (right) conditions.

3.4. Durability and Outdoors Service Stability of the Coating

In order to evaluate the durability of the fabricated PDMS/M-TiO₂ coating in actual environments, surface WCA tests under different pH conditions and anti-wear performance tests were conducted. As shown in Figure 8a, compared with the neutral solution, when placed in acidic or alkaline solutions, a slightly decreased WCA of the coating was observed, but the minimum contact angle is still higher than 140°, demonstrating excellent hydrophobic properties (Figure S7 shows the digital photos of the wettability of RhB on the surface of PDMS/M-TiO₂ coating). This good durability of PDMS/M-TiO₂ coating in different solution may be due to the inherent chemical inertness of the Si-O bond and the acid-base resistance of the PDMS coating. In addition, in the anti-wear performance test (Figures 8b,c), by applying weights to the sandpaper and moved to grind the coating surface, a simulated surface wear and tear environment was created. After six cycles of test, the coating still maintained a WCA of 150°. The sustained hydrophobic performance under aggressive conditions underscores the robustness of the Si-O-Ti interfacial linkages, which not only preserve the photocatalytic activity of TiO₂ but also maintain the structural integrity of the PDMS matrix against environmental attacks. This durability is the essential manifestation of a truly dynamic synergy, one that persists over extended service rather than fading after initial use.

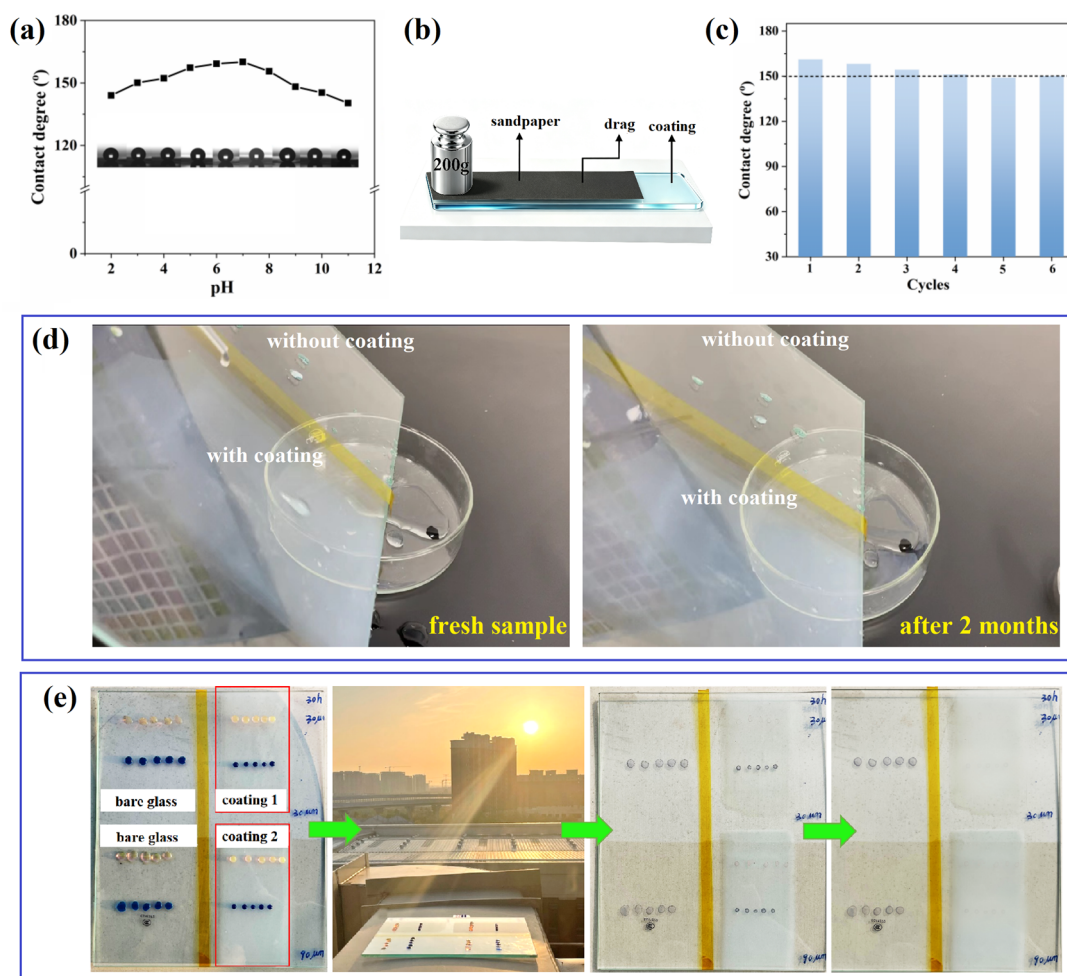


Figure 8. (a) change of the WCA on the surface of PDMS/M-TiO₂ coating in different pH values (three times repetition). (b) Schematic diagram of the wear test. (c) WCA of PDMS/M-TiO₂ coating before and after 6 cycles of tests (three times repetition). (d) self-cleaning test of the PDMS/M-TiO₂ coating placed outdoors before and after two months. (e) the degradation of RhB and MB solution on the bare glass and coated glass under natural sunlight for 2 months.

