Facile and Novel Eco-Friendly Poly(Vinyl Alcohol) Nanofilters Using the Photocatalytic Property of Titanium Dioxide

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ABSTRACT: This study aimed to develop a highly efficient nanofilter for capturing fine particles using electrostatic forces. Poly(vinyl alcohol) (PVA), a water-soluble synthetic polymer, was selected as the main component of the filter because it can be easily fabricated by electrospinning. Titanium dioxide (TiO2) nanopowder with an anatase structure was applied to the nanofilters as it has the highest photocatalytic activity among the existing photocatalysts. PVA nanofilters fabricated by electrospinning could still be dissolved in water by hydrolysis. Therefore, heat treatment was performed to make the nanofilters stable, thereby forming C═O bonds by keto−enol tautomerization. Structural changes in the PVA nanofilter before and after heat treatment were investigated by X-ray diffraction (XRD) and Fourier transform infrared (FT-IR) analysis. As the TiO2 concentration increased, the fiber diameter of the PVA nanofilter decreased and a homogeneous fiber was obtained. The filtration efficiency and pressure drop also improved significantly, compared to those of the PVA-only nanofilter. Moreover, we observed eco-friendly decomposition of the PVA/TiO2 nanofilter into water and carbon dioxide by a photocatalytic reaction under UV irradiation.

INTRODUCTION

In recent years, fine dust has emerged as a major problem worldwide. Fine dust is classified into the categories PM2.5 (particle diameter below 2.5 μm) and PM10 (particle diameter below 10 μm) according to the particle diameter. Fine dust contains particles of various pollutants, such as automobile emissions, pollens, and bacteria.1 A major problem is that existing filters for fine dust are disposed of in landfills or incinerated, which may cause environmental pollution. To solve this problem, studies on reusable filters and the use of biopolymers as a filter media have been conducted. For example, various types of filters that can be reused have been researched, such as a filter capable of washing the collected pollutants with water, a filter for burning off the contaminants at a high temperature, and a filter for cleaning the fine dust using a solvent.2–5 Moreover, various biopolymers, such as silk, chitosan, and keratin, have been used to produce filters, either on their own or mixed with synthetic polymers.6–9 However, reusable filters also have a restriction on the number of reuse cycles, so they could potentially be disposed of in the same way as a conventional filter. In addition, they have disadvantages such as a higher unit cost in the case of biopolymer filters, and physical properties that are not comparable to those of synthetic polymers.

Nanofilters present an advantage, as the filtration efficiency can be further improved compared to conventional microlevel filters due to their high surface area to volume ratio. There are various methods for fabricating nanofilters, such as melt-blown, compound spinning, and electrospinning methods. Among them, electrospinning is a method for easily producing nanofilters and is thus adopted in this study. Various polymers have been fabricated and studied as nanofiber filters through electrospinning.10 Among them, poly(vinyl alcohol), PVA, is water-soluble and nontoxic and is commonly used for producing nanofibers by electrospinning.11 It has good elasticity, excellent miscibility with various polymers, excellent chemical and thermal stability, and has been used in various research fields.12,13 However, electrospun PVA fibers are water-soluble, which presents a massive disadvantage. Therefore, numerous studies have been conducted to reduce their reactivity with water through chemical crosslinking or heat treatment as a physical method. Studies on chemical crosslinking have mainly used aldehyde-based crosslinking agents, such as glutaraldehyde and formaldehyde.14,15 Since these chemical crosslinking agents may be toxic to humans, heat treatment was used to increase the crystallinity of PVA to increase water stability.16

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There are various types of photocatalysts, such as titanium dioxide, zinc oxide, cadmium sulfide, and tungsten oxide. However, zinc oxide and cadmium sulfide decompose into zinc and cadmium, which are harmful to human beings and the environment after a photocatalytic reaction, respectively. Thus, these photocatalysts cannot be used any longer. Tungsten oxide exhibits excellent photoactivity but has a disadvantage of being responsive to only specific materials. On the other hand, titanium dioxide (TiO₂) has been widely used in the photocatalytic decomposition of organic pollutants as it exhibits the highest photocatalytic activity, high durability, and low toxicity among the existing photocatalysts.17 Among them, the TiO₂ anatase type is the most widely studied because it has the advantages of excellent photocatalytic activity, chemical stability, environmental friendliness, and low cost.18

