

# Preparation of a TiO<sub>2</sub>-coated photocatalytic air filter for use with an electrostatic air filter pack for xylene removal

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**ABSTRACT:** This study evaluated surface morphology, mechanical stability, and the effectiveness for xylene removal of coated fibre glass filters and coated high-efficiency particulate air (HEPA) filters for use with an electrostatic air filter pack (ESF pack). Different surface pretreatment methods, two TiO<sub>2</sub> coating suspensions with different amounts of TiO<sub>2</sub> and additive substances, varied baking temperatures, and various combinations between an ESF pack and a coated air filter were investigated. This investigation found that washing the substrates with 0.08 M sodium dodecyl sulphate and 0.01 M sodium fluoride before coating with 5% TiO<sub>2</sub> suspension and then baking at 50 °C for 30 min after coating could yield the highest loadings of 285 g/m<sup>2</sup> for the fibre glass filters and 182 g/m<sup>2</sup> for the HEPA filters. Examination of these filters revealed that the TiO<sub>2</sub> particles were agglomerated and stuck to the fibres as well, resulting in less loading loss. The highest removal efficiency of xylene (in 400–800 ppb) ranged from 41–49% when using a combination of one HEPA filter coated with 5% TiO<sub>2</sub> suspension together with four layers of ESF filters at an air flow rate of 10 m<sup>3</sup>/min and 45 ± 5% RH. The suitable preparation of photocatalytic air filters is of great importance for their practical use, ensuring the stability of the coating and resulting in a product that is safe for human health and friendly to the environment.

**KEYWORDS:** photocatalytic oxidation (PCO), volatile organic compounds (VOCs), surface pretreatment method

## INTRODUCTION

Accumulations of noxious volatile organic compounds (VOCs) are a major cause of poor indoor air quality, resulting in nuisance complaints, adverse health effects, and decreased work performance<sup>1</sup>. This problem could be remedied by the development of alternative air technologies for the removal of indoor VOCs.

Photocatalytic oxidation (PCO) is well-known for its effective performance in indoor air purification by removing gaseous pollutants<sup>2–6</sup>. This reaction can produce hydroxyl and superoxide radicals, resulting in oxidation of the VOCs into CO<sub>2</sub>, water, and some intermediate compounds<sup>5</sup>. Coating a photocatalyst, e.g., commercial P25 TiO<sub>2</sub>, onto a substrate and irradiating it with UV light is the most popular method.

Various studies have been performed using a combination of TiO<sub>2</sub>-coated substrates (mostly glass slides and activated carbons) and other air treatment technologies for VOC removal. For example, PCO in combination with filtration using an activated carbon fibre (ACF), with a plasma-driven photocatalysis

system and ACF, with ozone generators, and with dielectric barrier discharge have all been found to enhance indoor air purification<sup>7–10</sup>. A previous study using TiO<sub>2</sub> to coat an activated carbon filter found that the NO removal increased to 66%, and that benzene, toluene, ethyl benzene, and xylene (BTEX) were removed by more than 60%<sup>11</sup>. In a later study, toluene removal efficiency was increased from 32% to 78% when using a combination of PCO and ACF, and the efficiency was further improved to over 80% when the voltage of the plasma-driven photocatalysis system was increased from 8 to 10 kV<sup>9</sup>. Longer residence time in the reaction zone could cause additional adsorption and desorption processes, resulting in greater removal efficiency of VOCs<sup>9</sup>. However, this combination consumed high energy to enhance the toluene removal efficiency, and that ozone production increased linearly with the increase of applied voltage<sup>12</sup>.

The use of a combination of an electrostatic air filter pack (ESF pack) and PCO has not yet been studied. An ESF pack is commonly used in an air purifier, and adopts the principle of an electrostatic precipitator to

remove particles from the air. Briefly, the instrument has an electrode needle (corona wire) for charging the surrounding air to create an electromagnetic field. The ionized air will transfer the charged ions to particles, which will then migrate to the collecting plates or filters nearby<sup>13</sup>. A previous investigation found that a wire plate with four glass fibre layers (or collecting filters) was the most appropriate with ozone generation of less than 25 ppm. The ozone output is reduced by a larger number of filter layers due to the weaker corona discharges from both the corona wire and the glass fibre layer<sup>14</sup>. Our preliminary study found that an ESF pack could reduce airborne fungi (*Aspergillus niger* and *Penicillium citrinum*) and bacteria (*Staphylococcus epidermidis* and *Bacillus subtilis*) from initial concentrations of 34 000–80 000 cfu/m<sup>3</sup> to the recommended concentration of 500 cfu/m<sup>3</sup> within 30–40 min<sup>15</sup>. However, this air filter had an unsuccessful performance in removing VOCs from air. Hence enhancing the capability of an ESF pack to eliminate VOCs from the air is of great interest and would represent an important advance in alternative air treatment technology.

