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Enhancing Face Mask Protection: Graphene Oxide Modification for Improved Filtration and Electrostatic Charge

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In Loving Memory of Late Professor Doctor ""Mohamed Refaat Hussein Mahran""

Abstract
The global impact of the COVID-19 pandemic has prompted widespread use of face masks as a defense against respiratory transmission. While these masks provide some level of protection, there are limitations in terms of efficient bacteria filtration and the electrostatic charge needed to repel viruses. This study aimed to enhance the performance of commercially available face masks by modifying them with graphene oxide. A low-cost heat vapor deposition technique was employed to coat the textile of the face mask with graphene oxide. The morphology of the synthesized graphene oxide was examined using transmission electron microscopy, while X-ray diffraction analysis was used to assess its crystallinity. An antimicrobial test was conducted to evaluate the efficacy of the graphene oxide-treated face masks against bacteria. Results showed that the modified face mask exhibited reduced microbial growth, indicating enhanced bacteria filtration. Furthermore, the integration of graphene oxide significantly increased the electrostatic charge on the mask's surface, further enhancing its filtration capabilities. This study demonstrates the potential of graphene oxide modification in improving the protective properties of face masks, offering a promising solution to mitigate the spread of infectious diseases such as COVID-19.
Keywords: Face masks; Graphene oxide; Textile coating; COVID-19; Filtration capabilities.

1. Introduction

By 2019, COVID-19 had started spreading globally, eventually infecting billions and killing millions of people worldwide. Consequently, COVID-19 was classified as an infectious disease [1,2]. The World Health Organization (WHO) reported that the virus could quickly transfer through the mouths and noses of infected people to others in the form of respiratory droplets [3,4]. Therefore, the WHO recommended the utilization of face masks to reduce the subsequent clinical impact of increasing asymptomatic infections and to decrease the virus spread. According to US Centers for Disease Control and Prevention (CDC), wearing face masks has effectively reduced virus community spread and transmission by asymptomatic undetected carriers, who can be major drivers of transmission of COVID-19 [5,6]. Face masks are made of materials that can safeguard against breathable pathogens [7]. Face masks can be categorized as full, half, and quarter masks, where the efficiency of each depends on the mask material density [8]. The utilization of face masks without regular replacement or inappropriate washing leads to contaminating surfaces because humidity and temperature result in moisture that can produce microbial colonization.

Traditional medical mask filtration is based on an electrostatic charge applied to the mask by the meltdown process [9]. However, low-cost utilized masks have poor hydrophobicity, which encourages droplets to adhere to the mask surface [10,11]. Furthermore, the electrostatic filtration efficacy is

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reduced by contact with moisture, allowing viruses to pass through. Environmentally, the reusability of such low-cost masks is very low, and the disposal of these masks resulted in more than 250 thousand tons of waste worldwide [12]. Consequently, the demand for reusable and low-cost masks increases. Researchers have attempted to enhance the protection ability of such masks by modifying them with materials that add self-sterilization capabilities.

For a better understanding of the parameters that may affect the protective value of face masks, Li et al. [13] mentioned that masks, such as N95 or three-layer surgical masks, cannot maintain their protection effect if the mask surface is wet or hydrophilic. A pilot investigation proved that N95 masks are not water-repellent and a significant deterioration in the mask protection occurred when the masks were exposed to fluid splashes [14]. In addition, contaminated masks with infectious agents can allow microorganisms to penetrate the mask surface accompanied by droplets. Such problems encourage people to replace their face masks repeatedly; however, it is difficult to do this during specific emergency procedures, notwithstanding the cost. Moreover, the hands of people can be contaminated when replacing the face mask. Furthermore, the long-term use and the reuse of face masks because of global shortages during the COVID-19 pandemic have raised anxiety about bacterial contamination. To enhance the protection of such masks from infectious agent transmission risk, researchers recommended coating the surface of the mask with antimicrobial materials [15]. Others tried to enhance the performance of face masks by controlling the electrostatic properties of the face mask surface [16].