PVA and TiO₂ have been mixed and applied in various forms. Most studies have focused on the removal of organic compounds such as dye removal. For example, removal of methyl orange using a PVA/TiO₂ nanocomposite film and nanofibers, and photocatalytic decomposition of rhodamine B using nanofibers through electrospinning have been reported.12,19 In addition, there are several research studies on the decomposition of an aqueous solution of PVA using the photocatalytic activity of TiO₂.20,21 However, most photocatalytic decompositions of PVA were carried out in aqueous solution, and there have been few studies on decomposing solid PVA using a photocatalyst and there have been no reports of applying it to a filter.22,23 For these reasons, we tried to fabricate water-insoluble PVA/TiO₂ nanofilms by heat treatment, and the photocatalytic decomposition behavior of PVA/TiO₂ nanofilms was analyzed by UV irradiation.

### RESULTS AND DISCUSSION

The morphological changes of the electrospun nanofibers with changes in the PVA concentration were observed by field emission scanning electron microscopy (FE-SEM, Figure 1). It can be seen that the shape of the nanofiber changes depending on the concentration of the PVA solution. Low concentrations of PVA nanofibers contained beads, resulting in uneven fiber formation. When the concentration of PVA is more than a certain concentration, it can be seen that the fibers are not evenly formed. The optimal concentration of the PVA solution to produce nanofibers with no beads or even fibers was found to be 13%. Hence, all electrospinning solutions used a 13% PVA solution.

PVA nanofilms heat-treated at 155 °C for 5, 10, and 15 min were confirmed to be water-insoluble. When heat treatment was not performed, all of them dissolved in water by hydrolysis. However, when heat treatment was carried out for 5 min, a part of the PVA nanofilter tended to dissolve in water, and the PVA nanofilters heat-treated for 10 and 15 min did not dissolve in water at all. In addition, the PVA nanofilter discolored slightly after heat treatment, as shown in Figure 2A.

Thereafter, heat treatment of all samples was performed at 155 °C for 10 min. The weight losses of PVA nanofilms at different heat treatment times were also measured and are listed in Table 1.

### Table 1. Weight Loss of the PVA Nanofilts after Heat Treatment at Different Times

| heat treatment time (min) | 0 | 5 | 10 | 15 |
|--------------------------|---|---|----|----|
| weight loss (%)          | 100 | 6.72 | 0 | 0 |

Figure 1. FE-SEM images of PVA electrospun nanofilters with different PVA concentrations.

Figure 2. (A) Digital images of PVA nanofilters before and after heat treatment (photograph courtesy of Jihyun Yeo, Copyright 2020) and (B) X-ray diffraction (XRD) pattern of PVA nanofilters after heat treatment at different times.
The results of the wide-angle X-ray scattering (WAXS) analysis of PVA nanofilters before and after heat treatment at different times are shown in Figure 2B. The WAXS pattern interestingly changed at 2θ = 19.5°, which indicates that heat treatment causes a change in the crystallinity of PVA nanofilters. The crystallite size was calculated using the Scherrer equation (eq 1) and is summarized in Table 2.24,25

$$\tau (2\theta) = \frac{K\lambda}{\beta \cos \theta}$$  

(1)

$\tau$ is the average crystallite size to the peak width in radians, $K$ is the shape constant near unity, $\beta$ is the reflection width (full width at half-maximum, FWHM), $\theta$ is Bragg’s angle, and $\lambda$ is the wavelength of the applied X-ray source. As the heat treatment time was increased, it was confirmed that the crystallinity of PVA nanofilters increased, and as the FWHM also decreased, the crystallite size increased from 1.26 nm for 0 min to 2.38 nm for 15 min after heat treatment. This increase in the crystallite size was attributed to the removal of residual water in the PVA nanofilters after electrospinning by heat treatment. Moreover, the PVA polymer chain was regularly oriented in a certain direction, which contributed to the formation of additional crystal structures.

