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Research Article

A Comparative Evaluation of TiO_2 Suspension Coating Techniques: A Novel Technique to Achieve Optimal Thickness and Uniformity of Photocatalytic Film

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The most important parameters affecting the efficiency of catalysts for the oxidation of the surrounding materials are considered to be the thickness and uniformity of the catalyst layer. The present method for the determination of thickness used in most studies is the analysis of cross-sectional pictures from SEM imaging. This method, however, has several restrictions. This study proposes a feasible and simple method for evaluating the optimal thickness and uniformity using UVA light transmitted through the samples. Three techniques for catalyst deposition have been investigated in this study using UVA light transmitted through the samples and by measuring the photocatalytic activity. These methods include lowering, dip coating, and spraying, which showed coefficients of variation for the coated catalyst weight of 28.4%, 13.6%, and 3.24%, respectively. The samples from the lowering, dip coating, and spraying techniques showed UVA transmissions of 95.02%, 68.8%, and 15.6%, respectively. The spraying technique displayed the lowest values for both. The estimated removal efficiencies (%RE/mg·cm^{-2}) for lowering, dip coating, and spraying were 18.42, 16.84, and 24.15, respectively. Using these analyses it was determined that the spraying technique yielded the best photocatalytic oxidation performance of the three techniques studied.

1. Introduction

Photocatalysts are a group of nanoscale semiconductor materials that are able to generate free radicals after being stimulated by photons of light at certain wavelengths [1]. The free radicals that are generated cause the photocatalytic oxidation of surrounding materials [2, 3]. A variety of materials possess this capability including TiO_2, ZnO_2, ZrO_2, NiO_2, Fe_2O_3, and hybrid catalysts [3, 4]. Of these materials, TiO_2 has been widely used due to its lower cost and toxicity as well as its higher accessibility [2, 4, 5].

When evaluating the photocatalytic performance of materials in removing compounds in the gas phase, it is necessary for the catalysts to be coated on a fixed [6–8] or mobilised bed [9, 10]. Researchers have applied different techniques for coating the surfaces [1, 11]. One of the simplest methods of coating uses a suspension that includes a prepared photocatalyst [11].

Three different techniques that have been used for this coating method include dip coating and pulling up the substrate at a constant speed (dip coating) [6], dip coating and evaporation of the solvent containing the photocatalyst

(lowering) [12], and the technique of spraying the photocatalyst onto the surface (spraying) [13, 14].

The essential factor in order for these materials to show photocatalytic activity is the potential for all of the photocatalyst molecules to receive light irradiation with an appropriate wavelength. It is important, therefore, to determine the optimal thickness of the film that ensures the penetration of UV light throughout the entire depth of the photocatalyst layer.

In the evaluation of the activity of a photocatalyst such as TiO_2, the performance is usually reported in terms of the removal efficiency per milligram of catalyst in a given area. If the irradiated light penetrates only some parts of the coated layer of a photocatalyst, then only a small portion of the photocatalyst will be active.

If a photocatalyst's performance is measured in terms of removal efficiency per milligram of catalyst per square centimetre of area that is receiving the light ($\%RE/mg^{-1}\cdot cm^{-2}$), the performance will be underestimated. Xianyu et al. have confirmed the influence of thickness on TiO_2 photocatalyst performance and have shown that a thin layer of TiO_2 film with a thickness of 670 nm has a photocatalytic activity only 3.7 times that of films with a 70 nm thickness [15]. In another study conducted by Akhavan, the influence of thickness from 10 to 50 nm on TiO_2 antimicrobial performance was investigated, and the optimum thickness of the TiO_2 coating was 10 nm [16]. Chang et al. has studied the influence of thickness and roughness of doped TiO_2 films on ZnO_2 and found that increasing the thickness of the film from 30 nm to 950 nm increased the roughness [17].

In the study of Bessergenev et al., the large range of the coating film thickness (30 to 2000 nm) illustrates the importance of the thickness and uniformity of the sample to be evaluated as well as its effect on the measured photocatalytic activity [1].