Before the application of a TiO<sub>2</sub>-coated photocatalytic air filter to an ESF pack, the surface pretreatment method, TiO<sub>2</sub> suspension, and baking temperature were modified to achieve greater stability of the coating and improve performance. Few studies have reported on high-efficiency particulate air (HEPA) or fibre glass filters coated with a TiO<sub>2</sub> photocatalyst, and the effect of varying surface pretreatment methods, TiO<sub>2</sub> suspensions, and baking temperatures in the preparation of the coated filters<sup>16,17</sup>. The surface morphology and physical properties, as well as removal of xylene, which was selected as a representative gaseous pollutant for testing the effectiveness of the combination between an ESF pack and a photocatalytic air filter, are essentially unknown for these coated filters (PCO reaction) within an electromagnetic field. This study compared the loading, surface morphology, pressure drop across the air filters, and loading loss of the filters, using various surface pretreatment methods, coating suspensions, and baking temperatures in the preparation of the photocatalytic air filters. In addition, this investigation compared the xylene degradation in two modified types of ESF packs (three layers or four layers of ESF filters), with a photocatalytic coating on different substrates (HEPA and fibre glass filters). In this study, we found a method to improve the adhesion between TiO<sub>2</sub> and SiO<sub>2</sub> (a glass fibre substrate that is the main component of HEPA and fibre glass filters) by surface pretreatment methods. Physical treatment

(preheating in an oven) and chemical methods, i.e., sodium dodecyl sulphate (SDS) and sodium fluoride (NaF), can be used to develop interaction between the fibre and the matrix. This study prepared TiO<sub>2</sub> coating suspensions with different amounts of TiO<sub>2</sub> and additive substances. The two baking temperatures used (50 and 100 °C) were below and above the melting points of the additive substances: a dispersant (Duramax D-3005, m.p. 0–100 °C) and polyethylene glycol 4000 (PEG 4000, m.p. 53–56 °C). The coated fibre glass and HEPA filters were then investigated for improved surface morphology, mechanical stability and efficacy of xylene removal. The preparation of photocatalytic air filters under suitable conditions is of great importance for their practical use, ensuring the stability of the coating and resulting in a product that is safe for human health and friendly to the environment.

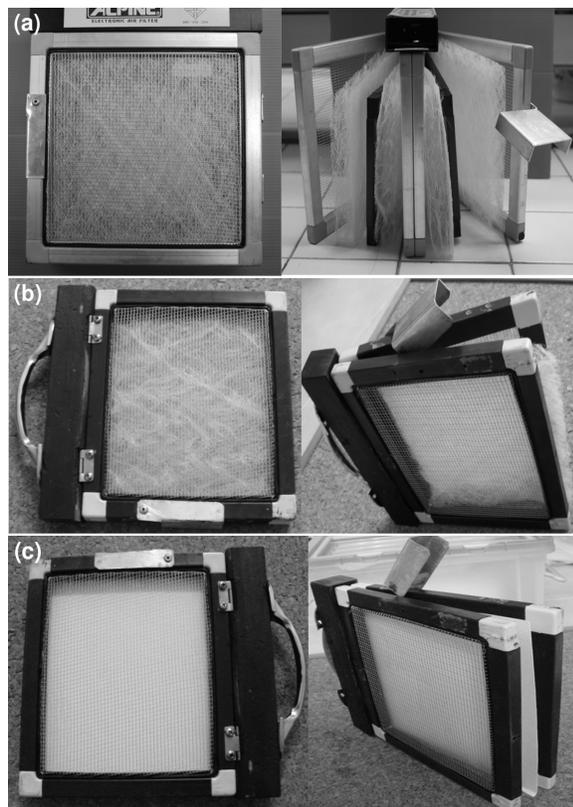
## MATERIALS AND METHODS

### Preparation of the photocatalytic air filters

Before dipping filters into 2% or 5% of TiO<sub>2</sub> photocatalyst suspensions, the fibre glass filter (supplied by Alpine Co., Ltd., Thailand) and the 99.99% efficiency HEPA filter (Bernard Dumas S.A.S., France) were pretreated in two steps. The first step was to eliminate the water resistance by washing with 0.08 M SDS for 10 min and then drying at room temperature for 24 h<sup>16</sup>. The optimum surface pretreatment method for removing water resistance and impurities from the filters during the production process was selected, using the maximum loading with the least TiO<sub>2</sub> fragility. For the second step, to improve TiO<sub>2</sub> coating adherence, filters were further pretreated with varying concentrations of NaF (0.01 M, 0.05 M, or 1 M) for 5 min, dried at room temperature for 24 h, and then desiccated for at least 2 h. Filters were weighed to determine their initial weight, and then coated by dipping into 2% or 5% of Degussa P25 TiO<sub>2</sub> suspension (50 m<sup>2</sup>/g, containing 75% anatase and 25% rutile) (Table 1) for 10 min, followed by drying at room temperature for 24 h. After coating with TiO<sub>2</sub>, the filters were baked at either 50 °C or 100 °C for 30 min, desiccated for at least 2 h, and then weighed to determine their final weight. After that, the optimum surface pretreatment method for increasing the bond strength – such as raising the surface roughness or changing the surface chemistry to produce a macro/microscopically rough surface<sup>18</sup> was selected, using the maximum loading and the least TiO<sub>2</sub> fragility, for improved mechanical stability (less cracking of TiO<sub>2</sub>) which governs the quality of an adhesively bonded joint. The coated filters were

**Table 1** The different TiO<sub>2</sub> suspensions.

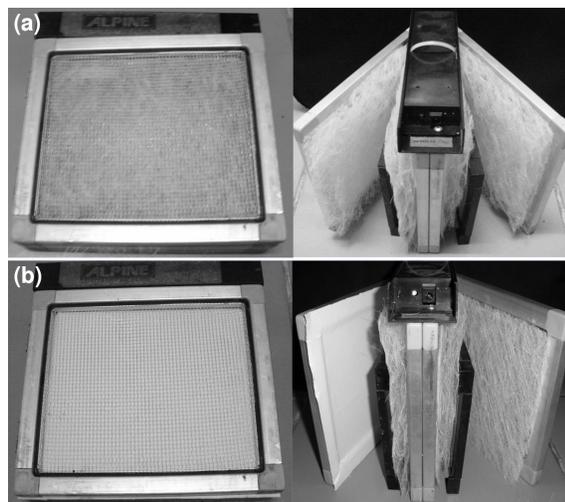
Type of TiO <sub>2</sub> suspension	TiO <sub>2</sub> (wt%)	Suspension medium	PEG 4000 (wt%)	Dispersant (Duramax D-3005) (wt%)	Baking temperature (°C)	Time of ultrasonic treatment (min)
Suspension 1	2	deionized water	–	1	100	2
Suspension 2	5	methanol	20	1	50	20

