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Always-on photocatalytic antibacterial facemask with mini UV-LED array

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ABSTRACT

The facemask is a device to protect yourself and others against pandemics, such as coronavirus disease 2019 (COVID-19), and adding a functional filter to the facemask could offer extra protection against infectious microbes (such as bacteria and viruses) to the wearer. Here, we designed and fabricated an always-on photocatalytic antibacterial facemask, which comprised a reusable polypropylene filter layer coated with the photocatalytic laminated ZnO/TiO2 bilayer and a separate UV-LEDs layer to supply UV light whenever necessary. The fabricated photocatalytic filter was able to be directly inserted into the reusable facemask together with the UV-LEDs layer. This facemask could be used repeatedly and sustainably anytime and anywhere regardless of solar illumination. The photocatalytic filter exhibited an excellent photocatalytic antibacterial effect likely due to recombination suppression of electrons and holes of ZnO/TiO2 bilayer and wetting transition from hydrophilic to superhydrophilic state on the surface of the filter. Thanks to the kirigami pattern in both photocatalytic filter and UV-LEDs layer, full-face covering, breathability, flexibility, and the snug fit are believed to be improved. Although further in-depth studies are still needed and there is a long way to go, we expect our design idea on the facemask to be considered in various fields.

1. Introduction

“An ounce of prevention is worth a pound of cure,” as Benjamin Franklin said. The wearing of a mask can cut down the chances of both transmitting and catching the microbes [1–3]. The chance of infection is revealed to be 3.1% with a facemask, as compared to 17.4% without a mask [4]. Unquestionably, wearing a facemask is one of the best preventable means against various contagious diseases, and adding a functional filter to the facemask could offer extra protection to the wearer. Most of the disposable facemasks contain a polypropylene (PP) melt-blown fabrics filter to partially prevent airborne pathogens such as fungi, bacteria, and viruses. However, the disposable mask with the PP filter has a limitation in the hours of use and the number of uses because of the seriously depleted filtering efficiency of the filter [5]. This limitation also has led to the environmental problem related to the enormously generated plastic wastes [6]. Besides, the abandoned masks after use can also induce secondary infection [7]. Recent studies have shown that the filters made of photocatalytic materials could not only inactivate the airborne pathogens but also enable the reuse of the mask under UV light [8–10]. However, the challenge is that the antiviral or antibacterial activity of the photocatalytic filter cannot be expected without UV and thus can work mostly only in the daytime under solar illumination. If it is inconvenient to disinfect the photocatalytic filters using UV in daily life, the used filters could also naturally become single-use plastic products, thereby posing even more serious environmental risks than the conventional filters. To resolve this issue, a more practical solution is needed and likely the highly reusable photocatalytic filter could be a sustainable option. The performance of the photocatalytic filter should be, of course, prefentially reliable and more importantly, UV sources need to be always accessible. It means that a new design of a facemask with an all-in-one photocatalytic filter and UV source is required. Here, we designed and fabricated a reusable photocatalytic antibacterial facemask decorated with a mini UV-LED array that can be used anytime and anywhere, regardless of solar illumination. The mask comprised a reusable polypropylene filter layer coated with the photocatalytic laminated ZnO/TiO2 bilayer and a separate UV-LEDs layer.

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layer to supply UV whenever necessary. These two layers were integrated into a conventional facemask to produce an always-on antibacterial facemask. What is more, the mask allowed a comfortable full-face covering when wearing thanks to the kirigami pattern applied on both photocatalytic filter and UV-LEDs layer (from the Japanese, “kiri,” meaning to cut, and “kami,” meaning paper [11]).

