



Article Photoaging Characteristics of Disposable Masks under UV Irradiation

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Abstract: The global outbreak of Corona Virus Disease 2019 (COVID-19) has led to an extreme increase in the use of disposable masks. If the used disposable masks are not appropriately disposed of, they will enter the natural environment and lead to environmental pollution. In order to understand the impacts of disposable masks after being disposed of into the natural environment, aging experiments with simulated natural conditions were performed on the outer, inner, and middle layers of the masks to verify the aging characteristics of disposable masks. We analyzed the mechanical behavior, surface morphology, and Fourier Transform Infrared Spectroscopy (FT-IR) spectra of disposable masks treated with different levels of UV irradiation to understand the possible changes in the masks under UV. Results showed that the elongation at break, tensile strength, and maximum force of all three polypropylene (PP) mask layers decreased after UV irradiation, indicating chemical bond breakage. In the process of photoaging, each layer of the disposable masks showed a different degree of microscopic surface changes after UV irradiation, and these changes gradually intensified with the extension of UV exposure time. FT-IR results showed that functional groups, such as hydroxyl and carbonyl groups increased in each layer after UV irradiation. The results of this study support that, although the different layers of the disposable masks are all made of PP, they age differently in the environment. With the ever-increasing number of disposable masks in the environment, we need to further study the aging and degradation of disposable masks to better understand their potential impacts on the environment in the future.

Keywords: photoaging; disposable masks; polypropylene; mechanical performance; scanning electron microscope; FT-IR spectra

1. Introduction

Disposable masks can act as physical barriers to prevent the entry of mucosal droplets into the nose and mouth [1]. They also limit the exhalation of potentially contaminated respiratory system droplets and aerosols emitted from the wearer to the surrounding environment, which can reach an average amount of 1000 respiratory system droplets per second when talking [2]. A common disposable mask is usually composed of three layers. The outer layer is hydrophobic, hence, it repels aerosols and water droplets from the outer environment [3]. The middle layer is a very fine and electrostatic meltblown nonwoven fabric that provides the function of filtration. It filtrates air particles and prevents particles of specific dimensions from entering either side of the face mask [4]. The main material of the meltblown nonwoven fabric is polypropylene (PP) [5]. PP is in high demand due to its nonabsorbent properties and the ability to repel humidity with good mechanical properties [6]. It is widely used in the production of disposable masks. Other fibers, such as polyester rayon, glass, and cellulose are also utilized. However, these fibers are less efficient than PP [7]. The inner layer is more hydrophilic than the outer and middle layers, and can absorb moisture and aerosols from the user. Compared with normal homemade



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). masks, certified disposable masks exhibit a high efficiency against influenza viral loads and can effectively limit the spread of the virus [8,9]. Because of this, since the outbreak of COVID-19, disposable masks have been widely used to fight against the virus. Recent studies estimated that an astounding 129 billion face masks are being used globally every month (three million/min) and most of these are disposable face masks made from plastic microfibers [10]. This puts disposable masks on a similar scale as plastic bottles, which is estimated to be 43 billion per month. However, contrary to plastic bottles, ~25% of which is recycled, used masks are not readily recyclable due to potential medical hazards. Therefore, they are more likely to be disposed of as solid waste [11]. If a large number of waste masks enter the environment, they will cause environmental pollution because they can persist in the environment for a long time.

With regards to the discarded masks, a great concern is that when breaking down in the environment, the mask may break down more easily and more quickly than bulk plastics, such as plastic bags [12]. They may also release more microplastics because they are made of microplastic fibers. Microplastics are ubiquitous in the environment and have been reported in aquatic habitats worldwide from the poles [13] to the equator [14]. Because of their hydrophobic surface, microplastics can adsorb and concentrate hydrophobic organic contaminants (HOCs) such as polycyclic aromatic hydrocarbons (PAHs), organochlorine pesticides, and polychlorinated biphenyls (PCBs) [15,16] to a high degree. They also accumulate heavy metals such as cadmium, zinc, nickel, and lead [17,18]. Due to the small particle size, microplastics, together with the adsorbed pollutants, can be taken up by zooplankton, benthic organisms, and fish in the marine environment, affecting the reproduction and growth of these living organisms. Therefore, microplastics have a wide impact on the marine ecological environment [19]. In addition, in the production of plastics, many additives are incorporated. These additives will be released during the process of degradation and could enter the aquatic environment through infiltration, causing environmental pollution. Organisms exposed to the contaminated environment will face toxic effects on their physiological metabolisms, thus endangering their health, and even leading to death.