**Fig. 1** The filters in the frame: (a) ESF pack (four layers), (b) coated fibre glass filter (one layer), (c) coated HEPA filter (one layer).

preserved in plastic bags, desiccated before use, and framed in filter holders for the test. Characteristics of the combinations between the ESF packs and the photocatalytic air filters are shown in Fig. 1 and Fig. 2.

#### Testing physical properties of the photocatalytic air filters

Before the experiment of xylene removal was conducted, the filters were tested for loading by weighing, and for loading loss by blowing air through the filter in a testing chamber for at least 10 h. Loading was determined by the difference in the weight of the filter before and after coating. Loading loss was measured from the difference in weight before and after blowing

**Fig. 2** The filters in the frame: (a) ESF pack (three layers) and the coated fibre glass filter (one layer), (b) ESF pack (three layers) and the coated HEPA filter (one layer).

and was calculated as percentage of loss from the original loading. The pressure drop across these filters was examined for different airflow rates using a water U-tube manometer to observe the pressure before and after filtration. The thickness of the coating was controlled by selecting a similar loading of the filters with 5% TiO<sub>2</sub> coating by weighing the coated filters, which should contain 200–280 g/m<sup>2</sup> of loading for a fibre glass filter and 170–180 g/m<sup>2</sup> of loading for an HEPA filter. Fibre morphology and TiO<sub>2</sub> distribution were examined using a scanning electron microscope (SEM) (model 1450VP; Leo, UK).

#### Testing the efficacy of a combination of a photocatalytic air filter and an ESF pack

All experiments were conducted in a chamber which consisted of 10 units, as illustrated in Fig. 3. The blower unit forced air through the testing chamber in a single-pass mode. When the chamber was ready and conditioned to 25 °C and 45 ± 5% RH, the ESF packs and the coated filter packs were simultaneously inserted into section 1 of the chamber (irradiation) and section 2 of the chamber (no irradiation). Before

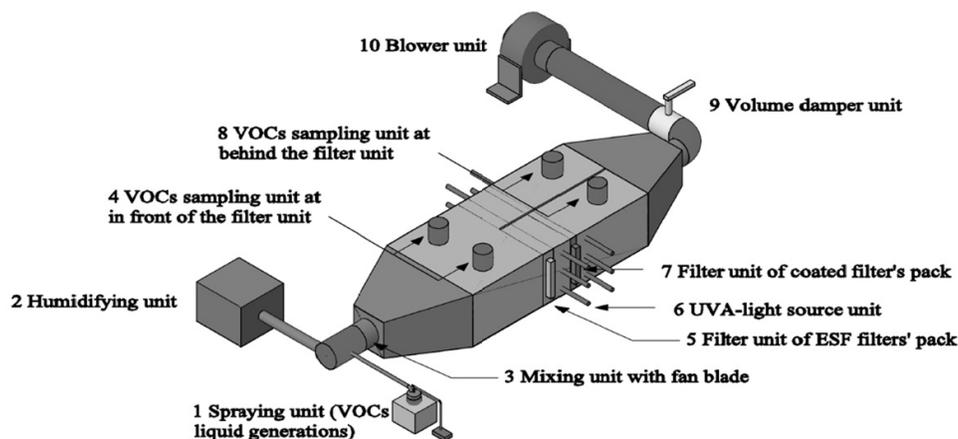


Fig. 3 Model of an experimental chamber.

the evaluation of xylene removal at a flow rate of 10 m<sup>3</sup>/min, steady-state gas concentration within the chamber was investigated under conditions in which xylene was constantly generated around 400–800 ppb using a one-jet collision nebulizer (model MRE-CN 241; BGI, USA) for 60 min while the blower was turned on (the lamps were turned off). In the mixing unit, the stream passed a fan (vane-axial) section to dry out the droplets before being filtered through the filter packs. The air and xylene in the chamber were mixed with the use of a mixer (three axial fans). The coated filter packs were separated into two testing groups: group 1 (irradiated) was for testing the efficiency of the coated filter with black light irradiation, while group 2 was a control that was not irradiated. When the concentration of xylene was stable, the light source in group 1 using six 36-W UVA lamps (Sylvania) at a light intensity of 2.73 mW/cm<sup>2</sup> on each side of the filter pack, as measured by a radiometer (model EW-09811-50; Cole-Parmer, USA) was turned on for irradiation times of 180 min. Group 2 was studied under the same conditions as group 1, but without a light source for the purpose of a control. Either a coated fibre glass filter or a coated HEPA filter was used in the filter packs. For each testing group, the filter with a frame and holder was inserted from the left side and right side of the chamber so that it could be pulled out for filter replacement. Rubber seals around the filter frames ensured a tight fit to prevent xylene leakage.

Both types of coated filters (fibre glass and HEPA filters) were compared for xylene degradation. The quantitative data of the concentration was analysed using a ppbRAE 3000 photoionization detector (PID) (model ppbRAE 3000; RAE systems, Inc., USA) with a photoionization sensor using a standard 10.6 eV lamp with an extended range of 1 ppb to 10 000 ppm.

