

Fluorinated Graphene-Coated N95 Respirators for Enhanced Protection against Viral Accumulation and Transmission

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Highly contagious respiratory infection diseases such as COVID-19 can be transmitted by inhaling virus laden liquid droplets and short-range aerosols, released by an infected person. Particularly, in hospitals, spraying of the respiratory droplets containing pathogens from the conjunctiva or mucus of a susceptible person plays a key role in transferring the infectious diseases. N95 filtering respirators are a critical personal protective equipment. However, due to concerns over the virus accumulation on the N95 respirators, there is an urgent need to make the N95 respirators less contaminated. To address this critical issue, a one-step spray coating approach is developed to coat the fluorinated graphene (FG) nanosheet onto the N95 respirators. The synergistic effect of the FG nanosheet with a low surface energy and the increased surface roughness by FG on the respirator's surface makes it superhydrophobic. For respiratory droplets like saliva and mucus, the FG-coated respirators also show excellent superhydrophobicity. Furthermore, the ability against virus accumulation on the FG-coated respirators is tested by using the mucus droplets laden with SARS-CoV-2. The results show that FG-coated respirators largely increase the virus repel efficiency even under multiple contacts and effectively reduce the virus accumulation.

1. Introduction

Respiratory infection diseases such as COVID-19 are primarily transmitted through inhalation of virus-laden liquid droplets and short-range aerosols by others in close contact, particularly for a crowd in a relatively closed area, and close contact with infected persons, such as in hospitals.^[1] Virus-laden respiratory droplets with various sizes are typically released by an infected person through coughing, sneezing, speaking, singing, and breathing.^[2–4] Recent study indicates that surgical face masks can prevent respiratory droplets from entering the lungs, reducing the infection risks from the virus.^[5–7] Masks, particularly N95 filtering facepiece respirators play a critical role in controlling the transmission of respiratory infection diseases such as COVID-19.

N95 respirators can protect against particles with a size as small as hundreds or micrometers with 95% filtration efficiency.^[8] Although the virus is around

150 nm, the size of the actual respiratory droplets containing various components, including proteins, enzymes, commensal microflora, cell debris, and so on, is over 1 μm .^[9–11] With the micrometer range, N95 respirators are believed to be sufficient for personal protection against most viruses. N95 respirators consist of four layers: a polypropylene hydrophobic microfiber layer to repel respiratory droplets, the support cellulose/polyester layer, a nonwoven melt-blown polypropylene layer as a filter to hinder the transmission of small particles and pathogens, and another polypropylene hydrophobic microfiber layer to prevent internally created moisture from entering the mask filter.^[12–14]

Although N95 filtering respirators are commonly used by healthcare workers, they still have certain limitations.^[15,16] For example, the hydrophobicity of the outer layer is not sufficient to prevent the adherence of the respiratory droplets on the surface, which can make the respirator become a hotbed of microorganisms after long-term use. Moreover, it is recognized that viruses shed from respiratory droplets can accumulate on the respirator's surface.^[9–11,17] This is particularly detrimental in the intensive care unit (ICU) environment where patients can release a large amount of virus-laden liquid droplets in a short period of time and can leave significant virus contamination on the

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respirators.^[18] The accumulated virus can infect healthcare workers in two ways: 1) unintentional touching of the contaminated respirator may bring the virus into the nose, eyes, or mouth, and 2) the virus may sneak through the respirator and enter the lungs during inhaling. To protect the healthcare workers, it is essential to make the N95 respirators less contaminated.

During the COVID-19 time, various strategies have been used to improve the respirators' performance, by adding superhydrophobic, or self-cleaning functionalities to extend the lifespan of respirators.^[19–25] Superhydrophobic surfaces exhibit extreme water repellence with a water contact angle larger than 150°. Water maintaining nearly spherical shapes can easily roll off them to achieve self-cleaning. It is expected that superhydrophobic coating can repel respiratory droplets away from the respirator's surface, prevent the contamination of the respirator from pathogens, and decrease the risk of infection. Previous studies have demonstrated that the superhydrophobic surface can be achieved by reducing the surface energy through attaching low surface energy materials directly in the form of a coating and adding micro- and nano-hierarchical roughness to the surface.^[26,27]

