Photocatalytic Rejuvenation Enabled Self-Sanitizing, Reusable, and Biodegradable Masks against COVID-19

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ABSTRACT: Personal protective equipment (PPE) has been highly recommended by the U.S. Centers for Disease Control and Prevention for self-protection during the disastrous SARS-CoV-2 (COVID-19) pandemic. Nevertheless, massive utilization of PPE encounters significant challenges in recycling and sterilizing the used masks. To tackle the associated plastic pollution of used masks, in this work, we designed a reusable, biodegradable, and antibacterial mask. The mask was fabricated by the electrospinning of polyvinyl alcohol (PVA), poly(ethylene oxide) (PEO), and cellulose nanofiber (CNF), followed by esterification and the deposition of a nitrogen-doped TiO2 (N-TiO2) and TiO2 mixture. The fabricated mask containing photocatalytic N-TiO2/TiO2 reached 100% bacteria disinfection under either 0.1 sun simulation (200−2500 nm, 106 W m−2) or natural sunlight for only 10 min. Thus, the used mask can be rejuvenated through light irradiation and reused, which represents one of the handiest technologies for handling used masks. Furthermore, intermolecular interactions between PVA, PEO, and CNF enhanced the electrospinnability and mechanical performance of the resultant mask, which possesses a 10-fold elastic modulus and 2-fold tensile strength higher than a commercial single-use mask. The porous structures of electrospun nanofibers along with strong electrostatic attraction enabled breathability (83.4 L min−1 of air flow rate) and superior particle filterability (98.7%). The prepared mask also had excellent cycling performance, wearability, and stable filtration efficiency even after 120 min wearing. Therefore, this mask could be a great alternative to current masks to address the urgent need for a sustainable, reusable, environmentally friendly, and efficient PPE under the ongoing COVID-19 contagion.

KEYWORDS: COVID-19, mask rejuvenation, mask reuse, N-TiO2, photocatalysis, environmental friendliness

The continuing COVID-19 is highly infectious; the coronavirus could transmit via short-range aerosols and respiratory droplets generated from sneezing, coughing, and even speaking and breathing.1−3 As of today, 17 months after the outbreak of this severe pneumonic disease, 222 countries and regions worldwide have reported the COVID-19 cases, with a total number of 172 million infections, 3.58 million deaths, and over 428 thousand new cases daily.4 These numbers are still increasing exponentially. The infected people mainly experience fever, fatigue, dry cough, and such,5 however, the transmission and pathogenesis of COVID-19 are still uncertain yet.6 Therefore, keeping personal protection from the infection remains an urgent and significant challenging issue worldwide.

Before any effective way can be implemented to prevent the spread of coronavirus, the use of PPE such as surgical or medical masks and keeping social distance are still the most efficient self-protection from COVID-19, as recommended by the U.S. Centers for Disease Control and Prevention (CDC) as well as many other governments. Currently, the dominant and mostly used masks are the N95 respirator and surgical masks, which contain a thin layer of polypropylene (PP) melt blown fabrics to filter out the particles and droplets. Generally, PP fabrics are charged to mount the negative charges on surface, which increases the filtration efficiency of masks. However, the N95 respirators are recommended to be limited to five reuses, and the surgical masks are suggested to be changed every 2 to 4 h,

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More importantly, the N-TiO₂ can efficiently rejuvenate the mask by face-light irradiation to kill the infected bacteria, which makes the mask reusable.

RESULTS AND DISCUSSION

Circular Designing of Photon-Rejuvenated, Reusable, and Biodegradable Mask. The massive consumption of the masks as a result of the COVID-19 breakout has rendered the shortage of PP polymers and the disruption of PP supply chains. The global PP market has been anticipated to grow as much as 7.09% over last year. On the other hand, most masks in current market such as N95 respirators, surgical masks, and single-use masks encounter significant decline in filtration efficiency after a short utilization, which rendered their poor reusability. These masks made of PP thus are generally discarded for post-handling, which has raised concerns of both a second infection and plastic pollution to environment. As shown in Figure 1, our design of the mask enables their antibacterial property and light rejuvenation (200−2500 nm, 106 W m⁻²) by photocatalysis so that the masks can be reused after facial light irradiation. TiO₂ has been known as one of the most efficient photocatalytic semiconductors, as long as its advantages of excellent stability and cost-effectiveness. Nevertheless, the high band gap of TiO₂ has limited its absorbance only in the UV range. Doping the nitrogen can reduce the band gap of TiO₂ and thus enhance its photocatalysis under visible light irradiation. As a result, N-TiO₂ can have an efficient photocatalysis under light irradiation. N-TiO₂ was thus employed to sterilize pathogens and rejuvenate the masks for reuse by simply irradiating the mask under the light for a certain time.

In the meantime, we fabricated the mask with a circular design by using biodegradable precursor polymers of PVA and PEO as plasticizer in the electrospinning dopes. Both effects largely improved the electrospinnability and mechanical performance of the prepared mask. All these designs can complement each other to fabricate reusable, biodegradable, high-quality, and environmentally friendly masks that are easy for handling after massive utilization as a result of the COVID-19 pandemic.
well as an environmentally friendly wood-derived nanocellulose instead of synthetic plastics. All these three components are biodegradable polymers that can be degraded by environmental microorganisms; for example, *Pseudomonas* strains are capable of degrading PVA and PEO, while many cellulolytic fungi and bacteria produce cellulase to biodegrade CNF. These polymer blends were designed for intimate intermolecular interactions that can enhance their processability and the mechanical performance of the resultant mask. We also selected the electrospinning to process polymers for mask manufacturing (Figure 1), which is to produce a nanomesh with fine nanofibrous and porous structures to promote both breathability and filterability of the mask. As a result of the selected biodegradable materials, high surface area from the electrospun nanofibers, photocatalysis, and circular designing, the produced mask is concurrently functionalized with superior filterability, breathability, mechanical strength, self-sterilization, and environmental friendliness. All these properties enabled an alternative way to fabricate and process next generation reusable and biodegradable masks sustainably.