Degradation of xylene was determined by comparing the concentration on the front of the ESF pack with that on the back of the coated filter pack. To simulate real conditions of filtration, the flow circulated constantly during nebulization and irradiation. Xylene concentration was recorded every 20 min throughout the 180 min test. This was based on the previous study<sup>19</sup> which found that after 4 h irradiation, steady-state concentration was achieved at 120 min, while a rapid drop of benzene, toluene, and xylene (BTX) concentrations occurred within the first 30 min. Xylene samples were obtained from both upstream and downstream of the filter pack and the PCO unit, and were analysed using PID technique to calculate the degradation efficiency.

After finishing each run of experiments, the chamber was blown thoroughly for at least 2 h to eliminate any remaining xylene, and the inside surface of the chamber was cleaned. Then, the remaining xylene was measured again using the ppbRAE PID to ensure that the chamber was clean before starting a new experiment.

All statistical analyses (ANOVA and *t*-test) were performed using SPSS version 12.

## RESULTS

### Loading

Table 2 shows that surface pretreatment methods for the substrates by washing with 0.08 M SDS and 0.01 M NaF before coating with 5% TiO<sub>2</sub> suspension and then baking at 50 °C for 30 min after coating yielded the highest loadings of 285 g/m<sup>2</sup> for the fibre glass filters and 182 g/m<sup>2</sup> for the HEPA filters. After chemical pretreatment and coating the substrates with 5% TiO<sub>2</sub>, the heat treatment process by baking at 100 °C was less productive because the PEG

**Table 2** Loading for fibre glass (FG) and HEPA filters pretreated by various methods ( $N = 10$ )<sup>a</sup>

Surface pretreatment methods	Loading for various air filters (g/m <sup>2</sup> ) <sup>b</sup>			
	FG filter coated with 2% TiO <sub>2</sub>	FG filter coated with 5% TiO <sub>2</sub>	HEPA filter coated with 2% TiO <sub>2</sub>	HEPA filter coated with 5% TiO <sub>2</sub>
Method 1 <sup>c</sup>				
NaF 0.01 M	190 ± 5	285 ± 87	61 ± 7	182 ± 7
NaF 0.05 M	162 ± 11	200 ± 7	49 ± 7	64 ± 12
NaF 1 M	162 ± 7	189 ± 10	47 ± 5	62 ± 12
Method 2 <sup>c</sup>				
NaF 0.01 M	249 ± 46	198 ± 7	147 ± 39	120 ± 12
NaF 0.05 M	153 ± 7	174 ± 15	130 ± 19	113 ± 11
NaF 1 M	150 ± 1	169 ± 15	88 ± 5	105 ± 11

<sup>a</sup>  $N$  denotes the number of tests performed on air filters (5 cm × 5 cm sections) for each surface pretreatment method.

<sup>b</sup> Values are mean (average TiO<sub>2</sub> loading from 10 tests) ± SD.

<sup>c</sup> Method 1 denotes washing filter with 0.08 M SDS once for 10 min, washing with NaF for 5 min, and baking at 50 °C for 30 min after coating. Method 2 denotes washing filter with 0.08 M SDS once for 10 min, washing with NaF for 5 min, and baking at 100 °C for 30 min after coating.

melted and made the filter surface sticky, changing it from a mechanically strong filter to a brittle filter. Hence the most suitable temperature for baking filters coated with 5% TiO<sub>2</sub> was 50 °C for 30 min. In contrast, baking at 100 °C for 30 min was the best heat treatment process for filters coated with 2% TiO<sub>2</sub> suspension. Hence washing with 0.08 M SDS and 0.01 M NaF before coating with 5% TiO<sub>2</sub>, followed by baking at 50 °C for 30 min was chosen as the surface pretreatment for this trial. This procedure ensured a higher loading, resulted in less damage of the fibre construction, less TiO<sub>2</sub> fragility or better governing of the quality of an adhesively bonded joint, and greater energy savings.

### Morphology and physical properties of the photocatalytic air filters

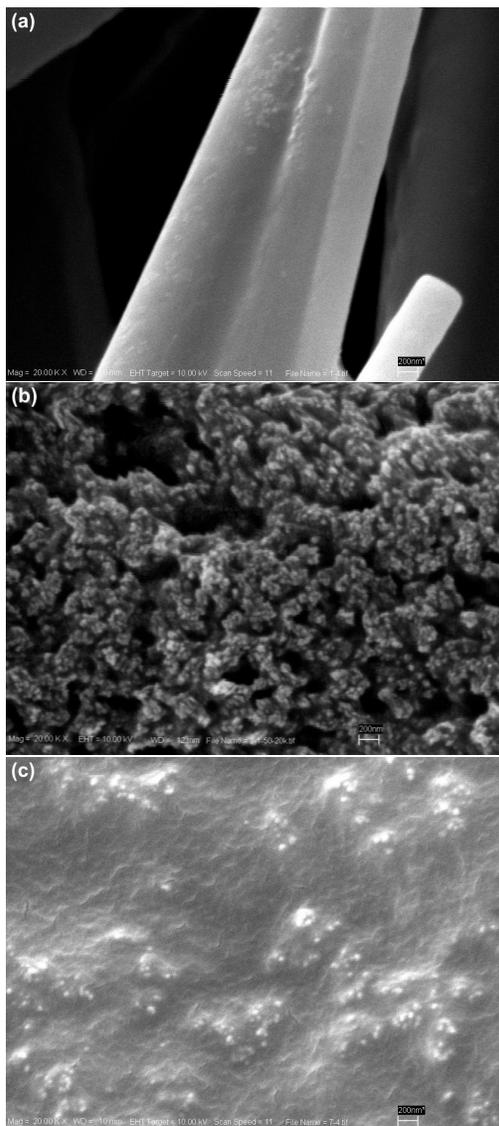
Fig. 4a (pretreated fibre glass filter) and Fig. 5a (pretreated HEPA filter) indicate that washing these filters with SDS and NaF as a surface treatment could lightly clean the fibre surfaces, and the macro/microscopically rough surfaces of the fibres could eliminate the water resistance.