Fluorinated materials are known to lower the surface energy and induce a water-repellent effect.^[28] Carbon nanomaterials like graphene are potential materials for superhydrophobic coating, and carbon nanomaterials also demonstrate extraordinary robustness to various environmental challenges like high pressure, mechanical abrasion, UV exposure, and resistance to dust accumulation.^[21,29–31] Besides, the spray coating of low

surface energy materials has been proven to be a facile and viable method for developing uniform superhydrophobic surfaces, while it has been seldom applied for respirators.^[19,32–35] In addition, previous studies are limited to water droplets. In practice, respiratory droplets are more complex than water. They are

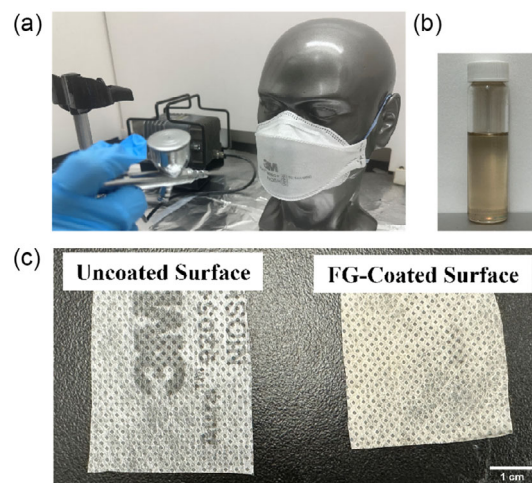


Figure 1. a) The spray-coating of FG on the N95 respirator; b) the well-dispersed FG-toluene dispersion; c) photograph of the pristine respirator's outer-layer surface (left), and the FG-coated outer-layer respirator's surface (right), respectively.

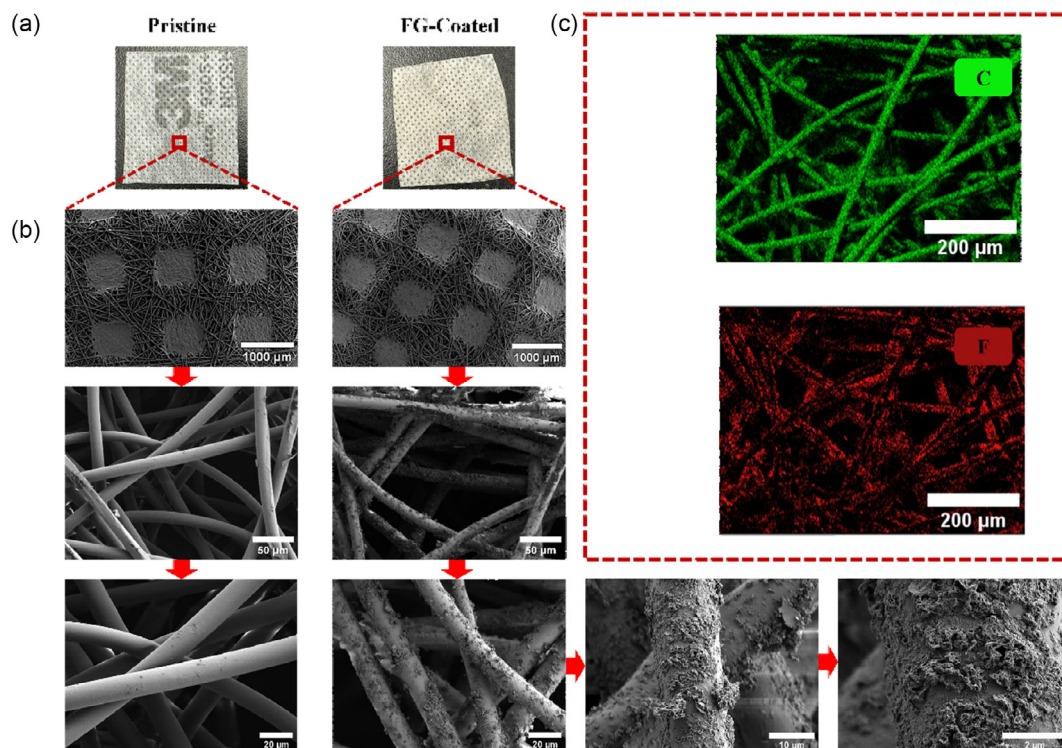


Figure 2. a) The photographs of pristine outerlayer (left) and FG coated outerlayer of respirators (right); b) the SEM images at different magnifications of the pristine (left) and FG coated (middle and right) outer layer of the N95 respirator; c) the EDS mapping images of the FG-coated outer layer of the N95 respirator.