Many researchers tried to treat the fabrics of the face mask with different antimicrobial materials, such as titanium dioxide nanoparticles, halogen compounds, and metallic compounds, to prevent the effect of the virus [17,18]. However, the toxicity of some materials, pathogen-specific activity, and slow antimicrobial activity represented obstacles in their activities to develop the desired protection from the mask. Furthermore, the use of such materials was sometimes incompatible with the existing fabrication processes [19].

Quaternary ammonium compounds are famous antimicrobial materials [20]. However, using such compounds with face masks has many limitations because of their weak bonds with the filter surface resulting in performance degradation [21], toxicity, unscalable, inefficient, and complex surface modification through plasma treatment [22]. The development of nanotechnology has proved that it is promising to achieve large impacts in different fields, biology, electronics, water quality, physics, medicine, biomechanics, and sensors [23]. Many researchers reported the ability of nanoparticles to kill vast types of organisms, such as gram-positive and gram-negative bacteria, in which their envelope and the cellular wall had disinfectant resistance [24]. Furthermore, some nanomaterials, such as titanium oxide, could kill fungi, protozoa, viruses, and algae [25]. Some investigations showed how nanomaterials could disinfect different masks [26]. Tripathi et al. [27] proved that coating a face mask with titanium oxide nanoparticles can save self-cleaning and antibacterial characteristics from dominating infectious diseases, such as COVID-19.

On the other hand, some teams have been working on enhancing the performance of face masks by modifying the electrostatic charge on the surface of the mask fabric. Choi et al. [28] developed a high-performance electrostatic filtration mask by coating the surface of polyester microfiber with aluminum. Kim et al. [29] coated the polyacrylonitrile microfiber with a copper layer after reducing carbon monoxide condensation. Both studies showed an enhancement in filtering quality. They found that enhancing the efficiency of filters could be increased by supplying continuous voltages to the surface of the mask fabric. Consequently, other groups worked on improving the performance of materials used in face masks by changing some of the structures to generate more electrostatic charge [30–32].

Graphene has attracted the interest of many researchers owing to its outstanding mechanical, electronic, and thermal properties [33]. Graphene can be found in many applications ranging from solar cells to biomedical devices. Graphene opened new windows for developing new materials with unique properties, such as graphene oxide [34]. Graphene oxide proved its strong antimicrobial characteristics to withstand and kill many microorganisms, such as phytopathogens, biofilm-forming microorganisms, and gram-negative and -positive bacterial pathogens. The prevention mechanism through graphene oxide is conducted by chemical and physical interactions when mingled with bacterial cells [35].

This paper reports a detailed procedure for coating commercial masks with graphene oxide to enhance their protective properties. First, graphene oxide was prepared and characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD), and Fourier transform infrared (FTIR) spectroscopy. Second, a convenient approach was used for coating the mask was applied. Third, XRD of the original and coated masks was performed to study the effects of graphene oxide on the crystallinity of the mask material. Fourth, an antimicrobial test was conducted to investigate the effects of graphene oxide on the performance of the face mask. The effects of the graphene oxide on the electrostatic charge generated on the surface of the face mask were evaluated.

2. Materials and Methods

2.1. Graphene Oxide Preparation

Graphite powder (purity 99.9), sodium nitrate ($NaNO_3$), hydrogen peroxide (30%, H_2O_2), hydrochloric acid (37%, HCl), methanol (CH_3OH), ethanol (C_2H_5OH) were purchased from Sigma-Aldrich, UK and El-Nasr Company, Egypt. Graphene oxide was prepared using the Hummers method. Graphite was mixed with concentrated sulfuric acid (98%, H_2SO_4), and a $NaNO_3$ catalyst in an ice bath. Potassium permanganate ($KMnO_4$) was added gradually to the mixture at 35 °C to achieve the graphite oxide. The mixture was left to stand for 24 hrs. until it exhibited a sticky dough-like consistency with a brownish-ray color. Distilled water was then added slowly at 90 °C. The material was left to cool before adding H_2O_2 to remove the manganese. The extracted material was washed with distilled water before applying the separation process by centrifuging the solution. The previous process was repeated until the pH of the solution reached 7. Finally, filtration and drying were conducted to obtain graphene oxide. The previous production process was conducted according to antecedent investigations [36–38].