Another feature that can have an effect on the photocatalytic activity is the uniformity of the coating layer on the film. As the uniformity of the coating layer increases, the average catalyst per square centimetre area will be closer to the true value. In the case of complete uniformity of the coating layer, these two values will be equal.

A review of the literature on this subject reveals that different techniques have been used to determine the photocatalyst layer thickness.

In the study conducted by Bessergenev et al, the coating film thickness has been measured by weight [1].

In several studies, Scanning Electron Microscopy (SEM) has been used to examine the thickness of the layer [6, 15, 18–20]. In a study performed by de Groot and de Lega [21], a white-light scanning interferometer was applied to evaluate the surface profile of transparent layers. Comparing the results of this technique with those using Atomic Force Microscopy (AFM), de Groot found this method to be very effective. The white-light scanning interferometer method is a precise optical method that is able to scan surface topography. In a study by Blount, the photocatalyst layer thickness was evaluated using the weight of the coating [22].

Despite the extensive research and presentation of these techniques in assessing and describing the coating layer thickness, it seems that a professional apparatus is needed. It is also important to note that the evaluation of the layer thickness and uniformity is impossible using previously reported methods.

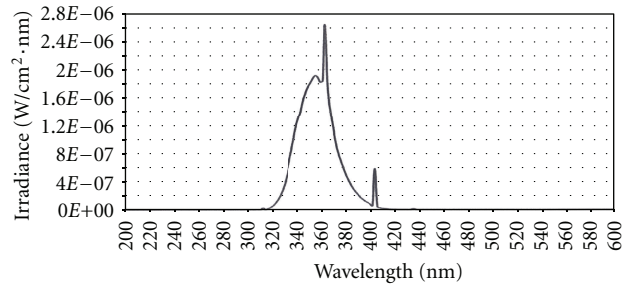


Figure 1: Irradiation spectrum of the UVA lamp used in this study.

One important limitation of testing catalyst samples by SEM is that the sample must be destroyed during testing and cannot be measured for photocatalytic performance after SEM imaging is completed. Furthermore, if results from SEM testing indicate an inappropriate thickness or uniformity, all photocatalytic tests should be repeated.

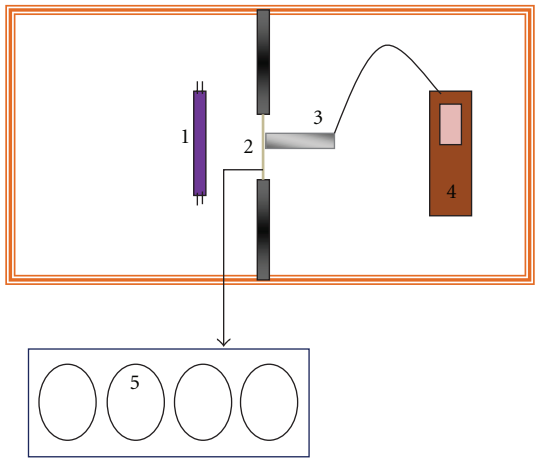
This study aimed to compare three catalyst coating techniques on a flat surface using the suspension method while introducing a testing technique that is innovative, practical, and easy to use to control the uniformity of the surface. Additionally, the method aimed to determine the optimal thickness of photocatalyst samples (the thickness in which the light irradiation is able to most completely penetrate) using UVA transmittance through the samples during the coating stages.

2. Materials and Methods

Since the condition of composing TiO_2 nanoparticles, specially the calcination temperature [23] and crystallization method [24], has been affected on the TiO_2 photocatalytic performance, commercial TiO_2 (P25 with a specific area of $50 \pm 10\,m^2/g$ and an average particle size of 21 nm) was used in this study as well as the gas chromatographic grade methanol (Merck, Germany). The substrate used was flat quartz (Shanghai Puya Quartz Glass Factory, China) with a thickness of 3 mm and dimensions of 2.5 by 7 centimetres. The UVA light source used in this study was an 8 watt lamp made by Sylvania Company, Japan. The irradiation spectrum of the UV lamp is illustrated in Figure 1.