2. Experimental section

2.1. Preparation of PP based photocatalytic filter

PP melt-blown fabric (thickness \( \approx 200 \) \( \mu \)m) was cut into square shapes (\(-5 \times 5 \) cm\(^2\)) and then loaded into an ALD chamber (S200, Savannah, Cambridge NanoTech Inc.). The deposition process of ZnO or TiO\(_2\) or laminated ZnO/TiO\(_2\) bilayer was performed at the working temperature of 70 \(^\circ\)C and pressure of \(-0.1\) Torr. Diethylzinc (Zn(C\(_2\)H\(_5\))\(_2\), DEZ, Sigma-Aldrich) and deionized water were used as precursors for ZnO deposition. The ZnO was deposited with the exposure mode (each cycle: 0.02 s pulse, 20 s exposure, and 30 s purge of DEZ; followed by 0.1 s pulse, 20 s exposure, and 30 s purge of deionized water). Similarly, TiO\(_2\) was also deposited with exposure mode (each cycle: 1 s pulse, 20 s exposure, and 30 s purge of titanium isopropoxide (Ti(OiPr)\(_4\), TIP, Sigma-Aldrich), followed by 1 s pulse, 20 s exposure, and 30 s purge of deionized water). A laminated ZnO/TiO\(_2\) bilayer was built by depositing ZnO at first, then continuously depositing TiO\(_2\) in one process. The thickness of each metal oxide layer was controlled by adjusting the number of deposition cycles. For each experiment, a piece of Si wafer was also loaded into the ALD chamber for growth rate measurement. The measured growth rates of ZnO and TiO\(_2\) were \(-2.5\) Å/cycle and \(-0.7\) Å/cycle, respectively. The denotation of each sample was based on the thickness of metal oxide films deposited on the PP filter. For example, PP/Z25, PP/T25, and PP/Z25T5 mean that PP with ZnO (~25 nm thick) after 100 cycles deposition, TiO\(_2\) (~25 nm thick) after 350 cycles deposition, and PP with laminated ZnO (~25 nm thick after 100 cycles deposition) and TiO\(_2\) (~5 nm thick after 70 cycles deposition), respectively.

2.2. Preparation of UV-LEDs layer

To fabricate the UV-LEDs layer, using Sn-based solder pastes, we transferred UV-LEDs (UV-A type, LHUV-0385-A040, LUMILEDS, \( \lambda = 385 \) nm, dimension of 1.7 mm \( \times \) 1.3 mm \( \times \) 0.68 mm) onto the flexible printed circuit board (FPCB) strip which is consisted of polyimide (thickness of \(-25 \) \( \mu \)m), copper, and gold. Then the long strip of the FPCB decorated with UV-LEDs was attached to PP fabric using the epoxy glue (Figure S1). To light the UV-LEDs, we applied an electric potential of 3 V between the electrodes of the FPCB.

2.3. Characterizations

Surface morphology and elemental composition of the samples were analyzed by a field-emission scanning electron microscope (FESEM, JSM-7000F, JEOL) equipped with energy-dispersive X-ray spectroscopy (EDX, Aztec X-Max, Oxford Instrument). X-ray diffraction (XRD) analysis was performed by a Panalytical Empy- rean diffractometer (Cu–K\(_x\) radiation, 40 kV, 30 mA, \( \lambda = 1.5418 \) Å). The water contact angle (CA) measurements were performed using a Drop Shape Analyzer system (DSA100, KRUSK GmbH) and repeated for several drops on different positions at each sample. UV-vis diffuse reflectance spectra (DRS) were obtained using a V-770 UV-Vis/NIR spectrophotometer. The room temperature photoluminescence (PL) emission spectra (excitation at 320 nm) were collected on a Horiba spectrophotometer (FluoroMax Plus). Uni-axial tensile tests of as-prepared samples were measured by a tensile tester (Tytron 250, MTS co.). The filters (raw PP and as-prepared photocatalytic PP filters) were carefully cut with a knife (8 mm-wide and 50 mm-long rectangular strips). The uniaxial tensile measurements were carried out at room conditions by a displacement control method (grip to grip Laser Extensometer, MTS CO., LX series) with a speed of 5 mm/min. The sample was extended until rupture occurred. Force (mN)–strain (%) data for each specimen were exported from the software of the machine and subsequently, all data were rescaled into the engineering stress (\( \sigma \))–strain (\( \varepsilon \)) curve. The thickness of each sample was measured by scanning electron microscopy (SEM) before and after ALD, respectively. For each measurement, at least eight identical samples were prepared and measured under the same conditions and one typical data set was selected for demonstration. All graphical works including data rescaling were performed with ORIGIN 2020.