The Fourier-transform infrared (FT-IR) analysis results used to observe the change in the chemical structure of the PVA nano filters before and after heat treatment at 155 °C for 10 min is shown in Figure S1. A new peak around 1142 cm$^{-1}$ was observed. Crystallization has also been reported to be associated with the changes in the chemical structure.26-28 A peak was observed near 1430 cm$^{-1}$, which is attributed to CH$_2$ bonds, and it was confirmed that it remained virtually unchanged even after heat treatment. After heat treatment, a weak peak appeared at 1560 cm$^{-1}$, which is due to the formation of a new C=O group during the heat treatment process. The new C=O bond is formed by the keto–enol tautomerization reaction, as shown in the reaction in Scheme S2, and appears to be in the keto form because, according to Erlenmeyer’s law, the keto form is generally more stable than the enol tautomer form.29,30

The change in the filtration efficiency of the PVA nanofilter with changing electrospinning times is shown in Figure 3. The filtration efficiency tended to increase as the electrospinning time increased, and is given by eq 2

$$E_f = 1 - \frac{N_{\text{down}}}{N_{\text{up}}}$$  

(2)

where $N_{\text{up}}$ and $N_{\text{down}}$ are the number of particles upstream and downstream, respectively. For the PVA nanofilters with a short electrospinning time (less than 3 h), the NaCl test particles were not collected properly due to the pores in the PVA nanofilters. The reason for the increase in the filtration efficiency of PVA nanofilters with an electrospinning time over 3 h is that the PVA nanofilter layer thickens and the amount of PVA nanofibers capable of trapping NaCl particles increases. The pressure drop also tends to increase with the electrospinning time, which appears to increase with the increasing friction between the injected fluid and the PVA nanofibers. The pressure drop is the pressure difference between the inlet and outlet of the fluid. The pressure drop was also measured as shown in Figure 4 and calculated using the cake filter equation

$$\Delta P = \frac{\mu V}{A} \left( \frac{\alpha_i K_i}{A} + \beta \right)$$  

(3)

where $\Delta P$ is the pressure drop, $V$ is the flow rate, $\mu$ is the viscosity of the fluid, $\alpha_i$ is the average specific filter cake resistance value, $K_i$ is the influence of the concentration and cake porosity, $A$ is the averaged particle deposition area of the filtration, and $\beta$ is the resistance of the filter medium. The PVA nanofilter with an electrospinning time of 7 h showed a higher filtration efficiency (88.61%) compared to the PVA nanofilter with an electrospinning time of 6 h. The performance of a filter is known to be higher when the pressure drop of the filter is lower and the filtration efficiency is higher.31 Therefore, PVA/TiO$_2$ nanofilters were fabricated using a PVA nanofilter that underwent 6 h of electrospinning. The thickness of the PVA nanofilter was measured and is listed in Table S2; the thickness is proportional to the electrospinning time of the PVA solution. Therefore, the filtration efficiency followed the same trend with the electrospinning time.
The morphological changes in PVA/TiO$_2$ nanofilters due to changes in the TiO$_2$ concentration were observed in FE-SEM images, as shown in Figure 4. As the TiO$_2$ concentration increased, the fiber diameter tended to decrease (Table S3). In addition, it was also observed that TiO$_2$ agglomerated on the fiber surface as the TiO$_2$ concentration increased. Generally, the viscosity of the electrospinning solution is one of the important factors affecting the shape and diameter of the nanofibers. It is known that a thinner fiber is formed as the viscosity of the solution decreases. For example, a fiber diameter of approximately 60 nm was obtained at a viscosity of $\sim$0.04 Pa·s, whereas that of approximately 1000 nm was obtained at a viscosity of $\sim$6–7 Pa·s. The viscosities of the electrospinning solutions of P6 and P6T10 are shown in Figure S2. The addition of TiO$_2$ particles reduced the viscosity of electrospinning solution and thus fibers with a thinner diameter than PVA-only fibers could be formed. As the fiber diameter decreased, the specific surface area relatively increased. Therefore, the PVA/TiO$_2$ nanofilter proved to be more advantageous as filters for capturing fine particles.