2.1. Catalyst Characterization. Regarding to this matter of fact that all three techniques applied in this research do not cause any changes in crystallized properties of the samples [23, 24], the morphology experiments were carried out on the primary photocatalyst.

The sample's morphology and thickness were investigated using a scanning electron microscope made by SERON Technology (AIS–2100) and Crystallinity of the TiO_2 thin film was determined by X-Ray Diffraction (Bruker, DBADVANCE, Germany, X-Ray Tube Anode: Cu, Filter: Ni).



1. UVA lamp
2. Quartz substrate coated with TiO_2
3. UV detector
4. UV meter
5. UV detector position

Figure 2: Schematic diagram for the UVA transmission measurement apparatus.

The absorbance of the film was measured by Ultraviolet visible diffuse reflectance spectroscopy (Uv-vis-DRS: JASCO V-670, Japan) and The oxidation state of TiO_2 was studied by X-Ray photoelectron spectroscopy (XPS: VG Microtech 8025-Twin anode X-Ray source-Germany).

2.2. Different Coating Techniques. Several coating techniques have been investigated in this study. The coating techniques included dip coating and pulling up the film at a constant speed (dip coating) [6], dip coating and evaporation of the solvent containing the photocatalyst (lowering) [12], and the technique of spraying the photocatalyst onto the surface (spraying) [25]. To avoid varying the TiO_2 concentration in all these experiments, a 5% suspension of TiO_2 in methanol was prepared by ultrasonic mixing for 15 minutes using a homogenizer apparatus (Bandelin, Germany).

All quartz substrates were used after washing twice with distilled water followed by washing with methanol. The substrates were then dried in an autoclave at 120°C overnight. A quartz film was chosen for these experiments because its transparency is conducive to UV illumination [26].

The UVA detector used in these experiments (Laybold, Germany) had a 1 cm diameter, and UVA light was transmitted through the samples at 4 different points on the coated surface. The setup schematic and the points on the surface to be examined in each sample are illustrated in Figure 2.

In the lowering technique [12], the film was placed in a watch glass horizontally and the catalyst solution was added to the point where it covered the surface. After submerging the film, the solution was heated to 80°C until it evaporated to dryness. The underlying substrate was then cleaned and weighed, and the uniformity test was performed

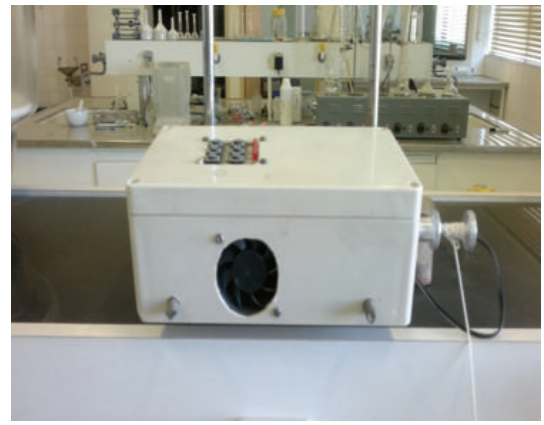


Figure 3: Custom-built apparatus for pulling up the substrate with constant speed in the lowering technique.

by transmitting UVA light through the samples. The coating of the samples was performed only once because light was not transmitted through the samples on more than one occasion.