**Fabrication of Masks.** To fabricate the mask with antibacterial and rejuvenation properties, commercial P25 TiO₂ was doped using urea as the nitrogen source and then deposited onto the masks to enable their photocatalytic capacity. After the doping, the color of the TiO₂ changed from white to yellow (Figure 2A). The elementary mapping under a scanning transmission electron microscope (S-TEM) confirmed the existence of the doped nitrogen in the N-TiO₂. As shown in Figure 2B, the element of nitrogen along with the elements of both titanium and oxygen have been determined. The atomic fraction of the nitrogen elements in the N-TiO₂ was semi-quantitatively analyzed by calculating the peak area of the energy dispersive X-ray spectroscopy (EDX). As shown in Figure S1, the ratio of titanium (26.6%) and oxygen (61.2%) was measured close to 1:2, which validated the EDX characterization. In the meanwhile, the atom fraction of the nitrogen was measured at 12.2%, demonstrating the successful doping of nitrogen in the N-TiO₂. The nitrogen doping has changed the band gap of the TiO₂. As shown in Figure 2C, TiO₂ without nitrogen doping displayed strong absorbance in the UV and near visible light region (320–405 nm). After nitrogen doping, the absorbance edge shifted to 448 nm, and consequently, the N-TiO₂ can significantly absorb visible lights. The band-gap energy was calculated using the Planck’s law

\[ E = h \times \nu = \frac{hc}{\lambda} = 1241/\lambda \]

where \( E \) is the band-gap energy (eV), \( h \) is the Planck constant, \( \nu \) is the frequency of the light, \( c \) is the speed of the light (m/s), and \( \lambda \) is the wavelength of the light (nm). The band-gap energy for TiO₂ was calculated at 3.06 eV, which was decreased to 2.77 eV for N-TiO₂. The mimicking light (200−2500 nm, 50 W) we used was with 5.2% UV light, 37.2% visible light, and 57.6% infrared light (Table S1); therefore, these results highlighted the photocatalytic activity of the N-TiO₂ under light irradiation by enhancing the visible light excitation.

The prepared N-TiO₂ was deposited onto the electrospun nanomesh to equip the mask with antibacterial and photocatalytic rejuvenation properties. We employed electrospinning technology to fabricate the biodegradable mask by using PVA polymer as the main component and CNF as the enforcement; however, PVA under our electrospinning conditions (at the concentration ∼8 wt %) had limited spinnability in that droplets were formed at the tip of the needle during the electrospinning (Figure 3A1) due to its high viscosity (Figures 3B and S2). Even worse, the spinnability has been deteriorated after adding CNF into PVA; the resultant CNF+PVA blend was not even spinnable (Figure 3A2). The reason could lie in the significantly increased viscosity by adding CNF into PVA (Figures 3B and S2). The spinnability has been much improved by using PEO as the plasticizer. As shown in Figure 3A3, a good shaped Taylor cone with a continuous nanofiber has been formed at the tip of the needle under the electrical field, resulting in excellent electrospinnability of the PVA+CNF+PEO blend. The largely improved spinnability by PEO was due to the reduced viscosity (Figures 3B and S2), which is rendered by the bad miscibility and compatibility between PEO polymer and PVA polymer. The poor compatibility between PEO and PVA can be further proved by the lowest viscosity of the PVA+PEO blend (Figures 3B and S2). Nevertheless, CNF in the PVA+CNF+PEO blend can serve as the compatibilizer between PVA and PEO polymers by forming abundant hydrogen bonding as deciphered in Figure 3C. As a result, the polymers in the PVA+CNF+PEO blend had intimate intermolecular interactions with decreased viscosity, excellent spinnability, and enhanced mechanical performance (to be discussed whereafter).

The resultant electrospun nanomesh made from the PVA+CNF+PEO blend was white, uniform, and had a size of 11 in. by 8.5 in. (Figure 3D). The morphology of the as-spun nanomesh was analyzed by using a scanning electron microscopy (SEM). As shown in Figures 3E and S3, the electrospun nanomesh was composed of uniform nanofibers with diameters of 790 nm that are much smaller than that of the traditional PP fibers (1.72 μm) made by melt blowing for surgical masks. The finer nanofibers can increase their contacting areas with guest particles and droplets, which can thus endow the mask with superior filterability of bacteria. Meanwhile, the nanomesh had poros at several micrometers formed in between (Figure S3) that could enable the excellent breathability. Subsequently, the as-spun mask was function-alized by hydrophobilization and then deposited with N-TiO₂.

First, hydrophobicity is an essential need for masks. pathogen is able to be transmitted through droplets from...
The hydrophobicity of the mask can repel the deposition of droplets on the mask and thus improve its filtration efficiency. The biodegradable precursor polymers for electrospinning, namely, CNF, PVA, and PEO, however, are hydrophilic. We thus employed a facile esterification strategy to convert surface hydroxyl groups of the as-electrospun masks into esterified hydrophobic moieties. As shown in Figure 3E1,E2,F1,F2, stearic acid has been grafted onto the surface of the nanofibers where its carboxyl acid ends react with the surface hydroxyl groups to form ester linkages, and its long hydrophobic moieties (C17) endow the hydrophobicity of the nanomesh. The contact angle analysis has demonstrated this hydrophilicity-hydrophobicity conversion. As shown in Figure 3G1,G2, the as-spun nanomesh had a contact angle of 39.6°, whereas the contact angle of the esterified nanomesh has increased to 91.9°. All these results highlighted that esterification has efficiently converted the highly hydrophilic as-spun nanomesh into hydrophobic.