The results of the WAXS analysis for P6 and P6T10 are shown in Figure 5A. A new peak was investigated at 2\(\theta\) = 4.69° in the XRD pattern of P6T10. This peak is a crystal peak of TiO$_2$ anatase, and it can be seen that TiO$_2$ particles were well dispersed in the PVA nanofilters. In addition, as the FWHM decreased, the crystallite size of P6T10 slightly increased than that of P6 and the crystallization also increased (Table 3).

![Figure 5. (A) XRD pattern of PVA/TiO$_2$ nanofilters and (B) filtration efficiency and pressure drop of PVA/TiO$_2$ nanofilters with different TiO$_2$ concentrations.](image)

Table 3. XRD Characteristic Data of the PVA/TiO$_2$ Nanofilters

| sample | FWHM (2\(\theta\)) | crystallite size (nm) | crystallinity (%) |
|--------|----------------|---------------------|------------------|
| P6     | 5.18           | 1.63                | 49.40            |
| P6T10  | 4.69           | 1.79                | 53.04            |

The filtration efficiency of the PVA/TiO$_2$ nanofilter is summarized in Figure 5B. After the automated filter test, the NaCl particles collected on the P6 and PVA/TiO$_2$ nanofilters were observed by FE-SEM as shown in Figure S3. It can be confirmed that the nanofilters containing TiO$_2$ particles have a higher filtration efficiency and a lower pressure drop than PVA-only nanofilters. This is related to the aforementioned fiber diameters, and as the fiber diameters decrease, the ratio of the surface area to volume increases and the pore size of the fiber decreases. Hence, the NaCl particles were collected more effectively and the pressure drop was reduced. In addition, the dispersity of the fiber diameter became narrow as TiO$_2$ was added, as confirmed in Table S3. The homogeneous distribution of the fiber diameters influenced the improvement of the filtration efficiency and the decrease of the pressure drop. There was no major difference between the filtration efficiency and the pressure drop between PVA/TiO$_2$ nanofilters. We concluded that the fiber diameter no longer affects the filtration efficiency and the pressure drop when it is less than a certain value.

The weight losses of PVA/TiO$_2$ nanofilters due to UV irradiation are shown in Figure 6A and Table S4. As the UV irradiation time and the TiO$_2$ concentration increased, the weight loss of the PVA/TiO$_2$ nanofilter increased. The weight loss of the PVA/TiO$_2$ nanofilter due to the photocatalytic reaction between PVA and TiO$_2$ increased. The photocatalytic mechanism between PVA and TiO$_2$ is shown in Scheme S3 and eqs 4–9.

\[
\text{TiO}_2 + h\nu \rightarrow e_{\text{CB}}^- + h^+_{\text{VB}} \\
\text{O}_2 + 2\text{H}^+ + 4e_{\text{CB}}^- \rightarrow 2\text{OH}^- \\
\text{OH}^- + \text{H}^+ \rightarrow \text{H}_2\text{O} \\
(\text{CH}_2\text{CH(OH)CH}_2\text{CH(OH)CH}_2) + h^+_{\text{VB}} \rightarrow (\text{CH}_2\text{CH(OH)CH}_2\text{CH(OH)CH}_2) + \text{H}^+ \\
(\text{CH}_2\text{CH(OH)CH}_2\text{CH(OH)CH}_2) + h^+_{\text{VB}} \rightarrow (\text{CH}_2\text{COCH}_2\text{CH(OH)CH}_2) + \text{H}^+ \\
\rightarrow \rightarrow \rightarrow \text{successive cleavages} \rightarrow \text{CO}_2 + \text{H}_2\text{O}
\]
The digital images of PVA and PVA/TiO2 nanofil ters decomposed by the photocatalytic reaction under UV irradiation are shown in Figure 6B. P6 retained its original form even after 9 days of UV irradiation, whereas P6T10 showed that the nanofilter was decomposed enough for the decomposition to be visually confirmed after 9 days of UV irradiation.