To investigate the aging characteristics of disposable masks exposed to the natural environment, photoaging experiments were performed on disposable masks. As aging under natural conditions is subject to temperature, humidity, air pressure, and the effects of light intensity, it is difficult to simulate natural aging conditions. In addition, the aging cycle under natural conditions is long. One experimental cycle could extend from a month to a year or even longer. To deal with this problem, ultraviolet light–aging instruments were used to simulate the aging process of disposable masks under natural conditions. Compared with natural aging experiments, simulation experiments provide a shorter experimental period, controllable experimental conditions, and a stable experimental environment, so that we can better understand the aging behaviors of the samples. UV photoaging experiments were performed on each of the three layers of disposable masks to evaluate the mechanical properties, surface morphology, and composition changes. The results would be helpful to better understand the aging of all layers of disposable masks and to provide basic data support to study the possible hazards of discarded disposable masks in the environment.

2. Materials and Methods

2.1. Sampling

In this experiment, we selected a brand of disposable masks that meets the YY 0969-2013 standards on the market as our experimental materials. Clean disposable masks were cut into thin strips of 5 ± 0.2 mm wide and 150 ± 0.2 mm long along the length of the masks. Masks were observed under a stereo–microscope (Nikon Inc., Tokyo, Japan). The average thicknesses of the outer, middle, and inner layers were separately measured with an electronic digital display micrometer (Lujiang Tools Ltd., Ningbo, China). Each thickness test was performed six times (N = 6). During the cutting process, a stainless–steel

ruler and a scalpel were used to ensure that the samples were cut as uniformly as possible. After each cut, samples were measured manually, strips that did not fit the size set for our experiment were not chosen. The cutting and measuring processes were performed in a clean environment to prevent sample contamination. After the above process, samples of each layer were separately stored in light–resistant sample bags for the following tests.

2.2. UV Photoaging Procedure

This photoaging experiment on disposable masks was performed in a photoreactor (Shanghai Bilang Instrument Manufacturing Ltd., Shanghai, China). In the photoreactor, mercury light was used to simulate a natural light source, and the mercury lamp power was 1000 W, with a light intensity of 160 W/m^2 . The mercury lamp was surrounded by high-temperature-resistant hollow quartz and with cooling water circulation to maintain a constant experimental temperature to prevent an impact from the high temperature. In addition, the photoreactor had an exhaust fan with continuous ventilation in the experimental environment. The average temperature in the photoreactor was 20 °C, and the mercury lamp was surrounded by an annular iron frame to fix the experimental material with a distance of 10 cm away from the light power. The disposable mask samples were fixed vertically to the iron frame, parallel to the mercury lamp. There was a distance separating the strips of each layer to ensure that the samples did not overlap with each other. In this way, the samples of each layer could receive enough irradiation from the lamp. At least three samples of each layer were taken every 4 h to measure the mechanical performance and FT-IR spectra. The experiment was carried out for 40 h altogether, but samples exposed into 40 h of irradiation time was fail to test mechanical performance, therefore, we only used data up to 36 h.

2.3. Mechanical Strength Test

The mechanical performance of the testing material was measured on an MTS criterion electronic, universal–material test machine (MTS System (China) Co., Ltd., Shenzhen, China) using a maximum load limit of 2000 N. Tensile testing speed was 30 mm/min, and the original gauge length was 15 mm. Each layer's elongation at break, tensile strength, and maximum force were recorded to evaluate the mechanical performance of the material. The tests were conducted in an air–conditioned environment at 20 °C and a relative humidity of 60%. Each test was conducted at least six times for statistical analysis and the results were presented as "average \pm std".