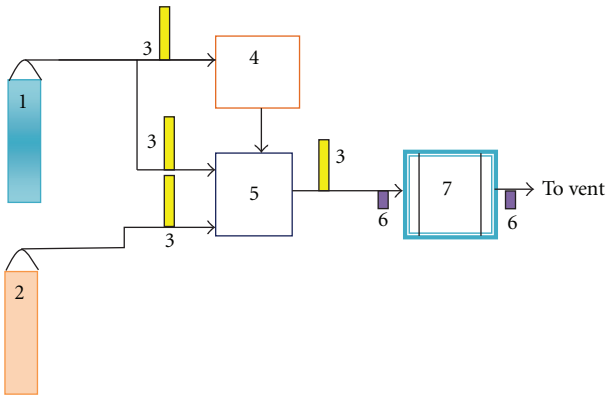
An electromechanical device was designed and built for the dip coating technique [6] (Figure 3) that had the ability of moving the sample at a completely constant rate. The sample in this experiment was pulled out of the photocatalyst solution at a speed of 10 mm/min, followed by removal of the photocatalyst coating on one side of the film. This section of the experiment was repeated and the samples were dried in an autoclave for 15 minutes at a temperature of 100°C. Subsequently, the samples were cooled in desiccators containing silica gel and each one was weighed prior to being examined with the UVA light. The coating technique was repeated until the UVA light transmittance through the coating reached zero at a minimum of one point. In other words, the coating was repeated so that the penetration of the UVA beam was ensured at the four points of the surface underlying the film showing that the catalyst layer thickness was at its optimal level.

The third technique, spraying [25], was completed by generating a catalyst solution and pouring it in a paint spray apparatus. The solution was then sprayed on the substrate surface with a constant speed of 4 cm/s, a flow rate of 25 mL/min, and a pressure of 2 Barr. To meet the desired parameters, the paint sprayer was fixed on a base and an electromechanical apparatus was used to pass the samples under the sprayer at a constant velocity. In this technique, the catalyst sample was dried following each spraying at a temperature of 100°C. The samples were then cooled in the desiccator and weighed before being tested for uniformity using UVA light. The number of spraying events for each sample was chosen using the same criteria as the previous technique to ensure that the catalyst layer thickness was at its optimal level.

2.3. Photocatalytic Activity Measurement. Using each technique separately, five laminates prepared were placed inside the dynamic apparatus made for the evaluation of the

Table 1: Coating frequency, UVA transmittance, and catalyst coating weight results for the lowering technique.

Sample code	Coating frequency	Catalyst weight (mg)	% UVA transmittance in 4 point (reference irradiation 8 W·m^{-2})				Mean	Standard deviation
			1	2	3	4		
G	1	39	12.4	16.1	7.2	5.5	7.8	3.1
K	1	67.3	0	6.1	2.3	4.4	3.2	2.64
E	1	84.8	0	0	3.5	1.8	1.3	1.68
P	1	83.1	2.6	1.4	0	0	1	1.25
L	1	58.6	9.8	1.1	3.4	6.9	5.3	3.83



1. Zero air
2. Xylene in N_2
3. Flow meter
4. Temperature and humidity unit control
5. Mixing chamber
6. Sampling probe
7. Photocatalytic reactor

Figure 4: Schematic diagram of the photocatalytic activity test apparatus for the determination of xylene removal efficiency for the samples prepared by the lowering, dip coating, and spraying techniques.

photocatalytic performance of removing xylene from an air stream. The schematic design of this apparatus is presented in Figure 4. The examination was conducted using the 8 watt UVA lamps used in the first part of the research and measuring the xylene concentration in both the input (C_{in}) and the output (C_{out}) of the reactor using a PID detector. Using this method, the removal efficiency of xylene was obtained from an initial concentration of 30 ppm, an air flow rate of 220 mL/min, and relative humidity of 50%. Removal efficiency is calculated by the following equation:

$$\text{Removal efficiency (RE\%)} = \frac{C_{in} - C_{out}}{C_{in}} \times 100. \quad (1)$$

The PID system performance has been confirmed in another study [27]. The results from each test are reported in terms of removal efficiency of the photocatalyst in mg/cm^2 of each sample.