Figure 8d displayed the adhere behavior of MB solution on the surface of bare glass and PDMS/M-TiO₂ coated glass before and after 2 months of service outdoors. After 2 months of exposure of the coating, the water droplets also slide rapidly off the surface of the coating, demonstrating the good super hydrophobic self-cleaning stability of the coating. In addition, when the PDMS/M-TiO₂ coated glass was left outdoors and exposed to the sunlight for 2 months, an efficiently degradation of RhB and MB dyes was also observed over the PDMS/M-TiO₂ coated glass (Figure 8e), confirming the good photocatalytic self-cleaning stability of the coating. In addition,

when exposed outdoors, the MB can be degraded after 50 min of sunlight irradiation with the immersion of PDMS/M-TiO₂ coating (Figure S8), demonstrating their good potential in practical application.

Collectively, the results presented above establish a coherent picture of dynamic synergy in the PDMS/M-TiO₂ coating system. The in-situ grafting strategy creates a covalent interface that simultaneously (i) promotes uniform dispersion of TiO₂ in the hydrophobic matrix, (ii) enhances surface roughness for super-hydrophobicity, (iii) preserves the photocatalytic activity of anatase TiO₂, and (iv) protects the PDMS matrix from photo-oxidative degradation. These four functions are not independent but interconnected through the Si-O-Ti interfacial chemistry, enabling the coating to dynamically respond to different types of contaminants, physical dust via water rolling, and organic stains via photocatalytic decomposition, while maintaining its performance under prolonged outdoor service. It is anticipated that this interface-engineering paradigm can be extended to other photocatalyst/polymer systems for diverse self-cleaning applications.

The photocatalytic performance of the coating after durability tests was investigated by putting the after-used coating into a 40 mL of 5 mg/L Sudan III solution and irradiated under 12 W of UV light (Figure S9). The results shown that the Sudan III can be basically degraded after 22 h of irradiation, indicating that the photocatalytic activity of the coating can be maintained during the durability tests.

4. Conclusions

In conclusion, based on the hydrophobic grafting modification of TiO₂, this work successfully developed a dual-functional self-cleaning coating that exhibiting both super-hydrophobicity and photocatalytic activity by integrating TiO₂ photocatalyst with hydrophobic PDMS together. The surface grafting effectively overcame the surface compatibility challenge between hydrophilic TiO₂ and hydrophobic PDMS in the composite system and facilitated the uniform aggregation of TiO₂ NPs. The resulting coating exhibited high contact angle, good light transmittance, excellent self-cleaning performance, as well long-term service stability. Benefiting from the mild, low-cost modification and dip-coating process, this work provides a practical route for fabricating long-life self-cleaning coatings for architectural glass.

Supplementary Materials

The additional data and information can be downloaded at: <https://media.sciltp.com/articles/others/2609290910019239/DN-26060254-SI.zip>. Details of experimental section. Figure S1: Dispersion of TiO₂ (a) and M-TiO₂ (b) in n-hexane solvent before and after 90 s of standing. Figure S2: (a) partial enlarged FTIR spectra of the samples from Figure 2a, (b) FTIR spectra of TiO₂ and M-TiO₂ after washed several times in n-hexane. Figure S3: Preparation of the coating standing through the dipping and pulling method. Figure S4: Average thickness of the coatings with different loading percentage of TiO₂. Figure S5: (a) transmittance of the coatings with different loading percentage of M-TiO₂, (b) comparison of the transmittance at different wavelength. Figure S6: Digital photos of the wettability of RhB on the surface of PDMS/M-TiO₂ coating. Figure S7: Digital photos of MB with a PDMS/M-TiO₂-0.75 wt% coated glass immersed in the solution after 5, 10, 20, 30, 40 and min of irradiation. Figure S8: Digital photos for the degradation of Sudan III with a PDMS/M-TiO₂-0.75 wt% coated glass immersed in the solution. Video R1: The process of water droplets scouring dust off the surface of the PDMS/M-TiO₂ coating. Video R2: The process of water droplets scouring dust off the surface of the PDMS/TiO₂ coating.

Author Contributions

D.M.: Conceptualization, Methodology, Software, Formal analysis, Writing—original draft; L.L.: Investigation, Data curation, Writing—original draft; J.L.: Visualization, Investigation; J.-W.S.: Writing—review & editing, Supervision, Project administration, Funding acquisition, Conceptualization, Formal analysis; D.H.: Software, Validation, Resources. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement

Ethical review and approval were waived for this study, due to the fact that this research did not involve human participants or animal experiments.

Informed Consent Statement

This study does not involve human subjects

Data Availability Statement

Data will be available on request due to privacy.

Conflicts of Interest

The authors declare no conflict of interest.

Use of AI and AI-Assisted Technologies

No AI tools were utilized for this paper.

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