2.2. Nano-mask Preparation

The 0.01% graphene oxide was mixed with acetic acid with the pH adjusted to 3 in the presence of 1% glycerol to delay acetic acid vaporization. Subsequently, 100 ml of the mixture was cast into a conical die. The conical die was inserted into a vacuum chamber with a magnetic heat stirrer. The face mask was placed in the path of the mixture stream, as shown in Fig. 1. The system was then operated under vacuum, agitation, and heat conditions. The operating temperature was set to 60 °C for five hours. The mask color was changed to light gray due to the deposited graphene oxide on its fabric. Before applying any characterization, the mask was left for two hours at room temperature.

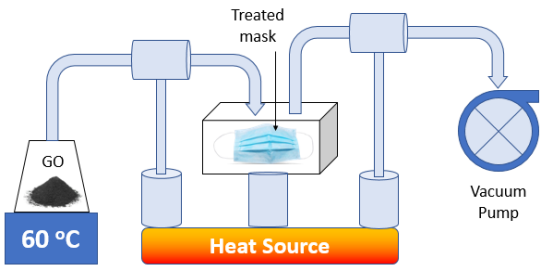

Fig. 1. Schematic for the Nano-mask preparation.

2.3. Characterization and Testing

The morphology of the synthesized graphene oxide was observed by TEM (JEOL – JEM-2100F, Tokyo, Japan). The crystallinity of the produced graphene oxide and face mask before and after treatment were examined by XRD (D8 Discover, from Bruker, Germany). The characteristic bond vibration mode of the synthesized graphene oxide was evaluated using the Fourier-transform infrared (FTIR: Vertex-70, Bruker, USA). The spectral measurements were recorded in the wavenumber range of 400–4000 cm−1. The data were processed by using OPUS 6.0 (Bruker) software, which was baseline corrected. An antibacterial test using the disc diffusion method was performed to examine the effects of graphene oxide on face mask protection against bacteria. A mix from different bacteria such as E. coli, Staphylococcus aureus and Salmonella typhimurium was used to perform antibacterial test. Screening of different samples of 4 discs were tested by disc diffusion method for 24 hours. Finally, the electrostatic charge of the face mask fabric was evaluated before and after depositing the graphene oxide on the face mask using ULTRA STABLE SURFACE DC VOLTMETER. The device measurement ranged from 0.1 V on the surface to 20 kV. The readings were usually recorded when the sensor was 25 mm away from the surface being tested.

3. Results and Discussion

Fig. 2 shows the typical TEM images of the synthesized graphene oxide, where the graphene oxide nanosheets exhibit flat flake-like shapes. The graphene oxide appears semitransparent, illustrating the instability of the material under the high-energy electron beam. In addition, the surface of the graphene oxide was rough and irregular but not crumpled, and there was no uniform size. Usually, the unwrinkled and disordered structure reveals the existence of oxygen atoms besides the high degree of exfoliation that occurred during the oxidation process [39]. Such irregularity and disorganized particle shapes could be attributed to the cracks in the structure that occurred during the oxidation process [40].

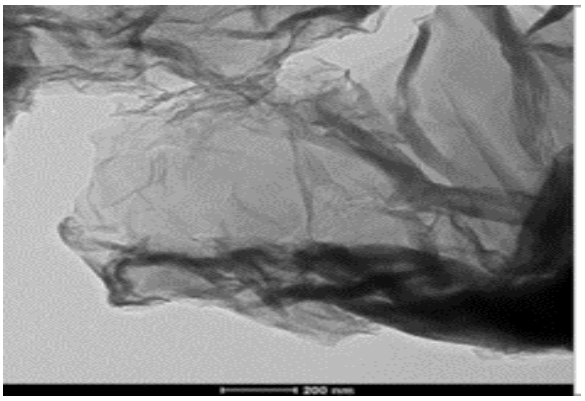

Fig. 2. TEM image of the synthesized graphene oxide.