2.4. Antibacterial test

Rod-shaped Gram-negative bacterium Escherichia coli (E. coli) (Biozoa Biological Supply, South Korea) was used for the antibacterial tests of the photocatalytic filter layers. Colony-forming units (CFU) were determined by the standard spread-plate colony count method. To prepare the E. coli suspension for testing, initial E. coli suspension (denoted as E. coli suspension 1) was diluted to 1:10 000 by deionized water (denoted as E. coli suspension-2. \(-5 \times 10^6\) CFU/mL). Then 10 mL of the E. coli suspension-2 was pipetted carefully into the glass vial, followed by adding the testing samples and covering tightly. The testing samples were prepared by cutting the raw PP filters (without treatment) and the photocatalytic PP filters (with TiO\(_2\) or ZnO coating) into a circle shape with a diameter of 14 mm (area \(-1.54 \text{ cm}^2\)) using a hand-operated punching tool (hole size: \( \approx 14 \) mm, WC-H125, WLCOS). A reactor for the antibacterial experiments (Figure S2) was composed of the Standard Doctor Capsule Multi Sterilizer to support the ultraviolet UV-C rays (\( \lambda = 100–280 \) nm) and a shaker to continuously stir at 300 rpm during each experiment. The photocatalytic antibacterial inactivation of the E. coli cells was assessed by pipetting a 10 \( \mu \)L volume of the E. coli suspension in each of the testing samples after 5, 10, 15, 20, 25, and 30 min, followed by carefully spreading onto agar plates and incubating overnight at 35 \(^\circ\)C. The number of colonies was counted easily after the incubation period, and the antibacterial efficiency (or E. coli survival) was calculated by equation (1). For the recycling test, after 15 min of 1st run, the sample was taken out and washed carefully with deionized water, followed by drying at 70 \(^\circ\)C for 1 h. Then, continuous testings and washings (2nd, 3rd,4th, and 5th run) were repeated. All the experiments were repeated at least three times with different PP filter sets. E. coli survival ratio was calculated using the following equation:

\[
E. \text{coli survival} = \frac{C_0 - C_t}{C_0}
\]

where \( C_0 \) and \( C_t \) are the numbers of colonies at the initial time (0 min) and at time \( t \) (min), respectively. After obtaining the values of E. coli survival ratio, the values were averaged. To measure the mass loss of the PP/Z25T5 filter after each test/washing procedure, at least 3 different filters were used and the masses were carefully measured using an analytical balance (SECURA225D – 1SKR, Sartorius Lab Instruments GmbH & Co. KG) and averaged.

3. Results and discussion

3.1. Analysis of photocatalytic PP filter

Photocatalytic properties of various metal oxide semiconductors, such as zinc oxide (ZnO) and titanium oxide (TiO\(_2\)),
have received endless attention in the field of photocatalysis [12,13] and lots of research are ongoing [14–17]. Introducing the photocatalytic filter as a subcomponent of the facemask is likely one of the effective ways to eradicate viruses or bacteria for anticontagion. Although the toxicity of nano-sized zinc oxide (ZnO) and titanium oxide (TiO₂) is still ambiguous and remains to be further validated [18,19], those oxides have long attracted significant attention thanks to their photocatalysis, antibiosis, low cost, and chemical stability [20–24]. Particularly, the ZnO/TiO₂ core-shell structure has been reported to exhibit superior activity in comparison with individual materials because of selective charge separation in the core-shell interface and recombination suppression of electrons and holes [25,26]. Using ALD, firstly, we sequentially deposited ZnO, TiO₂, or laminated ZnO/TiO₂ on the PP melt-blown fabric to fabricate the photocatalytic filter (Fig. 1a).

First of all, we performed diverse physical and chemical characterizations before validating the origin of the observed differences in photocatalytic performance. The thicknesses of the ZnO or TiO₂ deposited on the PP filters were estimated using FESEM images (Fig. 1b, c, 1d, and Figure S3) and were found to be around 25–30 nm. From EDX spectra of PP/Z25, PP/T25, and PP/Z25T5, clear Zn and Ti signals were detected. The mineralogical features of the PP filters were observed to remain nearly unchanged before and after ZnO or TiO₂ deposition (Fig. 2a). The x-ray diffraction (XRD) patterns of the PP/Z25 and PP/Z25T5 exhibited peaks at 14.0°, 16.7°, 18.4°, 21.0°, and 21.8°, which corresponded to the lattice planes (110), (040), (130), and (111), and (041) of monoclinic PP [27]. The intensity of the (111) plane peak was significantly reduced and its peak overlapped with the (041) plane peak. This was reported to be typical behavior of melt-blown PP fabric [27]. The PP/Z25 or PP/Z25T5 also showed typical diffraction peaks related to wurtzite ZnO (JCPDS: 043-0002) at 31.7° (100), 34.4° (002), and 36.4° (101). However, no diffraction peaks related to TiO₂ were observed. It has been reported that when the deposition temperature of TiO₂ is low (~70 °C), mostly the amorphous phase is formed [28].