Using a baking temperature of 50 °C for 30 min for the coated fibre glass filter (Fig. 4b) and the coated HEPA filter (Fig. 5b) produced more active TiO<sub>2</sub> particles in contact with the fibres, rather than using a baking temperature of 100 °C for 30 min (as shown in Fig. 4c for the coated fibre glass filter and Fig. 5c for the coated HEPA filter). After baking at 50 °C for 30 min in the present study, the TiO<sub>2</sub> particles were agglomerated and stuck to the fibres, and were well-distributed on the fibres as well.

### Pressure drop across filter pack

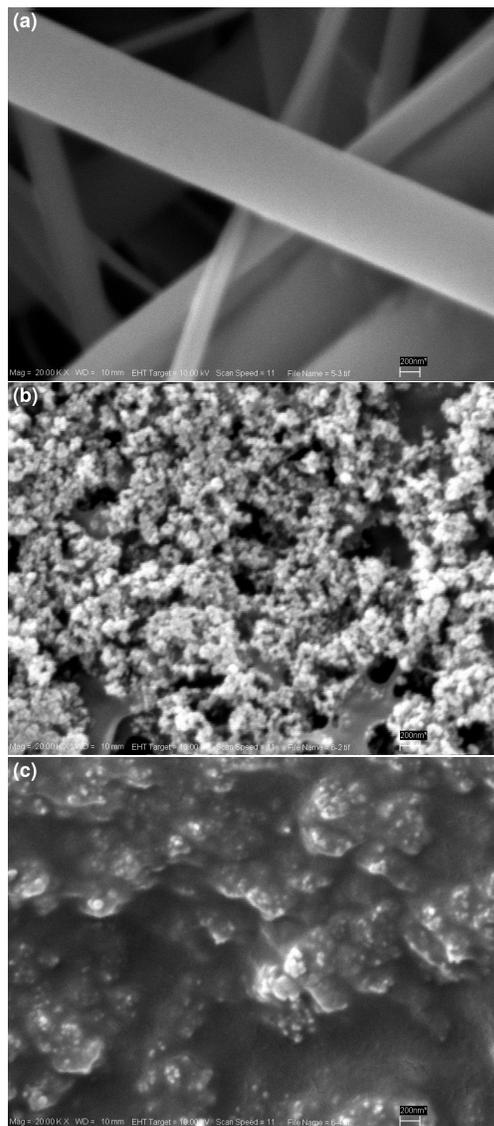
Compared to filter packs with untreated and/or uncoated fibre glass or HEPA filters, the pressure drop increased around 0.2–0.3 inch H<sub>2</sub>O when using a filter pack consisting of one fibre glass filter coated with 2% TiO<sub>2</sub> suspension and three layers of ESF filters, or using one fibre glass filter coated with 2% TiO<sub>2</sub> suspension and four layers of ESF filters, at 45 ± 5% RH and at 75 ± 5% RH. Additionally, the pressure drops increased around 0.3–0.5 inch H<sub>2</sub>O compared to those with no coating and no treating when using one fibre glass filter coated with 5% TiO<sub>2</sub> suspension and three layers of ESF filters, or using one fibre glass filter coated with 5% TiO<sub>2</sub> suspension and four layers of ESF filters, at 45 ± 5% RH and at 75 ± 5% RH. Hence the TiO<sub>2</sub> loading directly affected the pressure drop. When the pressure drops across the coated fibre glass filter packs were compared between 45 ± 5% RH and 75 ± 5% RH, it was revealed that the increase in relative humidity did not affect the pressure drop for filter packs with either the uncoated or coated fibre glass filters. The mean difference in pressure drop across the ESF pack and the coated fibre glass filter pack at 45 ± 5% RH and 75 ± 5% RH was in the range of 0.01–0.02 inch H<sub>2</sub>O.

Moreover, the pressure drops were increased around 0.2–0.4 inch H<sub>2</sub>O compared to those with no coating and no treating when using one HEPA filter coated with 2% TiO<sub>2</sub> suspension and three layers of ESF filters, or one HEPA filter coated with 2% TiO<sub>2</sub> suspension and four layers of ESF filters, at 45 ± 5% RH and at 75 ± 5% RH. Also, the pressure drop was enhanced around 0.3–0.7 inch H<sub>2</sub>O when using



**Fig. 4** Morphology of the fibre glass filter under different baking temperatures after washing the substrate and coating with 5% TiO<sub>2</sub> suspension: (a) washing with SDS and NaF (20k×), (b) coating with 5% TiO<sub>2</sub> and baking at 50 °C for 30 min (20k×), (c) coating with 5% TiO<sub>2</sub> and baking at 100 °C for 30 min (20k×).

one HEPA filter coated with 5% TiO<sub>2</sub> suspension and three layers of ESF filters, or using one HEPA filter coated with 5% TiO<sub>2</sub> suspension and four layers of ESF filters, at 45 ± 5% RH and at 75 ± 5% RH. Hence TiO<sub>2</sub> loading straightforwardly affected the pressure drop. When the pressure drops across the coated HEPA filter packs were compared between 45 ± 5% RH and 75 ± 5% RH, an increase in relative humidity did not affect the pressure drop for filter packs with