Afterward, both TiO2 and N-TiO2 and the N-TiO2/TiO2 mixture have been deposited onto the esterified masks. As shown in panels (3), (4), and (5) in Figure 3E,F, TiO2, N-TiO2, and the N-TiO2/TiO2 mixture have been evenly distributed on the nanofibers of the mask. The hydrophobicity of the TiO2-deposited mask has been further increased, as indicated by the increased contact angle (98.9°, Figure 3G3). However, this increment in the hydrophobicity for N-TiO2-deposited mask has decreased (contact angle was 68.3°, Figure 3G4) as compared with that of the TiO2-deposited mask, attributed to the slight hydrophilicity induced by nitrogen doping. This decrement in hydrophobicity remained (contact angle 68.0°, Figure S4A) even when N-TiO2 was deposited first and then the mask was esterified by stearic acid. To prepare the mask concurrently to have superior hydrophobicity and photocatalytic property,
developed a blending strategy in which the TiO₂ and N-TiO₂ were mixed at a ratio and then deposited on the esterified masks. TiO₂ could endow the hydrophobicity and photocatalytic activity and N-TiO₂ can further enhance the photocatalytic activity. TiO₂ and N-TiO₂ were mixed at the weight ratios of 7:3 and 1:1. As shown in Figure 3G5, the contact angle for the former one (TiO₂/N-TiO₂, 7:3) was at 98.0°, which is slightly higher than that of the latter mask deposited with 50% TiO₂ and 50% N-TiO₂ (90.4°, Figure S4B). This contact angle for the mask deposited with 30% N-TiO₂ and 70% TiO₂ (98.0°, Figure 3G5) was close to that of the mask deposited with only TiO₂ and was much higher than that of the mask deposited with only N-TiO₂. We thereof selected N-TiO₂/TiO₂ at a mixing ratio of 3:7 for making masks with photocatalytic activity. Overall, the hydrophobicity of the masks has been significantly enhanced by esterification with stearic acid, and the photocatalytic TiO₂ and N-TiO₂ have been deposited onto the masks to enable its antibacterial capacity and light rejuvenation for reusing.

Figure 4. Rejuvenation of our mask with facial light irradiation. (A) The mechanism of how N-TiO₂ mounted on a mask sterilizes bacteria under light irradiation. (B) Fluorescence microscopic images of E. coli and S. aureus mounted on our masks before and after light irradiation. Survival bacteria fluoresce green, whereas dead bacteria fluoresce red after staining with Live/Dead cell kit. In panel (B), the arrows in (b2) and (c2) indicate live bacteria (green). (C) Photo images of plate incubation of E. coli and S. aureus after washing the mounted bacteria on masks with and without light irradiation. (D) The number of CFUs on the plates. (E) Percentage of survival bacteria on the plates. In both (D) and (E), mask +N-TiO₂/TiO₂ represents deposition of N-TiO₂/TiO₂ at a 3:7 weight ratio onto the mask. (F) Mask rejuvenation under real sunlight. (1), (2), and (3) in panel (F) are the experimental setup for real sunlight irradiation, fluorescence microscopic image of bacteria mounted on the mask, and photo images of plate incubation after real sunlight irradiation, respectively. All scale bars in this figure are 20 μm.

Antibacterial and Photocatalytic Rejuvenation of Masks. As discussed above, the photocatalysts of N-TiO₂ have a decreased energy band gap toward conductive band that can enable their efficient excitation under visible light irradiation. As shown in Figure 4A, the generated free radicals of HO· and ·O₂⁻ under the light irradiation of N-TiO₂ can damage...
the cell walls, cell membranes, RNA, proteins, organelles, or DNA of the deposited pathogens, including bacteria, fungus, and virus, which thus achieve disinfection. By using this strategy, the designed mask possesses antibacterial features. More importantly, the N-TiO₂ containing mask can be facially rejuvenated through short time light irradiation to sterilize the deposited pathogens and for direct reusing.

In our study, we have selected two representative bacteria strains, Escherichia coli K-12 strain MG1655 (E. coli) and Staphylococcus aureus strain HG003 (S. aureus), to test the antibacterial performance and rejuvenating properties of our masks. As shown in Figure S5, two freshly prepared bacteria with a total ∼10⁹ colony-forming unit (CFU)/mL were incubated, mixed in a 1:1 ratio, and then loaded onto the masks with a certain size (Figure S6). Immediately after bacteria loading, the masks were treated with irradiation under a Xenon lamp light (200–2500 nm, 50 W) mimicking 0.1 sun (Figure S7). To avoid the impacts of elevated temperature induced by light irradiation on bacteria growth, we have carefully controlled the light density, the distance between the sample and light source, and irradiation time. As shown in Figure S7, after 10 min irradiation under 0.1 sun (light intensity of 106 W m⁻²), the temperature of the sample surface increased from 25.5 to 34.0 °C. Under this irradiation condition, most bacteria loaded onto the raw masks without any deposition of TiO₂ or N-TiO₂ maintained viability as assessed with a Live/Dead Viability Kit under an epifluorescent microscope (green color in Figure 4B-a2). Similar results were seen in positive controls in which the masks loaded with bacteria were maintained at the same condition but without light irradiation (green color in Figure 4B-a1). These data suggested that short-term light exposures and minor temperature changes have no significant impacts on the viability of bacteria on the mask without the TiO₂ or N-TiO₂. We further loaded the same bacteria titer onto TiO₂, N-TiO₂, and N-TiO₂/TiO₂ mixture deposited masks and investigated their respective disinfection performances. For the mask deposited with either TiO₂ or N-TiO₂, most bacteria were dead (red colors in Figure 4B-b2,c2) after light irradiation for only 10 min. In contrast, most bacteria remained viable without light irradiation (green colors in Figure 4B-b1,c1). Moreover, only one seemingly viable bacterium was detected on N-TiO₂-deposited masks as compared to approximately five viable cells found on the TiO₂-deposited masks under the same 0.1 sun illumination for 10 min (indicated by the arrow in Figure 4B-b2,c2). For the mask deposited with the N-TiO₂/TiO₂ mixture at a weight ratio of 3:7, most bacteria were alive without light irradiation (Figure 4B-d1). In contrast, all bacteria were found to be sterilized after light irradiation (Figure 4B-d2). The same result was observed when we changed the N-TiO₂/TiO₂ mixing ratio to 1:1 (Figure S8A). These data highlighted the superior antibacterial function of N-TiO₂ and the rejuvenating feature of the N-TiO₂ and N-TiO₂/TiO₂ mixture deposited mask under a short term of light irradiation.