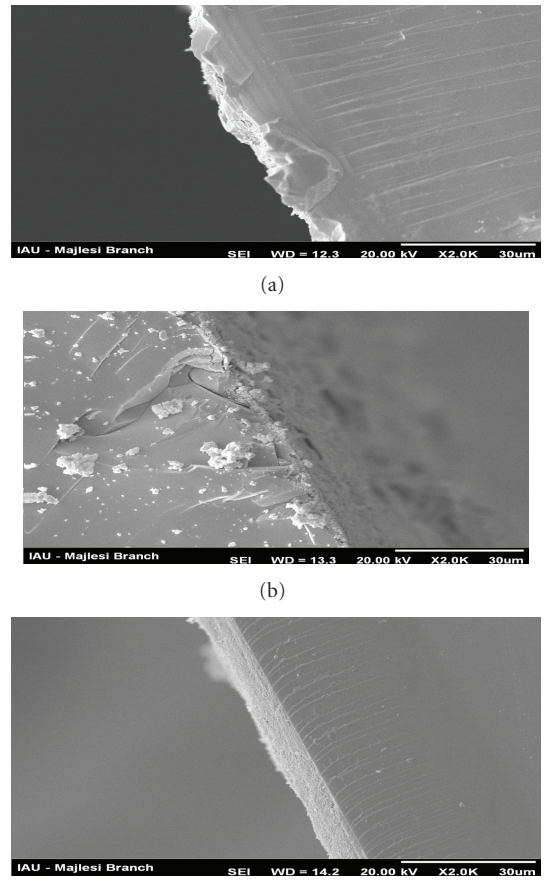


Figure 5: Scanning electron microscopy of the samples prepared by the lowering (a), dip coating (b), and spraying techniques (c).

3. Results

3.1. Sample Characterization. The cross-sectional images from the SEM are illustrated in Figure 5.

X-ray diffraction pattern and Uv-vis absorbance spectra of TiO_2 photocatalyst were illustrated in Figures 6 and 7, respectively.

The composition of TiO_2 thin film was characterized with XPS. The XPS results (Figures 8(a) and 8(b)) show that TiO_2 film deposited on quartz contains Ti, O, and C (18.3%, 65.7%, and 16% resp.).

Table 2: Coating frequency, UVA transmittance, and catalyst coating weight results for the dip coating technique.

Sample code	Coating frequency	Catalyst weight (mg)	% UVA transmittance in 4 point (reference irradiation 8 W·m^{-2})				Mean	Standard deviation
			1	2	3	4		
A	3	62	1.2	1.1	3.2	6.2	2.93	2.38
B	3	75.5	0.5	1	4.5	5.2	2.8	2.39
C	3	53	1.5	2.3	3.1	5.5	3.1	1.73
D	3	72.5	0.6	1.2	2.9	3.5	2.05	1.37
F	3	69.5	0.35	1.7	2.1	3.15	1.83	1.16

Table 3: Coating frequency, UVA transmittance, and catalyst coating weight results for the spraying technique.

Sample code	Coating frequency	Catalyst weight (mg)	% UVA transmittance 1n 4 point (reference irradiation 8 W·m^{-2})				Mean	Standard deviation
			1	2	3	4		
M	10	26	2.3	2.7	3	2.8	2.7	0.3
N	10	26	2.2	2.6	3.2	3	2.75	0.44
O	10	27	2	1.9	2.1	2.2	2.05	0.13
J	10	25	2.7	2.9	3	3	2.9	0.14
Q	10	25	2.9	3.1	3.2	3	3.05	0.13

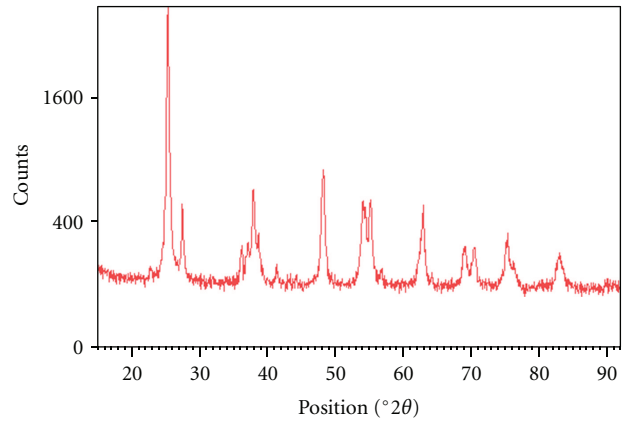


Figure 6: X-ray diffraction pattern of the TiO_2 photocatalyst.