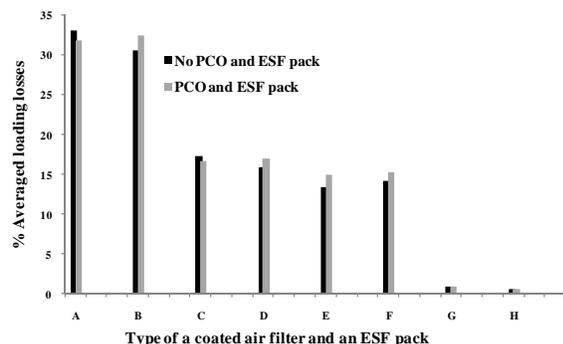


**Fig. 5** Morphology of the HEPA filter under different baking temperatures after washing the substrate and coating with 5% TiO<sub>2</sub> suspension: (a) washing with SDS and NaF (20k×), (b) coating with 5% TiO<sub>2</sub> and baking at 50 °C for 30 min (20k×), (c) coating with 5% TiO<sub>2</sub> and baking at 100 °C for 30 min (20k×).

either the uncoated or coated HEPA filters. The mean difference in pressure drop across the ESF pack and the coated HEPA filter packs at 45 ± 5% RH and 75 ± 5% RH was in the range of 0.1–0.2 inch H<sub>2</sub>O.

**Loading loss**

After coating with 5% TiO<sub>2</sub> suspension to the required loading of 200–280 g/m<sup>2</sup> for the coated fibre glass filter and 170–180 g/m<sup>2</sup> of loading for the coated



**Fig. 6** Average  $\text{TiO}_2$  loading loss for different types of air filters (size  $20\text{ cm} \times 20\text{ cm}$ ) at  $18\text{ m}^3/\text{min}$ , calculated from 3 tests. A: Fibre glass filter coated with 2%  $\text{TiO}_2$  suspension (baked at  $100^\circ\text{C}$  for 30 min) and three layers of ESF filters. B: Fibre glass filter coated with 2%  $\text{TiO}_2$  suspension (baked at  $100^\circ\text{C}$  for 30 min) and four layers of ESF filters. C: HEPA filter coated with 2%  $\text{TiO}_2$  suspension (baked at  $100^\circ\text{C}$  for 30 min) and three layers of ESF filters. D: HEPA filter coated with 2%  $\text{TiO}_2$  suspension (baked at  $100^\circ\text{C}$  for 30 min) and four layers of ESF filters. E: Fibre glass filter coated with 5%  $\text{TiO}_2$  suspension (baked at  $50^\circ\text{C}$  for 30 min) and three layers of ESF filters. F: Fibre glass filter coated with 5%  $\text{TiO}_2$  suspension (baked at  $50^\circ\text{C}$  for 30 min) and four layers of ESF filters. G: HEPA filter coated with 5%  $\text{TiO}_2$  suspension (baked at  $50^\circ\text{C}$  for 30 min) and three layers of ESF filters. H: HEPA filter coated with 5%  $\text{TiO}_2$  suspension (baked at  $50^\circ\text{C}$  for 30 min) and four layers of ESF filters.

HEPA filter, the coated filter packs were set in the reactor. Air at a high air flow rate ( $18\text{ m}^3/\text{min}$ ) was allowed to pass through the filters for 10 h to test for loading loss. The results of the percentage averaged loading losses in Fig. 6 verified 13% for the non-irradiated group and 15% for the irradiated group of using one fibre glass filter coated with 5%  $\text{TiO}_2$  suspension and three layers of ESF filters (Fig. 6 bar graph E). This was lower than the percentage averaged loading losses of using one fibre glass filter coated with 5%  $\text{TiO}_2$  suspension and four layers of ESF filters for both of the non-irradiated group and the irradiated group (Fig. 6 bar graph F). However, the loading loss was not significantly different ( $p = 0.444$  for the non-irradiated group and  $p = 0.434$  for the irradiated group).

The percentage averaged loading losses for the irradiated filter packs were 0.9% when using one HEPA filter coated with 5%  $\text{TiO}_2$  suspension and three layers of ESF filters, and 0.7% when using one HEPA filter coated with 5%  $\text{TiO}_2$  suspension and four layers

of ESF filters. On the other hand, the comparison of loading losses between using one HEPA filter coated with 5%  $\text{TiO}_2$  suspension and three layers of ESF filters, and using one fibre glass filter coated with 5%  $\text{TiO}_2$  suspension and four layers of ESF filters in the irradiated group were obviously seen to have insignificant losses of loading ( $p = 0.165$ ).