Fig. 1. Structure, surface morphology, and elemental composition of the photocatalytic PP filters. (a) Schematic of the prepared photocatalytic PP filter and the structure. ZnO, TiO₂, or laminated ZnO/TiO₂ were deposited on the PP melt-blown fabric filter using ALD. (b–d) FESEM images and EDX spectra of PP/Z25, PP/T25, and PP/Z25T5, respectively. Information on sample denotations can be found in the experimental section. See the text for details.
Unlike XRD results, in the Raman spectroscopy results, it was rather difficult to clearly recognize the change in the structure of PP before and after ZnO or TiO$_2$ deposition due to the overlapped peaks (Figure S4). However, ultraviolet-visible (UV-vis) absorption spectra exhibited a minor but noteworthy difference (Fig. 2b). The absorption spectra of the PP/Z$_{25}$T$_{5}$ and the PP/Z$_{25}$ showed a much stronger absorption in the region of 200 and 400 nm compared to the PP/T$_{25}$ (Fig. 2b). This result implied that the ZnO layer likely plays a critical role in the enhancement in absorption. The bandgap of PP/Z$_{25}$ and PP/T$_{25}$ (Figure S5a, S5b) was also estimated by Tauc plot and fitting of the linear region (Tauc equation: $(\alpha h\nu)^{1/n} = A(h\nu - E_g)$, where $\alpha$ is the optical absorption coefficient proportional to the absorbance, $\nu$ is Planck's constant, $n$ is the photon's frequency, $A$ is a constant and $n$ is a constant equal to 1/2 for direct bandgap semiconductor (e.g. ZnO) and 2 for indirect semiconductor (e.g. TiO$_2$), respectively). It was found that the PP/T$_{25}$ has a wider bandgap than the PP/Z$_{25}$. The wide bandgap semiconductor is known to only absorb ultraviolet light ($\lambda < 380$ nm) and significantly limit photocatalytic efficiencies. In contrast, the narrow bandgap semiconductor leads to a wider spectral response range to solar illumination. Therefore, it could use a wider spectral range more efficiently [29–31]. This means that the heterostructure of the PP/Z$_{25}$T$_{5}$ may be able to benefit from the extended spectral range and exhibit higher photocatalytic performance as compared to the PP/T$_{25}$ and the PP/Z$_{25}$ [32,33].

The photoluminescence (PL) spectra of the PP/T$_{25}$, PP/Z$_{25}$, and PP/Z$_{25}$T$_{5}$ were also measured in the wavelength range of 350–600 nm (Fig. 2c). The lower PL intensity means a lower recombination rate of electron-hole pairs and vice versa. Hence, the decrease in the intensity of the PL spectra indicated that the recombination of electron-hole pairs is reduced and photo-induced electrons are transferred more effectively. This recombination suppression of electrons and holes is well known to be one of the key factors to influence the photocatalytic efficiency of the PP filters [34], together with the surface wettability [32]. To validate the PP filters’ wettability, we measured contact angles of the four different filters (raw PP, PP/T$_{25}$, PP/Z$_{25}$, and PP/Z$_{25}$T$_{5}$). The measured angles were $\sim 134^\circ$, $\sim 126^\circ$, $0^\circ$, and $0^\circ$, respectively (Fig. 2d), which indicated the PP/Z$_{25}$ and PP/Z$_{25}$T$_{5}$ are nearly superhydrophilic. Considering that ZnO film deposited on Si wafer showed hydrophilic nature (Figure S6), the wetting transition from the Cassie–Baxter state [35] to the Wenzel state [36] is thought to occur on the fibrous PP/Z$_{25}$, and PP/Z$_{25}$T$_{5}$ likely because of little change in surface morphology [37].