Along with bacterial cell viability checking under the confocal fluorescent microscopy, we washed off the preloaded bacteria on masks after light irradiation with 0.85% sodium chloride solutions for further incubation and viability check (Figure S5). The collected bacteria were spread-plated on luria broth (LB)-agar plates and incubated overnight. The CFU number of the survival bacteria was counted thereafter. As shown in Figure 4C,D, 313 CFUs were found on the plate for the raw mask without light irradiation and 318 bacteria were counted on the plate for the raw mask with light irradiation, which indicated that

the mask without photocatalytic TiO₂ or N-TiO₂ does not have antibacterial and rejuvenation function. In contrast, 86 CFUs were counted for the TiO₂-deposited mask after light irradiation (Figure 4C,D), representing 32% of the bacteria survived from light irradiation (Figure 4E). Meanwhile, only one CFU was found on the plate for the N-TiO₂-deposited mask after light irradiation (Figure 4C,D), representing a 99.6% bacteria disinfection by N-TiO₂ under light irradiation (Figure 4E). Notably, no CFU was counted for the mask deposited with the N-TiO₂/TiO₂ mixture at both 3:7 (Figure 4C,D) and 1:1 ratios (Figure S8B), revealing a 100% bacteria sterilization by the N-TiO₂/TiO₂ mixture under light irradiation (also see Figure 4E). All these results together with the Live/Dead cell viability assays corroborated the excellent antibacterial and rejuvenating performance of our N-TiO₂ and N-TiO₂/TiO₂ mixture deposited masks.

Furthermore, to investigate the rejuvenation of our masks under the natural sunlight irradiation, we facially irradiated our mask with the deposition of the N-TiO₂/TiO₂ mixture (3:7) under the real sunlight (light density was measured at 664 W m⁻²) for 10 min (Figure 4F1). The surface temperature of the mask was increased from 16.1 to 27.6 °C (Figure S9), which is much lower than that irradiated under the solar simulator (Figure S7), which suggests no significant impacts of real sunlight and the elevated temperature on the viability of the bacteria. As deciphered in Figure 4F2, only dead cells were observed under the fluorescence microscope after the irradiation. In the meantime, we did not find any survival bacteria after plate incubation (Figure 4F3). All these data demonstrated excellent photocatalytic rejuvenation of our mask under real sunlight irradiation.

Overall, nitrogen doping of TiO₂ has enabled near complete disinfection of both E. coli and S. aureus loaded on the masks by 0.1 sun simulator irradiation for only a short period (10 min). Depositing the mixture of N-TiO₂ and TiO₂ has completely disinfected the bacteria loaded on the masks under the irradiations of both the 0.1 sun simulator and real sunlight. Our mask with deposited N-TiO₂/TiO₂ thus has superior antibacterial and photocatalytic rejuvenating properties in that the used mask can be sterilized by facially irradiating under light for reusing. This simple and efficient mask rejuvenation could also address the challenges in mask shortage, mask reusing, and waste mask handling.

Mechanical Performance, Filterability, and Breathability of Masks. In addition to the superior antibacterial and rejuvenating properties, the abundant hydrogen bonding system, nanofibrous structures, and strong electrostatic attraction properties equipped our mask with excellent mechanical performance, infiltrability, and breathability, all of which together with the hydrophobicity represent inherent characteristics of a high-quality mask. These performances of our mask (N-TiO₂/TiO₂ deposited PVA+CNF+PEO) were compared with those of a commercial surgical mask (Figure S10).

First, the enhancement of CNF itself along with the abundant hydrogen bonding formed between CNF and the precursor polymers has largely improved the mechanical performances of our mask, which is an important property of the mask to resist possible external physical damage for reusing.

CNF has formed ample hydrogen bonding between PVA and PEO. Plentiful hydroxyl groups exist in both PVA and CNF molecules, and ether linkages containing oxygen in PEO could serve as hydrogen bonding donors (Figure 3C). As deciphered
by the stress–strain curves (Figure 5A) and the calculated elastic modulus and tensile strength (Figure 5B), our masks had a more than 10-fold higher elastic modulus (1853 MPa vs 174 MPa) and more than 2-fold higher tensile strength (21.4 MPa vs 10.7 MPa) than the commercial mask. Furthermore, CNFs as crystalline nanofibers have inherently excellent mechanical properties that have been widely used to enhance the polymer composite.22,23 As compared with the as-electrospun nanofibers with and without CNF, the mask with CNF has increased the tensile strength from 39.9 to 58.9 MPa (1.48-fold) and the elastic

Figure 5. Mechanical strength, airflow, and particle filtration of masks. (A) Stress–strain curves. (B) Elastic modulus and tensile strength. (C) The schematic of measurement of the filterability and breathability. (D) Air flow rate and particle filtration of masks. (E) The mechanism of the infiltration of masks. (F) Electrostatic attraction of torn tissues by the filter of the commercial mask (1) and our as-electrospun mask (2). The unit lpm in panel (D) represents liters per minute.
modulus from 368 to 503 MPa (1.37-fold) (Figure S11 and Table S2). In addition, the ultimate strain at the fracture of our mask (Figure 5A) was much lower than that of the commercial mask PP filter, which can be attributed to the esterification treatment that has decreased the ductility of the mask (as comparing the PVA+CNF+PEO with and without the esterification, Table S2). All these data suggested the superior mechanical performances of our masks exceed the commercial counterpart in the elastic modulus and tensile strength.

Along with the superior mechanical properties, our mask also exhibited excellent filterability and breathability, which have been enabled by the electrospinning technology. Under the ASTM F2100-19E1, the filterability and breathability can be measured by filtering a NaCl aerosol mimicking the virus and bacteria filtration. Since the COVID-19 virus has a diameter at around 100 nm,24 the NaCl aerosol used in our test had even a smaller diameter of 75 nm that can effectively evaluate the filterability of the masks. As shown in Figure 5C, the mask was placed into an automated filter tester, where the aerosol was generated and then flowed to pass through the mask. The upstream aerosol above the mask and the downstream aerosol after the mask filtration were measured to calculate the filter efficiency; meanwhile, the airflow and the pressure through the mask were detected by a transducer, which represents the breathability of the mask. The filtration efficiency and airflow of our masks were compared with those of the commercial mask. As demonstrated in Figure 5D, our mask had a filtration efficiency of 98.7%, which is much higher than that of the commercial mask (81.6%). Along with the high filtration efficiency, the air flow rate of our mask (83.4 lpm) was close to that of the commercial mask (84.9 lpm), which suggested the good breathability (Figure 5D).