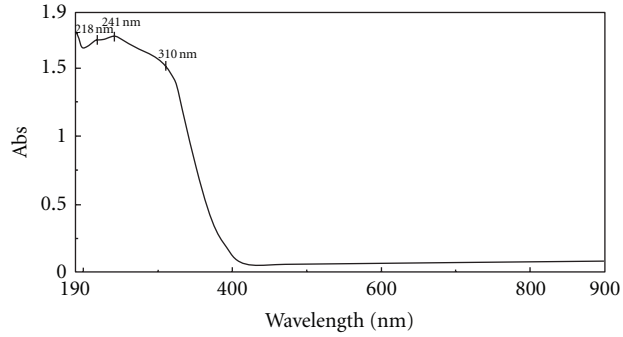


Figure 7: Uv-vis absorbance spectra of TiO_2 photocatalyst thin film.

3.2. Results of the Coating with the Lowering Technique. Five substrates were coated using the lowering technique. The catalyst weight and the percentage of UVA light transmitted through them (as the uniformity index) are presented in Table 1.

Table 1 indicates that the mean weight (standard deviation, SD) of the coated catalyst was 66.56 (±18.9) mg with a coefficient of variation (CV) of 28.4 and the mean percentage (SD) of UVA light transmittance was 3.73% (±3.54%) with a CV of 95.02%.

3.3. Results of the Coating with the Dip Coating Technique. Five substrates were coated using the dip coating technique, each with enough times so that the amount of UVA light transmittance did not reach zero. The catalyst weight (coated on each substrate) and the percentage of UVA light transmittance (as the uniformity index) were measured. The results are presented in Table 2.

We observed that the mean weight (SD) of the catalyst coated on the film was 66.5 (±9.06) mg with a CV of 13.6%. The mean percentage (SD) of UVA light transmittance was 2.54% (±1.75%) and the CV was 68.8%.

3.4. Results of the Coating with the Spraying Technique. Five substrates were coated using the spraying technique. The amount of the UVA transmittance through (as the uniformity index) the coated layer on each one is presented in Table 3.

Using this technique, the average weight of the coated catalyst was 25.8 (±0.84) mg and the coefficient of variation