Fig. 3 (a) presents the XRD pattern of graphene oxide, where the main peak appears at 10.1° 2θ, reflecting the interplanar distance between the graphene sheets (0.858 nm). After the heat vapor deposition of the graphene oxide on the fabric of the mask, graphene oxide was reduced, and its peak was shifted, as shown in Fig. 3 (b). The peaks for graphene oxide and the reduced graphene oxide in the current study align with Mikhaylov et al. [41] results.

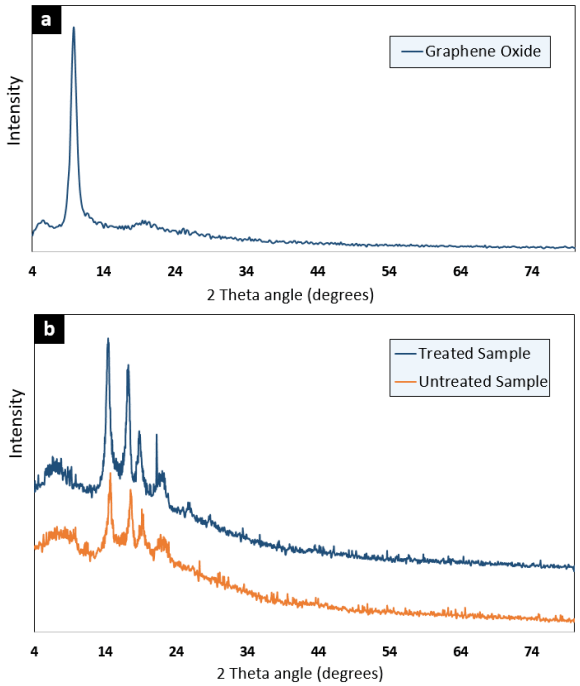

Fig. 3. (a) XRD patterns of the synthesized graphene oxide, and (b) XRD of treated and untreated face mask.

A graphene oxide sample was examined by FTIR spectroscopy to identify the characteristic bond vibrations among the graphene oxide elements, as shown in Fig. 4. The FTIR pattern of the graphene oxide accorded well with the results of previous studies [42]. Different oxygen configurations appeared in the structure of the graphene oxide with various vibration modes, such as epoxide (C-O-C), sp^2-hybridized (C=C), carboxyl (C-OH), ketonic species (C=O), and hydroxyl (C-OH) with C-OH vibrations from COOH and H_2O.

An antibacterial test was performed for three days to investigate the ability of graphene oxide to kill bacteria [43, 44]. On the first day, agar was melted in a water bath to encourage bacteria to feed on it. A layer from the agar was then taken on the inoculated agar plate, and cotton was dipped in a bacterial suspension to swab over the entire agar surface. Subsequently, it was left for 24 hours to allow the bacteria to activate and emerge on the agar surface. On the next day, the activated bacteria were collected from the surface of the solid agar and mixed in a new tube with a liquid-state agar to ensure a good distribution of bacteria in the new tube. The bacteria-agar mixture was taken in two new inoculated agar plates and placed in the freezer for 2-3 minutes to solidify the agar. Finally, an antibiotic disk dispenser was used to make four identical holes in each plate. Graphene oxide at 1% and 10% was added to the first and second plate, respectively; each plate had four holes. Subsequently, 25 µl microliters of graphene oxide were injected into the first hole of each one. The second, third, and fourth holes were injected with 50, 100, and 200 µl of graphene oxide, respectively. The two plates were then incubated at 37 °C for 24 hours.