The excellent filterability can be attributed to the structure and static electrons generated by the electrospinning. The filtration mechanism of masks was as displayed in Figure 5E.25 First, the large particles can be physically captured by inertial impaction and interception of the fiber mesh, while the small particles can be hindered by diffusion. Our masks fabricated by electrospinning had fine nanofiber structures (Figure 3E) that could have larger contact surface areas with the particles to
physically resist their penetration. Besides the physical filtration, electrostatic attraction can hinder both large and small particles (Figure 5E). Electrospinning can effectively charge the fabrics and thus significantly improve the filtration efficiency through the electrostatic attraction.26 Our electrospinning was carried out under a voltage as high as 30 kV, which has enabled the fabricated masks with excellent electrostatic attraction (Figure 5F2) that can attract torn tissues even more than the commercial mask does (Figure 5F1). Our mask with fine nanofiber structures and strong electrostatic attraction thus has enabled its excellent filterability.

In regard to the good breathability, it could be attributed to the porous structures of the electrospun nanomesh (Figures 3E and S3A). As deciphered in Figure 5E, the air can flow through these pores with the particles resisted by the nanomesh. Our electrospun nanomesh had porous structures with much smaller pore size and higher density than the commercial mask (Figure S3); therefore, the high airflow has not sacrificed the filterability of the mask. These fine porous structures together with the electrostatic attraction have synergistically enhanced the filterability and the breathability.

Although our mask has excellent properties in photocatalytic rejuvenation, mechanical strength, filterability, and breathability, a possible concern using TiO2 in the mask could be aroused from its toxicity. On the one hand, TiO2 has been widely used in both food and pharmaceutical industries; on the other hand, the toxicity of TiO2 nanoparticles has been reported, such as interfering with immune cells, promoting tumors, and inhibiting cell growth.27 Such toxicity is dependent on many factors, such as the size, shape, surface area, dose, and phase of the applied TiO2 nanoparticles.27,28 We thus characterized the possible leakage of the deposited TiO2/N-TiO2 from the mask by mimicking the human breath. As shown in Figure S12A,B, a mask was put in a vial with an inlet for air to flow in and an outlet for air to flow out so that the possible TiO2 leaking can be estimated by comparing the particle counts in the flow-in air (air in ambient environment) and the flow-out air. We used two PortaCount systems to detect the particles in the ambient and in the airflow through the mask separately (Figure S12B), as reported previously.29 As shown in Figure S12C, the particle count in the air flow after blowing the mask was similar to that in the ambient environment. We also calculated the relative particle count as per the particle count in the ambient environment. With either the vial with and without the masks, the relative particle count was around 100%, which indicated that the air flow through the vial with the mask did not render significant particle leakage. We further evaluated the possible TiO2 leakage under an environment with elevated humidity. The relative humidity was controlled at 80% RH by a humidifier. As shown in Figure S12D, both particle counts and relative particle counts in the high humidity air flow through the vial with a mask did not significantly increase, as compared with that without a mask. Moreover, the weight of the mask (0.0243 g) after the TiO2 leaking test kept constant with that before the test (Figure S13). All these results suggested no significant particle leakage from the mask, neither under ambient environment nor under a high humidity air flow. This could be attributed to the tight interaction of the TiO2/N-TiO2 with the nanofibers in the mask. Additionally, Laomettachit et al.27 have proved that TiO2 nanoparticles with a low dose are negligible for tissue damage, because of the renewal of cell division and the recovery of tissue mass. Considering the low dose used for TiO2/N-TiO2 deposition (29.7 mg/cm² of mask or 6.012% of mask weight) and the ignorable leakage, our mask should be safe for utilization, although further clinical research might be needed in the future to validate it.

Cycling Performance of Masks, Filtration Efficiency Testing for Wearing, Cost Analysis of Raw Materials, and Degradability/Dissolution in Water and Soil. To evaluate the potential of practical application of our mask, we further tested its cycling performance and filtration efficiency after wearing by the author and analyzed the cost of raw materials. First, the cycling performance of our mask was evaluated by its photocatalytic rejuvenation and the filter efficiency after each cycling test. The testing of filtration efficiency was conducted by using the PortaCount system as introduced in the TiO2 leaking testing (Figure S12B), but with a different self-designed sampling method. As shown in Figure 6A, the testing was performed in a closed room filled with particles along with the bacteria in advance by a particle generator, where the bacteria were loaded into the NaCl solution for particle generation. The particles in the room passing through the mask were counted by the PortaCount, and then the deposited bacteria on the mask went through the photocatalytic treatment for bacterial disinfection and rejuvenation. The mask was tightly sealed onto a vial with adhesive tapes to avoid possible leaking, and a grommet was fixed on the mask to allow air carrying particles to flow from the vial (in the mask) into the PortaCount system (Figure 6B).

As the first step of cycling performance testing, we decided on the period for each cycling according to how much bacteria can be deposited on the mask. As shown in Figure 6D-a1,a2, after 5 min filtration, we found sufficient bacteria have been deposited onto the mask by plate incubation and fluorescence microscopy observation, respectively. Therefore, the experimental conditions for the cycling performance testing were set as 5 min filtration that was followed by 10 min light irradiation, and the testing was repeated for three cycles (Figure 6C). As shown in Figure 6D, most bacteria were sterilized after each cycling. Especially, after the first and second cycles, only dead bacteria were found under fluorescence microscopy and no survival bacteria were found for plate incubation. We counted the CFUs on plates and calculated the percentage of the survival bacteria as related to the control (only 5 min filtration without light irradiation). As shown in Figure 6E,F, our mask maintained nearly 100% bactericidal ability throughout. Subsequently, we dynamically counted the particles in the room (left, PortaCount tester in Figure 6B) and the particles entered into the mask (right, PortaCount tester in Figure 6B) during each cycle. As shown in Figure 6G, no significant decrease in the filtration efficiency was detected after three cycling tests. All these data demonstrated the excellent cycling performance of our mask in photocatalytic disinfection, rejuvenation, and filtration.