non-Newtonian liquids. Coating can repel water droplets but may not be capable of repelling complex fluid droplets. Therefore, it is important to examine whether the respiratory droplets can easily roll off the superhydrophobic surface. Finally, many studies have not been directly tested against SARS-CoV-2 virus accumulation to demonstrate their effectiveness.

In this article, we present a simple one-step approach to spray coat a highly fluorinated graphene (FG) superhydrophobic layer on existing N95 respirators. The coated FG lowers the surface energy and forms an additional superhydrophobic nanostructured coating on the outer layer of the respirators. We demonstrate that the coated N95 respirator is capable of repelling respiratory droplets instantaneously and prevents the transmission of respiratory infection diseases like COVID-19. Moreover, we show that our highly FG superhydrophobic coating can effectively reduce the SARS-CoV-2 virus accumulation, compared to the uncoated surface. Such enhanced repellent behavior of the coated respirators significantly decreases the risks of healthcare workers and ensures their safety especially when they are exposed to high-risk environments such as in the ICU.

2. Results and Discussion

Herein, we developed a simple and convenient method to increase the hydrophobicity of the respirator's outer layer by directly spray-coating highly FG nanosheet onto commercially

available N95 respirators. Before coating, the chemical structure and crystallinity of FG were characterized using X-ray diffraction and an attenuated total reflection Fourier transform infrared spectrometer (ATR-FTIR), revealing the presence of C–F covalent bonds and a high fluorine content (Figure S1, Supporting Information). **Figure 1a** demonstrates the process of the one-step spray-coating method. To get a uniform FG coating, the FG nanosheet was first well-dispersed into the organic solvent. Four different solvents, i.e., ethanol, methanol, toluene, and benzene, were applied to disperse the FG nanosheet. As a result, the FG nanosheets were well-dispersed into the toluene with an optimal concentration of 1 mg mL^{-1} through ultrasonication, and the FG-toluene dispersion can remain stable for at least one hour (Figure 1b). Then, the well-mixed FG dispersion was spray-coated onto the outer layer of the N95 respirator by the airbrush gun. After spray-coating, the outer layer color of the coated respirator turns a little brown compared with that of the uncoated one (Figure 1c).

To analyze the difference between the surface structure of respirators before and after spray coating, we used scanning electron microscope (SEM) to characterize the surface morphology, as shown in **Figure 2a,b**. After spray-coating, the polypropylene fibers of the respirator's surface, originally with a smooth surface, are wrapped by the FG nanosheets, forming a rough surface structure composed of FG nanosheet aggregates. These aggregates are uniformly distributed on the respirator's surface by spray-coating. Meanwhile, the SEM-energy dispersive