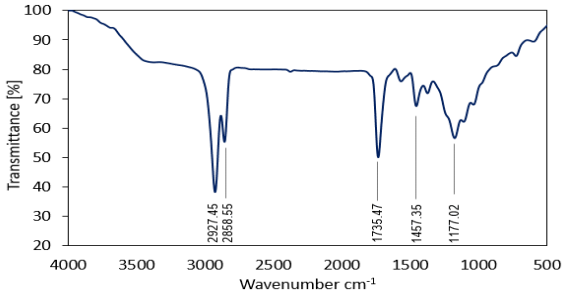

Fig. 4. FTIR spectra of the synthesized graphene oxide.

The test results were released on the third day, as shown in Fig. 5. For the first plate, where the graphene oxide concentration was 1%, the diameter of the inhibition zone was different because of a change in the quantity of graphene oxide added in each hole. The diameter of the inhibition zone was 10, 13, 21, and 34 mm after adding 25, 50, 100, and 200 µl of graphene oxide, respectively. The results illustrate the proportionality between the amount of graphene oxide added and the antibacterial efficiency of the face mask. The same performance was achieved with a graphene oxide concentration of 10%.

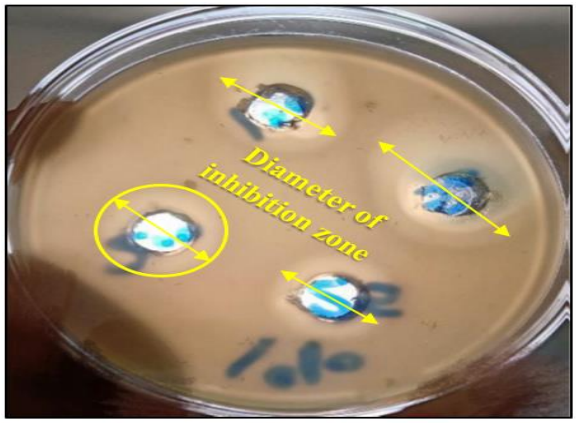

Fig. 5. Antibacterial test results for the synthesized graphene oxide with a 1% concentration.

The electrostatic charge (V) on the surface of the face mask before and after graphene oxide vapor deposition was measured using ULTRA STABLE SURFACE DC VOLTMETER, as shown in Fig. 6. The device measurement ranged from 0.1 V on the surface to 20 kV. The readings were usually recorded when the sensor was 25 mm away from the surface being tested. The electrostatic charge on the surface of the face mask was negative (−3.88 kV). After depositing the graphene oxide, the electrostatic charge increased by 0.02 kV to 3.9 kV. The increase in electrostatic charge on the surface of the face mask fabric indicates improved mask filtration [29].

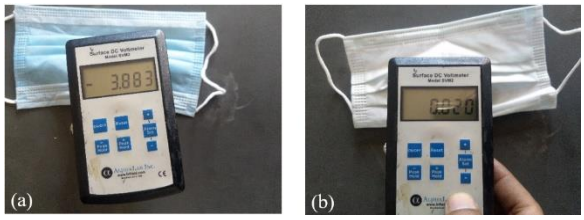

Fig. 6. ESC on the surface of the face mask (a) before and (b) after treatment

4. Conclusions

Current events have highlighted the need for face masks to protect against different viruses, especially COVID-19. The current conventional face masks work as a filtering system without any ability to deactivate bacteria and viruses. Consequently, these face masks may become microbial colonization sites for viruses or microorganisms. In the current study, a novel Nanomask was produced based on the deposition of graphene oxide on its surface. A complete process of graphene oxide production was conducted. A low-cost heat vapor deposition was used to cover the face mask fabric with graphene oxide. An antibacterial test was performed to evaluate the ability of graphene oxide to attack bacteria. The electrostatic charge on the surface of the face mask was measured before and after the coating. The graphene oxide could kill bacteria, and its efficiency increased with increasing volume fraction. Furthermore, the graphene oxide could increase the electrostatic charge on the surface of the face mask, which enhanced mask filtration.

5. Conflicts of Interest

The authors declare no conflict of interest.

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