Second, we tested the filtration efficiency of our mask worn by the author to demonstrate its practical application. As shown in Figure 7A, we cut a commercial mask and replaced its two outer layers with our material. After sealing the two outer layers, our prototype mask was quite similar to the commercial one but only with a slightly smaller size (Figure 7B). Our prototype mask also had good wearability as the commercial mask did, with good facial coverage (Figure 7C). In order to prevent possible leaking during the dynamical measurement of filtration efficiency for a long term, we covered both the commercial mask (Figure 7D1) and our prototype mask (Figure 7D2) by using nylon overlayers as reported previously.29 As displayed in Figure 7E, our mask had a stable filtration efficiency higher than 90% even after
continuously wearing for more than 120 min, while the filtration efficiency of the commercial mask began to deplete below 90% after about 65 min wearing. This depletion of the filtration efficiency continued to about 80% after 90 min wearing, which is significantly lower than that of our mask when wearing for the same term. All these results highlighted the great advantage of our mask over the commercial one regarding the stability in the filtration efficiency for practical utilization.

We further analyzed the reason for the superior filtration efficiency of our mask over the commercial one, which could be enabled by the fine nanostructures of the electrospun nanomesh. As revealed by the SEM analysis, our mask (Figure S3A) had much finer pore structures than the commercial masks (Figure S3B). We thus analyzed the distribution of the deposited particles in both masks by SEM after wearing and the filtration efficiency testing. Most particles or bacteria were found to deposit on the surface of our mask (Figure 7E2), whereas these particles/bacteria were found to enter the inside of the commercial mask (Figure 7E1). SEM analysis of the cross sections of both masks after wearing and filtration testing further confirmed that the particles entered the inside of the commercial mask (Figure S14A,B) but only deposited on the surface of our mask (Figure S14C,D). The fine pore structure of our mask out of electrospinning thereof has resisted the entrance of the particles/bacteria into the mask.

These results together with the cycling performance further highlighted the excellent rejuvenation performance, stable long-term filtration efficiency, and practical implementation of our mask.

Third, we analyzed the cost of raw materials used for fabricating our masks. As estimated in Table S3, the main components of PVA, PEO, CNF, stearic acid, and TiO2 were considered for cost calculation. In the meantime, the costs of raw material of PP for making most commercial masks and the nonwoven meltblown filter fabric for the commercial mask were compared with our mask. The estimated total cost of raw materials of our mask is at $0.0211/m² mask (or $1.2584/kg mask), which is about half of the price of PP ($0.0470/m² mask, or $2.10/kg mask) and one-third of the nonwoven fabrics ($0.0650–0.0874/m² mask, or $2.9–3.9/kg mask), demonstrating the cost-effectiveness of our mask. The main contribution to the cost of our mask was PVA, which is followed by the stearic acid. The costs of these two raw materials were calculated at $0.0113/m² mask (or $0.6725/kg mask) and $0.0064/m² mask (or $0.3810/kg mask), accounting to 3.6% and 30.3% of the total cost, respectively. Most costs from PVA and stearic acid might be because these two polymers are the main components of our mask (Table S4). As a contrast, the cost of photocatalytic N-TiO2 was estimated at $0.0016/m² mask, contributing only 0.6% to the total cost. The relatively low contribution of N-TiO2 to the cost is attributed to its lower usage, whose amount deposited on our mask was measured at 29.7 mg/cm³ mask (6.0% of mask weight, Table S4). Overall, in addition to the excellence in photocatalytic rejuvenation, mechanical strength, and reusability, our mask also represents a cheap and biodegradable mask that addresses the challenges in sustainable development of personal protection equipment.

Finally, our mask displayed good degradability/dissolution in both water and soil. We used both tap water and artificial seawater (ASW, 3.5% of sea salts solution) to mimic river water and seawater, respectively. The testing was carried out at both room temperature and 80 °C for 3 days. In both tap water (Figure S15A,C) and ASW (Figure S15D,F) at room temperature, our mask was partly degraded/dissolved, whereas the mask was completely degraded/dissolved in both tap water (Figure S15B,C) and ASW (Figure S15E,F) when the water was kept at 80 °C for 3 days. These results suggested that our mask is degradable in both river and sea. Moreover, we buried our masks along with the commercial masks in soil to test their biodegradability/dissolution. As shown in Figure S15G, after 10 days burying, a very slight change was observed in the morphology of our masks, whereas the buried commercial masks did not show apparent changes. After burying in soil for 1 month, our masks completely disappeared but the commercial masks remained intact. These results indicated that our mask is also degradable in soil. All the above results highlighted the degradability of our masks.

In summary, endowed with the enforcement of CNF, the abundant hydrogen bonding, the fine nanofiber and porous structure, and electrostatic attraction, the masks made in this study had excellent mechanical performance, filterability, and breathability that exceed or are comparable with those of commercial masks. Along with the photocatalytic rejuvenating feature, reusability, biodegradability, and environmental friendliness, our material represents a promising alternative to the manufacture of masks for personal protection during this ongoing COVID-19 pandemic.
CONCLUSION