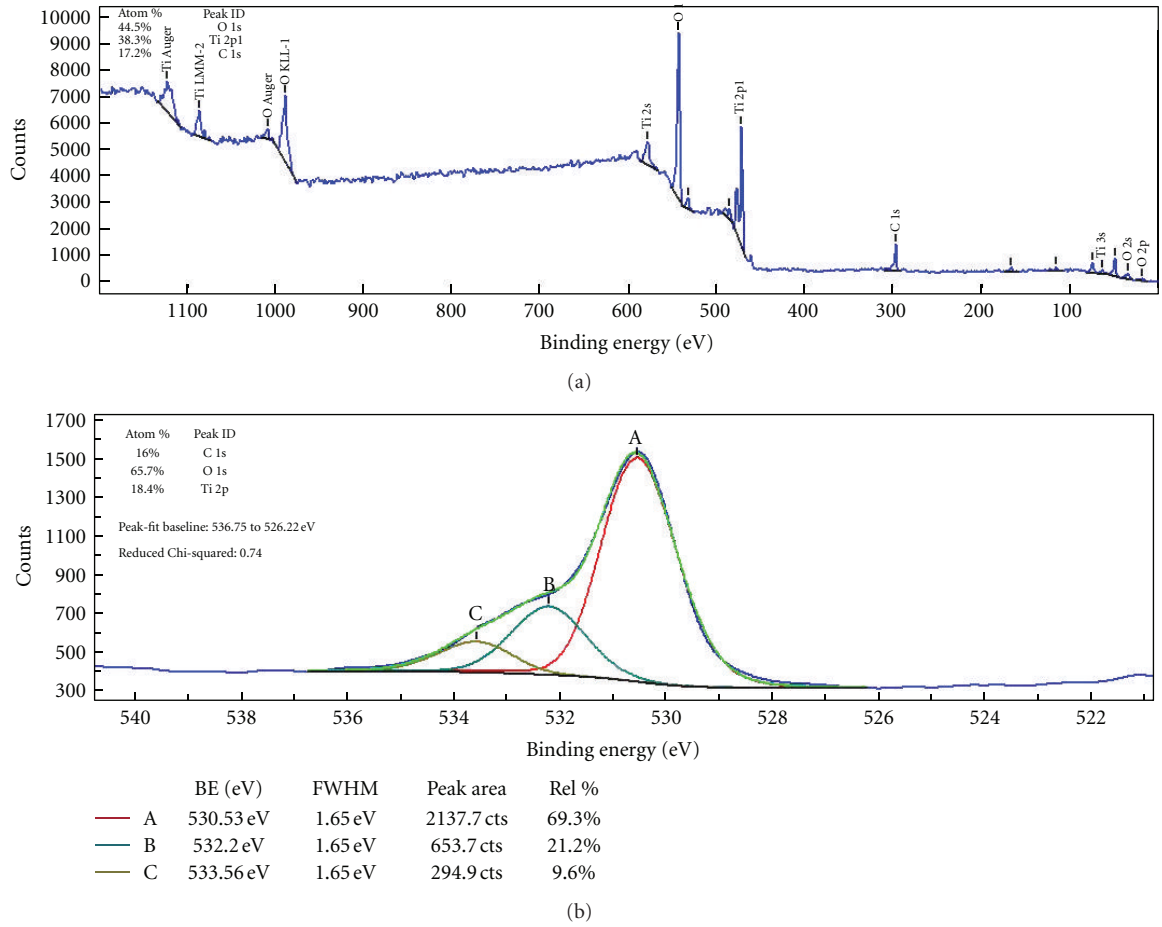


Figure 8: XPS spectra of the TiO_2 thin film (a) and O1s region of the TiO_2 film (b).

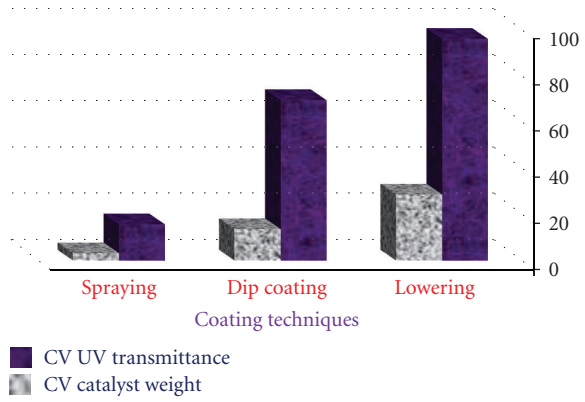


Figure 9: A comparative chart for the coefficient of variation of the catalyst weight coated on a substrate and UVA transmission percentage in the lowering, dip coating, and spraying techniques.

was 3.24%. The average amount of UVA light transmittance was 2.69% (±0.42%) and its coefficient of variation was 15.6%.

The comparison between the CV of the catalyst weight and the UVA light transmittance is illustrated in Figure 9.

3.5. Results of Photocatalytic Activity Tests in 3 Coating Techniques. The results of the photocatalytic activity tests of the samples prepared using the three different techniques show that the removal efficiency (%RE) of xylene from the air was 70%, 64%, and 35.5% for dip coating, lowering, and spraying, respectively. The xylene removal efficiency (%RE/mg·cm^{-2}) estimated for dip coating, lowering, and spraying was 18.42, 16.84, and 24.15, respectively.

4. Discussion

In this study, the commercial P25 photocatalyst was used. The commercial P25 specifications were evaluated by using XRD, XPS, and Uv-vis-DRS. The morphology results of P25 TiO_2 have been confirmed by the results of other studies [28–30]. The SEM images of the samples show the different surface morphologies in the three techniques.

Performance evaluation of photocatalysts for the transformation of gas phase substrates is generally accomplished by coating them on a substrate which is either fixed [6–8] or unfixed [9, 10].

In few studies the interactional effect of the semiconductor film composition method on the thickness has

been considered as well as its effect on the photocatalyst's performance [23, 24].