2.4. Morphological Analysis

The experimental materials collected at 0 h, 20 h, and 36 h UV irradiation were selected to check the morphological changes before and after UV treatment with a scanning electron microscope (Hitachi Inc., Tokyo, Japan). Voltage was set at 10 kV.

2.5. Fourier Transform Infrared Spectroscopy (FT-IR)

The FT-IR spectra of the disposable mask samples were acquired using Fourier transform infrared spectroscopy (Perkin Elmer Inc., Llantrisant, UK) in attenuated total reflection (ATR) mode, with a spectral scanning range of 4000–650 cm⁻¹ and a resolution of 1 cm⁻¹. The carbonyl index (CI) was calculated based on FT-IR spectra between the absorbance of the carbonyl (C=O) peak from 1760 to 1680 cm⁻¹ and that of the methylene (CH₂) peak from 1500 to 1420 cm⁻¹ [20,21]. Six FT-IR scans were performed on each sample and the results were expressed as "average \pm std".

3. Results and Discussion

3.1. Morphological Features of Disposable Masks under Light Microscope

As shown in Figure 1, a disposable mask consists of three layers: outer layer, middle layer, and inner layer. These three layers are all made of PP. The outer layer is blue, and the inner layer and middle layer are both white. The fiber density of the middle layer is

greater than the outer and inner layers. The shape of the disposable masks used in this experiment was a sheet–like structure composed of many fibers with gaps between fibers, and the average thickness of the outer, middle, and inner layers was 0.126 ± 0.015 mm, 0.089 ± 0.011 mm, and 0.093 ± 0.011 mm, respectively. The diameter of the polypropylene fibers in the outer layer was equal to the inner layer and greater than the middle layer.



Figure 1. Representative images of the outer layer (**A**), inner layer (**B**), middle layer (**C**), and a cross section of all three layers (**D**) of a disposable mask under a light microscope.

3.2. Changes in the Mechanical Strength of the Masks during UV Degradation

UV irradiation significantly changed the mechanical performance of each layer of the mask. As shown in Figure 2A, the elongation at break of each layer of the disposable masks decreased with increasing UV irradiation time. The elongation at break of the outer, middle, and inner layers only retained 52%, 5%, and 17% of the initial values after 36 h of irradiation. In Figure 2B,C, there was a slight rise in tensile strength and maximum force of the outer and inner layers within 8 h. After that, the tensile strength and maximum force started to decrease. During the later stage of the experiment, the tensile strength and maximum force increased slightly. However, the middle layer did not follow the same trend. The middle layer almost maintained its mechanical properties within the first 16 h of irradiation and then started to show a sharp decrease. At the end of the experiment, the maximum force remained 86%, 26%, and 46%, and the tensile strength remained 82%, 50%, and 36% for the outer, middle, and inner layers, respectively. The reason for the decrease in elongation at break is that the energy of the main chemical bonds in the polymer just corresponded with the wavelength energy in the ultraviolet light, so the polymer material could absorb the corresponding ultraviolet light, resulting in chemical bond fracture [22], thus leading to the decrease in elongation at break. PP belongs to a semicrystalline polymer [23]. The amorphous region of PP breaks first after UV irradiation, leaving the entangled molecular chains released and rearranged [24]. Under UV irradiation, the PP crystallinity tended to increase first and then decrease. The increase of PP crystallinity was the result of the competition between two different factors. On the one

hand, the chemical chains of the amorphous region of PP break and release the molecular chains, resulting in the formation of new crystals. This kind of mechanism operated mainly at the beginning of the photoaging process. Therefore, there were increases in tensile strength and a maximum force at 8 h of the outer and inner layers. On the other hand, when the formation speed of new crystals was slower than the disassemble speed of the crystals, the crystallinity of PP began to decrease [25]. This process happened in the later stage of photoaging [21]. In addition, the chemical bonds broke after UV irradiation, and at the end of the experiment, the reconnection and cross–linking of the released chemical bonds maintained the performance of tensile strength and maximum force. This is why there were slight recoveries in tensile strength and maximum force in the later stage of UV irradiation.