In this study, the concerns of a second infection and environmental impacts by the massive waste masks have been addressed by developing a rejuvenation strategy to fabricate a revivable, biodegradable, and reusable mask through electrospinning technology and employing a photocatalytic N-TiO₂. The precursor polymers of our mask were composed of PVA, CNF, and PEO, which are inherently biodegradable and environmentally friendly. Utilization of these biodegradable polymers instead of the synthetic plastic of PP as the raw materials for mask manufacturing can thus facilitate the handling of the used masks and significantly reduce their environmental impacts. The fabricated mask with deposited N-TiO₂/TiO₂ can be facially rejuvenated by irradiating under 0.1 sun light (200–2500 nm, 106 W m⁻²) or real sunlight (664 W m⁻²) for only 10 min to achieve 100% bacteria disinfection. The employed electrospinning technology for mask manufacturing has concurrently enhanced its filterability and breathability, in which the produced fibers maintained uniform diameters and strong electrostatic attraction have endowed the mask with superior filterability of pathogens and the micropores formed between the nanofibers enabled the excellent breathability along with the superior filterability. In the meantime, the enforcement strategy has been developed by using CNF to increase the mechanical strength of masks. The CNF itself with excellent mechanical strength enforced the nanofibers. The short chain PEO polymers served as the plasticizer to improve the spinnability of the PVA and CNF blend, while CNF formed complex hydrogen bonding among PVA and PEO to further increase the mechanical properties of the mask. The resultant mask thereof had synergistically high mechanical strength, filterability, and breathability. Moreover, the fabricated mask exhibited excellent cycling performance in filtration and photocatalytic rejuvenation, good wearability tested by the author, stable long-term filtration efficiency exceeding that of the commercial mask, and cost-effectiveness in raw materials. These qualities adding together have demonstrated the great potential of our mask in practical application. The advances in such a reusable, biodegradable, and high-quality mask could also inspire the utilization of other biodegradable and renewable polymers, photocatalytic materials, and advanced technologies for manufacturing next generation PPEs with minimum post-handling and environmental impacts.

EXPERIMENTAL SECTION

Materials. Poly(ethylene oxide) with an average molecule weight of 1500 g mol⁻¹ and stearic acid were purchased from Sigma-Aldrich. Polyvinyl alcohol (hydrolyzed, code: S25473B) was purchased from Fisher Science Education. Before the utilization, PVA was dissolved into water at 90 °C for 3 h to get a solution with the concentration of 8 wt %. Titanium dioxide (Degussa P-25) was purchased from Sigma-Aldrich. All chemicals and materials were used as received.

Nitrogen Doping of TiO₂. Doping of Degussa P-25 was conducted as reported before. Briefly, urea was mixed with TiO₂ at a weight ratio of 4:1 and then ground manually by a mortar and pestle for 20 min. The mixture of urea and TiO₂ was then annealed under an air atmosphere in a tube furnace (1200X, MTI Corp.) with a temperature of 400 °C, heating rate of 10 °C min⁻¹, and holding time of 1 h. After the annealing, the mixture was washed with deionized water for three times before drying at 70 °C. The prepared samples were then ground into powders before use.

The morphology and elemental analysis of the N-TiO₂ were characterized by using a scanning transmission electron microscopy (S-TEM, FEI Titan Themis 300 system) with EDS mapping. The band gaps of the TiO₂ and N-TiO₂ were determined by using a UV–visible/NIR spectrophotometer (V-770, JASCO, Easton, MD).

Preparation of CNF. CNF was prepared by TEMPO (2,2,6,6-tetramethylpiperidine-1-oxide radical)-mediated oxidation, followed by mechanical disintegration in water. First, softwood pulp (10 g) was immersed into aqueous TEMPO (0.02 g/g of pulp) and NaBr (0.15 g/g of pulp) solutions. After that, 12 mmol/30 g pulp of 12.5% NaClO solution was added dropwise. Aqueous NaOH solution (0.5 mol/L) was then used to adjust the pH value to 10.5, and this pH value was kept by using the same NaOH solution during the reaction. When the pH value was stable and unchanged, the oxidation reaction was quenched by adding 5 mL of ethanol. The resultant TEMPO-oxidized cellulose was then filtered and neutralized with deionized water. Finally, the oxidized cellulose was suspended in water at a consistency of 2.0 wt %. The suspension was ultrasonicated at 4 °C for 60 min to get the final product of CNF.

Fabrication of Mask. Electrospinning. The masks were prepared by using an electrospinning unit (AEM-NFES-12, AEM Energy Co., Ltd., Linyi, China). The electrospinning dopes were composed of PVA, 2% of CNF (based on the dry weight of PVA), and 10% of PEO (based on the total dry weight of PVA and CNF). First, PEO was dissolved in 0.5 g of water. After that, CNF was added into the PEO solution. After homogeneously mixing PEO and CNF with a mixer (DAC 330-100 PRO, FlackTek SpeedMixer, Landrum, SC), 10 g of PVA solution (8 wt %) was added and then mixed. For electrospinning, the dopes were injected with a 10 mL syringe with the needle of 22 gauge. The electrosprining was carried out under 30 kV voltage, 2 mL/h injection speed, 15 cm needle-to-collector distance, and about 45% relative humidity (RH).

Esterification. As-electrospun masks were esterified by stearic acid to enable their hydrophobicity. Stearic acid (5 wt %) was dissolved in ethanol under 30 °C. The as-electrospun masks were immersed into the stearic acid solution for 15 min. After that, the esterification reaction was carried out by placing the mask into a 100 °C furnace for 15 min as reported.

NIR spectra of TiO₂/N-TiO₂ Deposition. As the final step, TiO₂ and N-TiO₂ were deposited onto the mask. The surfactant of cetyl trimethylammonium bromide (CTAB) was dissolved into ethanol at a concentration of 1.0 wt %. After that, TiO₂ and N-TiO₂ with the dosage of 1.0 wt % were dispersed into the ethanol. The esterified mask was then immersed into the TiO₂/N-TiO₂ suspension for 10 min. After the deposition, the mask was taken out of the TiO₂/N-TiO₂ suspension, air-dried, and stored for utilization. The amount of N-TiO₂ and TiO₂ deposited on the mask was calculated by the weight increment after the deposition, which was at 29.7 mg/cm² mask (or 6.012% as shown in Table S4) for both N-TiO₂ and TiO₂.