The morphological changes of a series of PVA and PVA/TiO2 nanofil ters with the UV irradiation time are shown in Figure 6C. P6 seemed to exhibit no changes in the fiber shape and diameters. P6T2.5 and P6T5 showed pores under UV irradiation after 9 days due to photocatalytic decomposition. P6T7.5 and P6T10, which exhibited higher photocatalytic decomposition, showed that most of the fibers were broken and did not maintain the filter structures after 6 days. In addition, P6T7.5 and P6T10 also showed more agglomerates, owing to the photocatalytic decomposition of the PVA fiber, and PVA decomposition seemed to be accelerated.

FT-IR spectra of PVA/TiO2 nanofil ters with different TiO2 concentrations and UV irradiation times are shown in Figure 7. In the case of P6, the peak around 1560 cm\(^{-1}\) does not disappear but it disappeared after 9 days of UV irradiation for PVA/TiO2 nanofil ters, according to Figure S4. Thus, it was confirmed that the PVA chain decomposed into CO\(_2\) by photocatalytic decomposition with TiO2 following eqs 5–10. Interestingly, for P6T10, this peak quickly disappeared after 3 days of UV irradiation.

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**EXPERIMENTAL SECTION**

**Materials.** Poly(vinyl alcohol) (PVA) was purchased from Duksan Pure Chemistry (average \(M_w = 66000\)). TiO\(_2\) nanopowder with a particle size of 18 nm was purchased from US Research Nanomaterials.

**Preparation of PVA/TiO\(_2\) Nanofil ters.** The fabrication process of a series of PVA/TiO\(_2\) nanofil ters is shown in Scheme 1. PVA was dissolved in distilled water at 80 °C for 2 h to prepare PVA solutions having concentrations of 9, 10, 11, 12, 13, 14, and 15%. The prepared solution was placed in a 10 mL syringe, and four syringes were mounted on an electrospinning device to perform electrospinning. The voltage was maintained at 20 kV, the diameter of the syringe nozzle was 22 gauge, the tip-to-collector distance was 30 cm, the solution discharge rate was 0.3 mL/h, and the collector rotation speed was 100 rpm. The temperature and humidity were maintained at 25 °C and 43% with a thermo-hygrostat, and electrospinning was performed for an hour. An aluminum window screen serving as a support for the filter was fixed to the collector. After confirming the proper concentration of the PVA solution, the PVA solution was electrospun from 1 to 7 h under the above conditions to confirm the optimal electrospinning time to fabricate the PVA nanofil ters. In the case of PVA/TiO\(_2\) nanofil ters with different TiO\(_2\) concentrations, however, the electrospinning process of the PVA/TiO\(_2\) solution was performed for 6 h under the same spinning conditions. The fabricated PVA/TiO\(_2\) nanofil ters and their compositions are denoted as P\(_{x}T_{y}\) according to the electrospinning time and the TiO\(_2\) concentration and are listed in Table 4.
Heat Treatment of PVA/TiO₂ Nanofilters. To prevent the electrospun PVA and PVA/TiO₂ nanofilters from dissolving in water, additional heat treatment was performed in a vacuum dryer at 155 °C for 5, 10, and 15 min, respectively.

Characterization. The change in the morphological structure of PVA nanofilters due to the PVA concentration, the average diameter of the electrospun PVA/TiO₂ nanofiber and NaCl particles collected on the PVA and PVA/TiO₂ nanofilters, as well as the change in the morphological structure of PVA and PVA/TiO₂ nanofilters after photocatalytic decomposition were confirmed by field emission scanning electron microscopy (FE-SEM, SUPRA 55VP, Carl Zeiss, Germany). The changes in the crystal structure of PVA nanofilters that underwent heat treatment and PVA nanofilters with different TiO₂ concentrations were analyzed using a wide-angle X-ray scattering system (WAXS, D8 Discover, Bruker, Germany). The change in the chemical structure of PVA nanofilters after heat treatment, photocatalytic decomposition behavior, and decomposed products formed under UV irradiation were investigated using Fourier-transform infrared spectroscopy (FT-IR, Nicolet iSS, Thermo Fisher Scientific) in the range of 400–4000 cm⁻¹. The changes in the viscosity of the electrospinning PVA solutions with different TiO₂ concentrations were determined using a rheometer (MARS III, Thermo Fisher Scientific).