Figure 2. Change in mechanical performance of each layer of the masks under different UV irradiation time. (**A**) Variation of elongation at break; (**B**) tensile strength; (**C**) maximum force. (For each condition, N = 6).

Our results indicated that the mechanical properties of the masks change within a day under UV irradiation. In the natural environment, the UV irradiation strength varies with different regions and times. Therefore, the time for a mask to show mechanical change in the real environment would vary accordingly. In North China, the average radiant energy for one year is 200 W/m^2 [26], if the UV ratio is 5%, the UV radiant energy would be 0.24 KWh/m²/d. Then, the radiant energy in this experiment was 0.16 KWh/m²/h, therefore, 1.5 h in this experiment corresponded to one day in the natural environment [27]. So, if the masks were exposed to the natural environment, the outer and inner layers would start to show changes in mechanical properties within 24 days, and the middle layer would become brittle within 13 days.

3.3. Surface Morphology of Disposable Masks during UV Irradiation

The morphological features of each mask layer were observed under SEM. Before UV irradiation, as shown in Figure 3A,C, the PP fibers of the outer and inner layer had no peeling or cracks, no fracture, and no excessive distortion nor deformation of the fibers. Unlike the outer and the inner layers, the middle layer fibers showed some irregular arrangement and uneven diameter and length (Figure 3B).



Figure 3. Representative SEM images showing the changes in the surface morphology of different layers of the mask after 0 h, 20 h, and 36 h of UV irradiation. (**A**) Outer layer without UV irradiation; (**B**) middle layer without UV irradiation; (**C**) inner layer without UV irradiation; (**D**) outer layer with 20 h of UV irradiation; (**E**) middle layer with 20 h of UV irradiation; (**F**) inner layer with 20 h of UV irradiation; (**G**) outer layer with 36 h of UV irradiation; (**H**) middle layer with 36 h of UV irradiation; (**H**) middle layer with 36 h of UV irradiation; (**H**) middle layer with 36 h of UV irradiation; (**H**) middle layer with 36 h of UV irradiation; (**H**) middle layer with 36 h of UV irradiation; (**H**) middle layer with 36 h of the fibers.

As shown in Figure 3D–F, after 20 h of UV irradiation, the fibers of the outer, middle, and inner layers began to show aging characteristics, such as fiber bending and distortion. The fibers' appearance was not as smooth as the samples without UV irradiation. The surface of the fibers began to peel, and small broken pieces started to appear on the surface of the fibers. The middle–layer fibers began to break after UV irradiation and the stripped surface was similar to that of the outer and the inner layers. The stripped polypropylene material was not scattered between the fibers, but was distributed on the surface of the fibers in an attached state.

Compared with samples under 0 h and 20 h of irradiation time, all layers aged significantly after 36 h of UV irradiation, as shown in Figure 3G–I. The surface morphology transformed from smooth to coarse with significant fiber fracture and debris formation. After 36 h of irradiation, there were different degrees of microdebris formation on the surface of the fibers, and the amount of debris caused by UV irradiation increased with longer exposure times [28]. In addition, the degree of fiber stripping and fracture was

deepened after UV irradiation [29]. The areas of stripped material and the degrees of fiber fracture were more severe than those at 0 h and 20 h of UV irradiation time. In the aging process, stripping or peeling, to some extent, influenced the fibers' surface morphology, which directly increased their specific surface area, therefore, the aging PP fibers had larger specific surface areas than the original PP fibers [30].

3.4. FT-IR Analysis

After UV irradiation, specific FT-IR spectra peaks of the polypropylene material changed in specific regions, such as in the carbonyl groups (1650 cm^{-1} – 1800 cm^{-1}) and the hydroxyl groups (3250 cm^{-1} – 3600 cm^{-1}), similar to published studies [31–35].