### Degradation of xylene

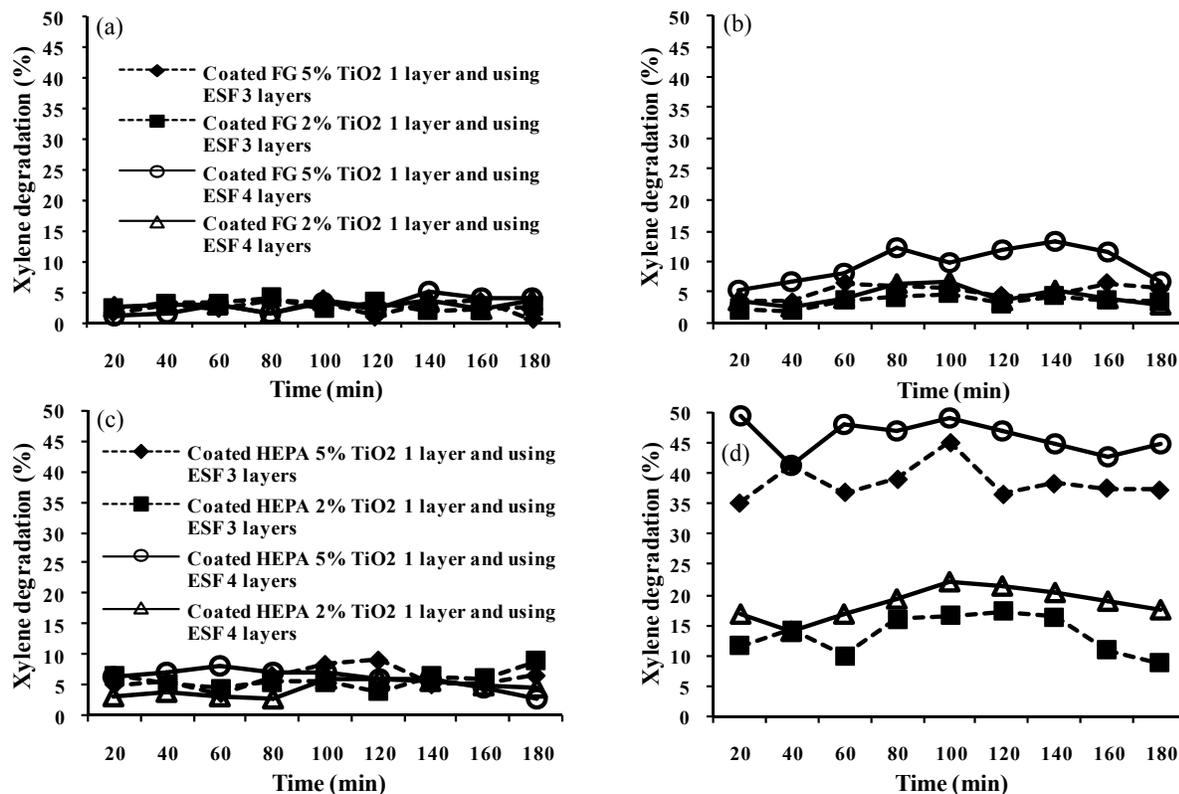
Comparison of xylene degradation of two modified types of ESF packs for use with a photocatalytic air filter was investigated by using one coated filter (coated fibre glass filter or coated HEPA filter) with three layers of ESF filters, where all filters were placed in an ESF pack, compared with using one coated filter (coated fibre glass filter or coated HEPA filter) with four layers of ESF filters, where the coated air filter was placed in a coated air filter pack and all ESF filters were placed in an ESF pack. The fibre glass and HEPA filters were coated with two different  $\text{TiO}_2$  suspensions, 2% or 5%  $\text{TiO}_2$ .

In the case of using a coated fibre glass filter, it was found that using one fibre glass filter coated with 5%  $\text{TiO}_2$  suspension and four layers of ESF filters resulted in the highest degradation efficiency of xylene, which ranged from 5–13% in the irradiated group (Fig. 7b). This was a significant difference when compared with the other filter pack configurations ( $p < 0.0001$ ). The results from the non-irradiated group are shown in Fig. 7a. The degradation efficiency of xylene was not significantly different for all types of filter packs with coated fibre glass filter.

In the case of using a coated HEPA filter, it was found that using one HEPA filter coated with 5%  $\text{TiO}_2$  suspension and four layers of ESF filters resulted in the highest degradation efficiency of xylene, which ranged from 41–49% in the irradiated group (Fig. 7d). This was a statistically significant difference when compared with the other filter pack configurations ( $p < 0.0001$ ). The results of the non-irradiated group are shown in Fig. 7c. The degradation efficiency of xylene was not significantly different for all types of filter packs with coated HEPA filter.

### DISCUSSION

Although investigation of the roughness of the fibres of filters pretreated with SDS and NaF was difficult based on SEM results, the loading of these filters was higher than that of filters treated with SDS alone. This high loading might depend on various factors such as decreased water resistance, increased roughness, or higher adhesion of  $\text{TiO}_2$  with negative charge on the surface of the filter. This result was consistent



**Fig. 7** Degradation of xylene (in 400–800 ppb) from various air filters at 10 m<sup>3</sup>/min: (a) coated fibre glass (FG) filter, not irradiated (no PCO with ESF), (b) coated fibre glass (FG) filter, irradiated (PCO with ESF), (c) coated HEPA filter, not irradiated (no PCO with ESF), (d) coated HEPA filter, irradiated (PCO with ESF).

with a previous study<sup>20</sup> which found that this chemical surface treatment provided not only increased roughness of the fibre surfaces but also increased the interaction between the TiO<sub>2</sub> and the substrate. According to the previous investigations<sup>16,17</sup>, using 0.08 M SDS could reduce the water resistance of the fibres of the pretreated HEPA filters, which were the same type of HEPA filter used in this study. In the present experiment, some fibres and other wastes were found in the SDS suspension, and these wastes also changed the colour of the suspension. This indicates that the first step of surface treatment could eliminate any initially weak outer-fibre layers as well as other surface impurities on the filters left from the manufacturing process. The experimental results show that washing with NaF suspension as a surface treatment of the air filters improves loading. This pretreatment method could cause a negative charge to be deposited on the surface of the air filters because of the fluoride-saturated TiO<sub>2</sub> surface, and might increase the roughness of the filter surface due to the HF in the NaF suspension, leading to the attraction of TiO<sub>2</sub> and its consequent adsorption onto the filters. This

interpretation is in agreement with the later study<sup>20</sup>, which found that surface pretreatment with NaF suspension created a fluoride-saturated TiO<sub>2</sub> surface on the filters, resulting in a negative charge and significantly increased TiO<sub>2</sub> interaction with the filters, while also enhancing the roughness of the substrate surface. A previous study<sup>21</sup> also confirmed that the photocatalytic degradation of organic pollutants was greatly enhanced by using surface fluorination of TiO<sub>2</sub> rather than bare TiO<sub>2</sub>. This enhancement is mainly due to the increased generation of hydroxyl radicals due to the presence of more available holes.