Filtration Test. The filtration efficiency and pressure drop of PVA and PVA/TiO₂ nanofilters were measured using an automated filter tester (8310A, TSI Inc.) at a concentration of 8 ± 4 mg/m³, a flow rate of 95 L/min, and humidity of 60%. NaCl particles with an average particle size of 0.6 µm were used as the test particles following Korea Filter 80 (KF80) standard.

Photocatalytic Decomposition Test. The sample was irradiated with ultraviolet light (UV) using an accelerated weathering tester (QUV, Q-Lab) to evaluate the photocatalytic decomposition behavior. The wavelength of the UV irradiation was 340 nm, and the intensity of UV irradiation was 0.68 W/m². The temperature of the chamber was 60 °C, and the humidity was 50%. To confirm the photocatalytic decomposition behavior at different UV irradiation times, the samples were exposed to UV irradiation for 3, 6, and 9 days. The weight losses of PVA nanofilters after heat treatment, and the

Figure 7. FT-IR spectra of PVA/TiO₂ nanofilters at different UV irradiation times: (A) P6, (B) P6T2.5, (C) P6T5, (D) P6T7.5, and (E) P6T10.
Scheme 1. Schematic Illustration of the Fabrication Process of the PVA/TiO\textsubscript{2} Nanofilter

Table 4. Composition and Fabrication Conditions of the PVA/TiO\textsubscript{2} Nanofilters

| sample | TiO\textsubscript{2} concentration (wt %) | electrospinning time (h) | heat treatment condition |
|--------|---------------------------------|-------------------------|-------------------------|
| P6     | 20                              | 6                       | 155 °C for 10 min       |
| P6T2.5 | 2.5                             | 6                       | 155 °C for 10 min       |
| P6T5   | 5                               | 6                       | 155 °C for 10 min       |
| P6T7.5 | 7.5                             | 6                       | 155 °C for 10 min       |
| P6T10  | 10                              | 6                       | 155 °C for 10 min       |

PVA/TiO\textsubscript{2} nanofilters after the photocatalytic decomposition were calculated using (eq 10).

\[
\text{weight loss (\%)} = \frac{W_0 - W_f}{W_0} \times 100
\]  

where \(W_0\) is the weight of the filter before heat treatment or photocatalytic decomposition, and \(W_f\) is the weight of the filter after heat treatment or photocatalytic decomposition.

**CONCLUSIONS**

In this study, photocatalytically degradable PVA/TiO\textsubscript{2} nanofilters were fabricated by electrospinning to overcome the limitations regarding the disposal of existing filters by incineration or in landfills, which might cause environmental pollution. The optimum concentration of the PVA solution was confirmed by FE-SEM images of the electrospun PVA nanofibers. Changes in the crystallinity, filtration efficiency, and the pressure drop of PVA nanofilters were observed before and after heat treatment. In addition, when TiO\textsubscript{2} particles were added, the fiber diameter decreased by lowering the viscosity of the PVA solution and the filtration efficiency was effectively improved compared to that of the PVA-only nanofilter. Moreover, we also confirmed the photocatalytic decomposition of PVA/TiO\textsubscript{2} nanofilters by investigating the disappearance of the C==O peaks under UV irradiation. We finally concluded that PVA/TiO\textsubscript{2} nanofilters fabricated in this study can be used as eco-friendly filters.

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.9b03944.

FT-IR spectra of the PVA nanofilter before and after heat treatment; keto-enol tautomerization of the PVA nanofilter during heat treatment; average filter thickness of PVA nanofilters; shear viscosity of electrospinning solutions of P6 and P6T10; average fiber diameter of PVA/TiO\textsubscript{2} nanofilters; FE-SEM image of P6 and P6T10 nanofilters after the filtration test; photocatalytic decomposition of the PVA/TiO\textsubscript{2} nanofilter; weight loss of PVA/TiO\textsubscript{2} nanofilters at different UV irradiation times; FT-IR spectra in the range of 1200–1800 cm\textsuperscript{-1} for P6 and P6T10 (Figures S1–S4, Schemes S1 and S2, Tables S1–S3) (PDF)

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**Notes**

The authors declare no competing financial interest.

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