In Figure 4A–C, the peaks around 3400 cm⁻¹ and 1600 cm⁻¹ of the outer, middle, and inner layers all increased after UV irradiation. The 3400 cm⁻¹ absorption peaks were the stretch of hydroxyl, which was caused by the oxidation of the C–H bonds of tertiary carbons. This indicates that the hydroxyl groups around 3400 cm⁻¹ were produced after UV irradiation in the outer, middle, and inner layers of the masks. After UV irradiation, the absorption around 1600 cm⁻¹ all increased in the FT-IR spectra of the three layers, which were caused by the stretch of the carbonyl groups. These were the consequences of oxidation from UV light, indicating that each layer of disposable medical masks produced carbonyl groups after UV irradiation. Thus, it could be suggested that carbonyl and hydroxyl groups were generated during the photoaging process.



Figure 4. Changes in FT-IR spectra of the outer (**A**), middle (**B**), and inner (**C**) layers after 0 h and 36 h of UV irradiation. (**A**) Comparison between 0 h and 36 h of irradiation time of the outer layer on FT-IR spectra changes; (**B**) comparison between 0 h and 36 h of irradiation time of the middle layer on FT-IR spectra changes; and (**C**) comparison between 0 h and 36 h of irradiation time of the inner layer on FT-IR spectra changes. (For each condition, N = 6).

In the process of UV photoaging, carbonyl and hydroxyl groups were generated. Due to the presence of oxygen, the tertiary carbon radicals produced by PP photoaging were easily oxidized, forming carbon and oxygen double bonds or single bonds. Because of the sufficient oxygen and water in the air, the photoaging of the material was fully conducted, generating a large number of carboxylic acid products, which corresponded to a series of products, including acids, ketones, esters, acid anhydrides, and lactones [21].

Figure 5 shows the carbonyl index of disposable masks, the carbonyl index trend of the middle and inner layers did not change significantly after 36 h of UV irradiation, while the increasing trend of the outer layer was more apparent than those of the middle and inner layers. Regardless of the layer, outer, middle, or inner, within the first 0-12 h of UV irradiation, the carbonyl index increased slightly with time. In the irradiation time of 12-20 h, the carbonyl index decreased, and the decreasing trends of the outer and inner layers were similar and different from that of the middle layer. After 36 h of UV irradiation, the carbonyl indices of each layer all increased, compared with the samples at the beginning of UV irradiation. The study above and the FT-IR spectra changes confirmed that UV irradiation caused chemical bond fractures, formed carbonyl groups, and increased the content of carbonyl groups to a certain extent. Thus, after 36 h of irradiation, the carbonyl index of each layer increased with continuous irradiation time. However, the value of the carbonyl index of the outer layer was different from the middle and inner layers, which may be related to the special weaving process involved in the manufacturing of this layer or to the different components added during the production process. However, the specific reasons still need further study.



Figure 5. Changes in the carbonyl index of the outer, middle, and inner layers of disposable masks with UV irradiation time. (1) Black line is the carbonyl index of the outer layer. (2) Red line is the carbonyl index of the middle layer. (3) Blue line is the carbonyl index of the inner layer. (For each condition, N = 6).

4. Conclusions

For each layer of a three-layered disposable mask, the elongation at break, tensile strength, and maximum force all changed dramatically after different UV irradiation durations. The maximum force and tensile strength performance of each layer were similar. Though all layers are made of PP, after ultraviolet irradiation, the middle layer aged faster than the outer and inner layers. SEM analysis indicated that samples of the different layers aged significantly after UV irradiation, as shown by the surface morphology changing from smooth to coarse. UV irradiation will strengthen fiber stripping and fracturing, leading to different degrees of microdebris formation on the surface of the fibers. The micro-debris formed is a potential source of microplastics in the environment. FT-IR spectra showed that hydroxyl and carbonyl groups were produced in each layer of the disposable masks after UV irradiation. Lactones, anhydrides, fatty acids, esters, ketones, aldehydes, and other products might have also been produced during photoaging. Furthermore, the

carbonyl index of each layer increased after 36 h of UV irradiation. Due to the long duration and broad presence of the COVID-19 pandemic, the use of masks will remain at a high level. Therefore, there is a great potential risk of microplastics formation from improperly disposed masks. The results of this research can provide data support for studies of mask photoaging and microplastics released into the environment by disposable masks.

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