Photocatalytic Rejuvenation and Antibacterial Testing of Mask. The procedure for photocatalytic rejuvenation and antibacterial test was as shown in Figure S5. Strains of E. coli and S. aureus were cultured in lysogeny broth (LB) medium at 37 °C for 16 h. After incubation, 0.5 mL of E. coli and 0.5 mL of S. aureus were then washed, combined, and resuspended in 0.85% NaCl solution to get 1 mL of cell mixture, with a cell density of 10⁶ CFU/mL. After that, the cell mixture was diluted 10-fold with the above NaCl solution into a cell density of 10⁵ CFU/mL mask (without TiO₂ or N-TiO₂, with TiO₂, and with N-TiO₂) material cut into the size of 10 cm × 5 cm (Figure S4) was loaded with 100 μL of the bacteria mixture. Right after loading the bacteria, the masks were treated under light irradiation by using a solar simulator (HSX-F300, NBeT, China). Light density was measured by using a solar power meter (VLP-2000, NBeT). The distance between the samples and the light was optimized and kept at 25 cm (Figure S6), and the irradiation was carried out for 10 min. The temperature was detected by using a thermal image camera (HT-18, HTI, Dongguan, China). Right after the light irradiation, the masks were washed twice with 3 mL of 0.85% NaCl solution and finally resuspended into 200 μL of NaCl solution (D0 in Figure S5). The viability of bacteria in D0 was directly assessed via Live/Dead cell viability assays under a fluorescence microscope (Axioplan 2 with HBO 100 illuminating system, ZEISS, White Plains, NY). The rest of the cell suspension (100 μL) underwent
serial dilution and further viability assessment (CFU counting). Briefly, the D0 cell suspension was diluted to D1, D2, and finally D3, with a dilution factor of 100. After that, 100 μL of D3, with an estimated cell density of 10^5 CPU/mL, was spread-plated onto LB agar plates. The plates were then incubated overnight (~16 h), and the CFUs were counted to report the number and ratio of the survival bacteria under the light irradiation.

The controls without the light irradiation for all masks (without TiO2 and N-TiO2, with TiO2, and with N-TiO2) were kept in the dark until the start of the assays. The observations of survival bacteria under fluorescence microscopy and by plate incubation were carried out in the same way presented above.

To test the photocatalytic rejuvenation and antibacterial performance of the mask under the real sunlight, the mask was cut, followed by bacterial loading as presented above. After that, the sample was immediately transferred into real sunlight for irradiation as displayed in Figure 4F1. All other experimental conditions were the same as those of the sample treated by the solar simulator.

**Characterization of Mask. Scanning Electron Microscopy (SEM).** The morphologies of the masks were observed under a Hitachi S4800 SEM, which was operated at 10 kV voltage and 8 mm working distance. Before the observation, the samples were coated with 10 nm gold/palladium.

**Mechanical Test.** The mechanical tests were carried out by using an Instron 5567 unit (Norwood, MA). The load cell used was 1 kN, and the displacement speed was set at 10 mm min^-1. For the measurement, the samples were cut into the size of 25 mm in length and 3 mm in width. To calculate the area for plotting the stress-strain curves, the thicknesses of masks were measured. Our mask has a thickness of 34 ± 2.8 μm, which is much smaller than that of the commercial mask used in this study (80 ± 3.2 μm).

**Contact Angle Test.** The contact angles of all masks were measured by a contact angle analyzer (Phoenix 150, SEO, Korea) at room temperature. The sample was mounted by using adhesive tapes onto a glass slide for the measurement. For each sample, two measurements were recorded. The contact angle of all masks was measured by a contact angle analyzer (Phoenix 150, SEO, Korea) at room temperature.

**Filterability and Breathability Test.** An automated filter tester, TSI Model 8130A (Shoreview, MN), was used for particle filtration and air flow testing. The sample was cut into a size of 6 in. by 6 in. The flow rate (liter per minute, lpm), pressure drop (mm H2O), and penetration (%) were recorded. The filtration efficiency (%) was calculated by (100 – penetration).

**TiO2 Leaking Test.** To test the possible leakage of the TiO2 from the mask, we used two PortaCount Plus Model 8028 instruments (TSL Inc., Shoreview, MN) with a self-designed small vial with both an inlet and an outlet for air flow (Figure S12). The mask was sealed in the vial, and the outlet of the vial was connected to the PortaCount system to count the number of existing particles. The air flow was kept at 1.67 cm^3/s, which mimics human breath. The operation of the PortaCount system and the collection of the data were as reported before. To test the TiO2 particle leaking under a high humidity air flow, the humidity was controlled at 80% RH by using a humidifier. The relative particle count was calculated from the particle in the air flow by dividing that in the ambient environment.

**Cycling Performance Testing.** The cycling performance of the mask was tested by using the above-mentioned PortaCount system. The bacteria were loaded into the NaCl solution used for particle generation. Before the testing, the room was continuously filled with particles for 20 min. Each cycling was carried out for 5 min filtration, followed by bacterial counting using both fluorescence microscopy and plate incubation as presented above. Three cycles were conducted in total.

**Wearability Testing.** The mask was worn by the author to test the wearability and long-term filtration. The filtration efficiency was tested in the way as cycling performance testing but with a time longer than 90 min. After the testing, the distribution of the particles in the mask was analyzed by the same SEM as presented above.

**Degradability/Dissolution Testing.** The degradability or dissolution of our masks was tested in both water and soil. Tap water and artificial seawater (3.5% aqueous sea salt solution) at both room temperature and 80 °C were used. The mask was cut in a sheet and then put in around 1 L of water for 3 days. After that, the mask that remained in water was picked up and dried. The weight of the mask before and after keeping in water was measured. To test the degradability/dissolution of the mask in soil, our mask was buried in soil for 1 month. The morphological change of the mask was recorded at 10 days and 1 month.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsnano.1c03249.

Figures showing EDX of N-TiO2, viscosity of electrospinning dopes, SEM images of both surfaces and cross sections of our mask and a commercial mask, contact angle of mask, schematic of antibacterial and photocatalytic rejuvenation test, photo images of as-electrospun mask, thermal images of temperature distributions under mimicking sunlight and real sunlight, fluorescence microscopy and photo images of bacteria mounted on mask, photo images of a commercial mask, stress—strain curves and the calculated mechanical performance, TiO2 leaking test setup and results, and biodegradability/dissolution test setup and results of our mask in both water and soil; tables containing composition of mimicking sunlight, details of the calculated mechanical performances, details of cost estimation, and composition of our mask (PDF)

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