To examine the change in the mechanical properties of the PP filter before and after ZnO or TiO$_2$ coating, we performed uniaxial tensile tests. As can be seen in Fig. 2e, both ultimate and yield strengths of the PP/Z$_{25}$T$_{5}$, PP/T$_{25}$, and PP/Z$_{25}$T$_{5}$ increased as compared to the raw PP. However, the ductilities were observed to decrease and the increase in the stiffness was less significant. In our previous work, we observed a similar phenomenon [38]. Namely, it was observed that, after ZnO ALD on PTFE (polytetrafluoroethylene), the ductility of PTFE notably decreased, although the ultimate strength increased. We found that the molecular structure of the PTFE significantly changed by the infiltrated Zn, thereby leading to the drastic change in the mechanical properties of the PTFE. We believe that the current PP filters likely experienced an analogous change in the molecular structure after ZnO or TiO$_2$ ALD and thus the PP filters exhibited an increase in strength and a decrease in ductility.
3.2. Antibacterial performance

To evaluate the photocatalytic effect of the prepared PP filters, antibacterial tests were carried out. In detail, we monitored the survival rate of *E. coli* (rod-shaped Gram-negative bacterium *Escherichia coli*) under various illumination conditions. As shown in Fig. 3a, the survival rates of the *E. coli* in the pure PP filter were constant or slightly increased due to the natural replication of the

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**Fig. 3.** (a) Time-dependent *E. coli* survival ratio curves of various PP filters (PP, PP/Z25, PP/T25, and PP/Z25T5) with/without UV illumination. (b–d) Time-dependent *E. coli* survival ratio curves of different filters (b) ZnO@PP, (c) TiO₂@PP, and (d) ZnO/TiO₂@PP with different thicknesses of photocatalytic metal oxides. (e) The mass change of PP/Z25T5 (thickness ~200 μm, area ~1.54 cm²) after each test/washing procedure. (f) *E. coli* survival ratio curves of the PP/Z25T5 filter after repeated uses (15 min each time, five times in total).
E. coli in a dark environment [28]. In contrast, under UV, the survival rate notably decreased. The PP filter coated with both TiO2 and ZnO (PP/Z25T5) exhibited the lowest survival rate as compared to the filters coated by ZnO (PP/Z25) or TiO2 (PP/T25) only, likely due to the recombination suppression of electrons and holes of ZnO/TiO2 bilayer (Figure S7). By PP/Z25T5, more than 90% of E. coli was rapidly destroyed within 5 min. Moreover, the PP/Z25 showed slightly better performance than the PP/T25 in the presence of UV. It is thought that the E. coli solution thoroughly filled the interbrillar spacing of the PP/Z25 and PP/Z25T5 filters thanks to the wetting transition, thereby maximizing the contact area between the E. coli solution and the filter, and eventually leading to an increase in the photocatalytic efficiency. It was also observed that the efficiency is pronouncedly varied with the thickness of ZnO or TiO2 deposited on the PP filter (Fig. 3b-d). Although the PP filter with thick ZnO generally showed better efficiency than that with thin ZnO, the filter with thick ZnO was found to exhibit deteriorated efficiency (Fig. 3b). The filter with the TiO2 layer and laminated ZnO/TiO2 bilayer also showed a similar thickness dependency (Fig. 3c and d). To examine the washability and reusability of the PP filter with the ZnO/TiO2 layer, firstly, we thoroughly cleaned the filter with deionized water and dried it at 60°C for 1 h after each antibacterial test, then we reused that filter for the next tests. Because only the deionized water without any detergents was used for washing, the loss of TiO2 and ZnO is believed to be negligible. As shown in Fig. 3e, the loss of our photocatalyst was only ~0.0225 mg after the fifth run. This result indicates that the loss of TiO2 and ZnO on the filter is less significant. In addition, unlike physical vapor deposition and other coating techniques, ALD coating induces covalent bonds between the substrate and the deposited material, thereby offering good adhesion. It means that the TiO2 or ZnO layer could be well preserved unless harsh mechanical deformation is applied. As demonstrated in Fig. 3f, even after the fifth successive recycling test, the filter still exhibited excellent photocatalytic activity. As compared to other filters reported in recent studies (Table S2), the overall photocatalytic performance of our filter was highly comparable.