This trial found that loading of the coated air filters using PEG 4000 as additive substances in the TiO<sub>2</sub> suspension was better than those using Duramax D3005 as additive substances in the TiO<sub>2</sub> suspension due to increased adhesion between the photocatalyst and the substrates because of the presence of PEG 4000. Similarly, a previous investigations using PEG as an additive substance in a TiO<sub>2</sub> suspension also resulted in improved adhesion of the TiO<sub>2</sub> powder to the glass fibres<sup>22,23</sup>.

In the present study, we found that the optimal

temperature for heat treatment significantly increased the loading. This is supported by a study<sup>24</sup>, which examined the adhesion of TiO<sub>2</sub> films to glass by a scratch test. The films evaporated onto the heated substrates leading to excellent abrasion resistance, which depends more on adhesion than on differences in the hardness of the film for such materials. A later study<sup>25</sup> investigated the effects of different heating pretreatments on TiO<sub>2</sub> film structure, and found that the pretreatment process was important during preparation of the TiO<sub>2</sub> coating, and had an obvious effect on TiO<sub>2</sub> film structure due to the different heating mechanisms. Baking at 50 °C for 30 min after coating with 5% TiO<sub>2</sub> not only yielded higher loading than baking at 100 °C for 30 min, but these air filters also achieved greater strength, resulting in a more practical use for air treatment. Moreover, this baking method after coating produced better energy-saving air filters than the previous studies<sup>6,16,17</sup>. When comparing 2% and 5% TiO<sub>2</sub> loading after baking at 50 °C for 30 min, the highest loading was achieved by coating with 5% TiO<sub>2</sub>. In addition, a study<sup>11</sup> found no deactivation of benzene, toluene, ethylbenzene, or xylene (BTEX) when using 5% TiO<sub>2</sub> (Degussa P-25) coated on a glass fibre filter as a substrate. They also reported that these catalytic filters had a high degradation efficiency for BTEX (around 70–90%). A study<sup>26</sup> varied TiO<sub>2</sub> content in a range of 2–10% w/w, and found a similar degradation efficiency of benzamide with varying TiO<sub>2</sub> content. Thus using 5% TiO<sub>2</sub> (Degussa P-25) coated on the air filters in the present study was chosen due to achieve a high TiO<sub>2</sub> with minimal or no cracking of TiO<sub>2</sub> on the surface. Additionally, loading higher than 285 g/m<sup>2</sup> for the fibre glass filters and 182 g/m<sup>2</sup> for the HEPA filters resulted in TiO<sub>2</sub> cracking on the filters, and was hence deemed inappropriate for use.

According to the experimental results, enhancing efficiency of the ESF pack with the coated HEPA filter was better in enhancing xylene degradation than coated fibre glass filter. The explanation may follow from the results of SEM that found that the active sites of the coated HEPA filter were higher than those of the coated fibre glass filter. In addition, the loading losses of the coated fibre glass filter were higher than those of the coated HEPA filter. This caused less mechanical stability of coating of TiO<sub>2</sub> onto the coated fibre glass filter. This might result in lower xylene degradation when compared with enhancing efficiency of the ESF pack with the coated HEPA filter. This study shows that high binder dosage of PEG 4000 might result in enhancing the coating agglomerate size and decreasing the surface area limiting the contact

between TiO<sub>2</sub> and xylene molecules. This interpretation is in agreement with a later study<sup>27</sup>. However, the xylene degradation with PCO may be limited by other parameters such as adsorption, UV light intensity, relative humidity, mass transfer rate<sup>4,5,28</sup>.

## CONCLUSIONS

With regard to commercial-scale applications, the surface pretreatment methods from this study before coating with TiO<sub>2</sub> photocatalyst should apply to other similar substrates composed of SiO<sub>2</sub> for achievement of well absorption of the titania, fine adhesion of TiO<sub>2</sub> on the surface of the filter as well as high loading. PEG should apply to be the additive substance into the TiO<sub>2</sub> suspension for increasing the effective nano TiO<sub>2</sub>-material. However, development of the new photocatalytic air filter is still going on such as preparation of various TiO<sub>2</sub> suspensions with the different PEG molecular weight and with various suspension medium (i.e., ethanol, double distilled water) depending on characteristic of each substrate.

In further studies, the evaluation of the homogeneity and characteristics of the nano-TiO<sub>2</sub> photocatalyst onto the substrate should investigate with intensive measurements of X-ray diffraction and X-ray photoelectron spectroscopy. Also, lifetime investigation of using the coated HEPA filter with the ESF pack should be investigated. Using the combination between the coated HEPA filter and the ESF pack should test the effectiveness in large scale, such as application in an air handling unit for VOCs removals in a gross anatomy laboratory room, a process of car refinishing in some automobile repair shops, or some press publication shops to reduce chemical and biological hazards in the indoor air. The proper safety program for using the nano-material technology of the air filter should be established and enforced by the policy maker in Thailand such as Thai Industrial Standards Institute.

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