One of the easiest techniques when performing a fixed-bed study is coating the catalyst using a suspension in which the prepared photocatalyst is dispersed in an appropriate solution and coated on the substrate [11].

Although the catalyst layer thickness has been investigated in several studies [16, 31–36], only a limited number of studies on the layer thickness have been conducted [15–17], and none of them have presented a suitable method to evaluate the uniformity of the photocatalyst layer.

Three coating techniques have been compared in this research in terms of the sample uniformity and optimum thickness regarding the amount of UVA light transmittance. The interaction of the factors affected on the semiconductors composition method, such as the calcination temperature [23] and crystallization method [24], has been investigated in the previous studies. Using commercial P25 TiO_2, these factors can be considered as the controlled factors. So this study indicates the effect of catalyst thickness on the photocatalytic performance.

These techniques include dip coating [6], lowering [12], and spraying [25].

The comparison of the three techniques shows that the method of performing the experiment does not have any influence on the amount of catalyst coated on the substrate or on its uniformity. This feature of the experiment makes it possible to directly compare the photocatalytic properties of the different coating processes.

The coating results from the lowering technique are illustrated in Table 1.

The coefficient of variation related to the percentage of UVA light transmittance through these samples was 95.02%, which is a sign that the thickness changes dramatically using this technique. Blount has confirmed this result in his study [22]. Another feature of this technique is that a very large amount of the photocatalyst is coated on the substrate in just one run, and as a result, UVA light does not pass through some parts of the sample after the first coating. This is a sign of high thickness in some sections of the substrate.

It can be inferred from the results for the lowering technique that it is impossible to control the amount of coated catalyst weight and the thickness of the film layer. In addition, the thickness is more than the optimum level at least in some sections of the coated layer. Because the efficiency of catalysis is reported in removal efficiency per mg of photocatalyst per unit area, the efficiency results for the lowering technique are less than the true value.

The results from the dip coating technique show that the coefficient of variation is lower than that for the lowering technique due to the higher uniformity of the photocatalyst film. However, the results demonstrate that the mean (SD) amount of transmitted UVA light, 0.83% (±0.5%), 1.46% (±0.58%), 3.16% (±0.86%), and 4.71% (±1.32%) for points 1 through 4, respectively, is not equal at the evaluated points. These differences are due to the nature of the method in which the sample is removed vertically from the suspension containing the catalyst. This may cause increased thickness in the lower parts of the substrate. Bennani also has confirmed this point in his study [19].

The results from the spraying technique show the lowest coated catalyst weight after 10 spraying processes. In other words, this technique has the potential to accurately coat a layer of catalyst at the optimum level. The coefficient of variation of this technique was 15.6%, which is lower than those for the other two techniques (dip coating had a CV = 68.8%, and lowering had a CV = 95.02%).

The SEM images of the coating profiles (illustrated in Figure 6) confirm the hypothesis that choosing the optimal thickness seems possible.

The results from the examination of the photocatalytic performance of the samples prepared by the three different techniques in removing xylene from the air stream indicate that the maximum removal efficiency (in $\%RE/mg\cdot cm^{-2}$) is achieved with samples from the spraying technique. The spraying technique also provides the lowest thickness. These findings agree with those from the studies conducted by Xianyu et al. [15] and Shang et al. [37].

Akhavan also has illustrated similar conclusions about the effect of thickness on the antimicrobial function of TiO_2 [16]. In the study performed by Bessergenev et al, the effect of physical and chemical properties as well as the thickness has been investigated on the photocatalytic performance. According to this study, the photocatalytic performance efficiency will de decreased due to the declining of the UV light absorption, in those cases that the photocatalyst layer is not sufficiently thick. Thus, the evaluation of the thickness by measuring the transmitted UV light can prevent any error occurred due to the low thickness of the catalyst [1].

5. Conclusion

The current study indicates that by measuring the UVA light transmittance through the samples, it is possible to choose the optimal thickness of the catalyst coating layer with high accuracy without wasting the sample.

Using this method it is also possible to measure coating uniformity. Compared to other techniques, the spraying technique provided the most uniform coating.

Acknowledgments

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