The adsorption of viruses or bacteria onto the surface of metal-oxide photocatalyst has been reported to be the key to the antiviral and antibacterial effect [39–41]. In our ex-situ experiments, the medium (e.g. water in the E. coli experiment) played an important role in adsorption enhancement. In case the photocatalytic filter is inserted into the real mask and used in daily conditions, the medium will be air. Since air is also a good medium for the enhancement of the adsorption of viruses or bacteria onto the surface of the photocatalytic filter, our photocatalytic filter could also function well [42]. The reactive oxygen species (ROS, such as $\cdot$OH, $\cdot$O2, $\cdot$$\cdot$$\cdot$$\cdot$O2, and $\cdot$$\cdot$$\cdot$$\cdot$OOH) are also known to play an essential role in semiconductor photocatalysis [24,43]. However, how the semiconductor affects ROS generation remains to be answered [44]. Although the key reason why these filters showed a noteworthy difference in the photocatalytic efficiency is still less apparent, these differences are thought to be largely attributed to the surface wettability and the optical characteristics of the filters before and after ZnO or TiO2 coating.

3.3. A prototype of an always-on antibacterial facemask

Because most commercial facemasks do not conform to the contour of our faces, they do not provide a snug fit when we talk,
yawn, or smile in daily life. The mask should fit snugly on our faces to prevent viruses or bacteria from entering and leaving as we breathe in and out. The snug fit can be achieved when both the photocatalytic filter and UV-LEDs layer are turned into the 3D shape of our face. Inspired by the papercraft kirigami, we put a pattern of the cuts in those two layers to improve their stretchability and allowed them to morph into 3D shapes (Figure S8). Fig. 4, and Movie S1 show the experimental realization of our photocatalytic antibacterial face-mask assembled with 24 mini UV-LEDs. The mask was designed so that the photocatalytic layer and UV-LEDs layer can be readily replaced whenever necessary. Thanks to the cutting pattern, the mask fit above the nose and below the chin snugly and revealed operational stability even under external loading like bending and twisting. The heat generated by the long operation time was also insignificant. UV-LEDs with other wavelength ranges can be, of course, directly employed depending on specific purposes. It is believed that this always-on photocatalytic facemask could function well at any time and anywhere both during the day and at night.

4. Conclusion

In summary, we demonstrated a new design of a reusable photocatalytic antibacterial facemask decorated with a mini UV-LED array. That mask comprised an exchangeable polypropylene filter layer coated with the photocatalytically laminated ZnO/TiO2 bilayer and a separate UV-LEDs layer to supply UV whenever necessary. Thanks to the kirigami pattern of those layers, breathability, flexibility, as well as a snug fit, are believed to be improved. We think that the UV-LED array could allow the masks to be effectively disinfected when the need arises, regardless of where and when they are used. We found that the core-shell heterostructure consisting of ZnO and TiO2 induces the recombination suppression of electrons and holes. By virtue of the bilayer structure, the photo-induced electrons are thought to be transferred more effectively, thereby increasing the photocatalytic efficiency. Besides, the PP filters underwent the wettability transition from hydrophobic to superhydrophilic surface after ZnO and ZnO/TiO2 coating, which likely led to a further increase in the photocatalytic efficiency. Although further in-depth studies are still needed and there is a long way to go, we expect our custom facemask to be considered in various fields.

CRediT authorship contribution statement

Uyen Nhat Trieu Nguyen: Conceptualization, Material synthesis, Characterizations, Data curation, Methodology, Formal analysis, Writing draft. Khai Hoang Do: Material characterizations, Manuscript review. Bongkyun Bang: Analysis, Manuscript review. Kyung Shik Kim: Mechanical testing and analysis, Manuscript review. Jae-Hyun Kim: Conceptualization, Funding acquisition, Project administration, Manuscript review. Seung-Mo Lee: Conceptualization, Visualization, Supervision, Funding acquisition, Project administration, Writing – review & editing. Acknowledgments.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at [https://doi.org/10.1016/j.mtsust.2022.100117].

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