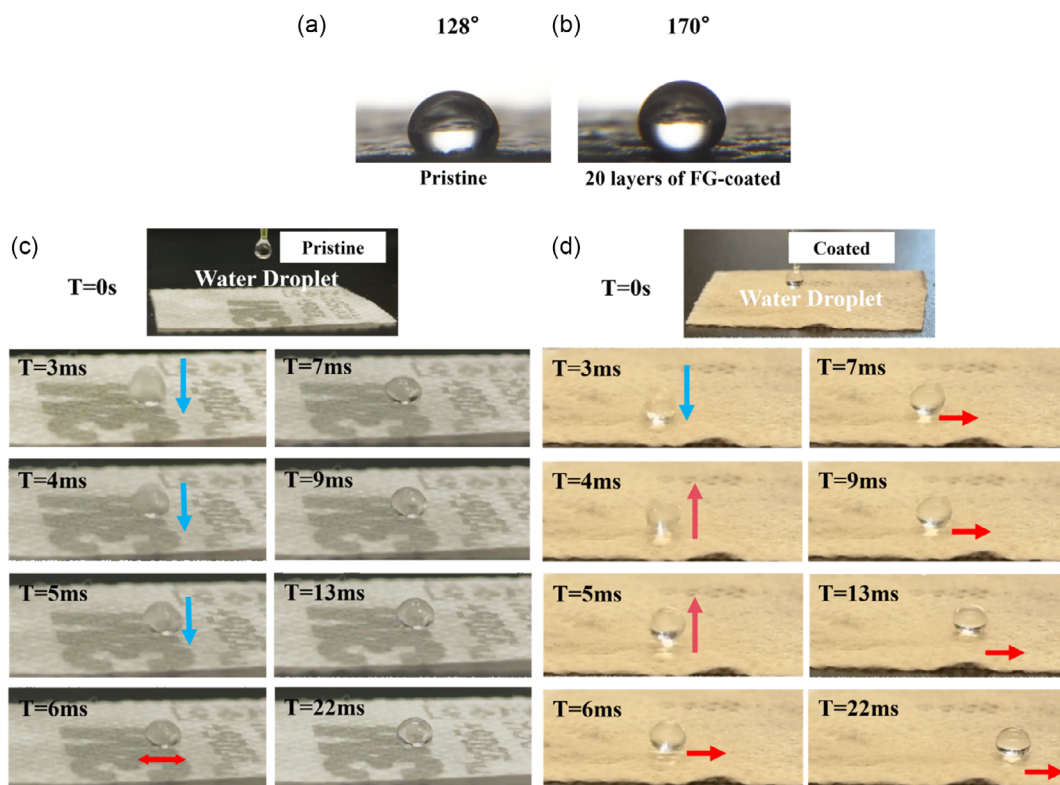


Figure 3. The water contact angles on a) the pristine respirator's surface and b) the FG-coated respirator surface. c) The snapshots of a water droplet released from the transfer pipette adhering to the pristine respirator's surface. d) The snapshots of a water droplet released from transfer pipette bouncing on the FG-coated respirator's surface.

spectroscopy (SEM-EDS) analysis was also conducted to evaluate the elemental composition of the coating and the uniformity of the coating. The carbon component is derived from the respirator's fabric. As seen from Figure 2c, the uniform distribution area of fluorine (F) on the surface of the respirator is almost the same as the distribution of original carbon (C) component of the respirator fiber, indicating the excellent uniformity of the FG coating. Carbon and oxygen are original from the respirator fabric, and the F element occupies 21.29 wt% of the coated respirator's surface. Thus, the presence of low surface energy element F in combination with the increased surface roughness by coating can increase the hydrophobicity of respirators and make them superhydrophobic. Meanwhile, the graphene potentially ensures better respiration when embedded with the N95 respirators, as graphene has a 2D structure in which sp^2 -hybridized carbons are arranged hexagonally in a honeycomb lattice.^[36] The chemical composition and the uniformity of the FG coating were further confirmed by the X-ray fluorescence spectroscopy (XRF), as shown in Figure S2, Supporting Information. As the N95

respirator consists of multiple layers of polypropylene fabrics, we also carried out the XRF tests on the respirator's different layers after coating with FG. The measured XRF spectra show that the F peak can only be noticed on the outermost layer of the respirator, suggesting that there is no FG presence inside the N95 respirator (Figure S3, Supporting Information). To be noticed, the weight of an N95 respirator is around 8 g and the amount of FG used for coating is around 0.12 g. Therefore, the weight percentage of the FG is as low as 1.5 wt%.

To evaluate the hydrophobicity of the respirator's surface, we measured the water contact angle. The pristine N95 respirator exhibits a hydrophobic property, as water droplets stay on its surface in a hemispherical shape with a contact angle of 128° (Figure 3a). After coating with FG, the hydrophobicity of the respirator surface improves significantly with a contact angle of 170° (Figure 3b). Interestingly, without coating, a water droplet (20 μ L) can adhere to the hydrophobic respirator's surface after being released from the pipette (Figure 3c). In stark contrast, with the FG coating, the water droplet begins to bounce and rolls

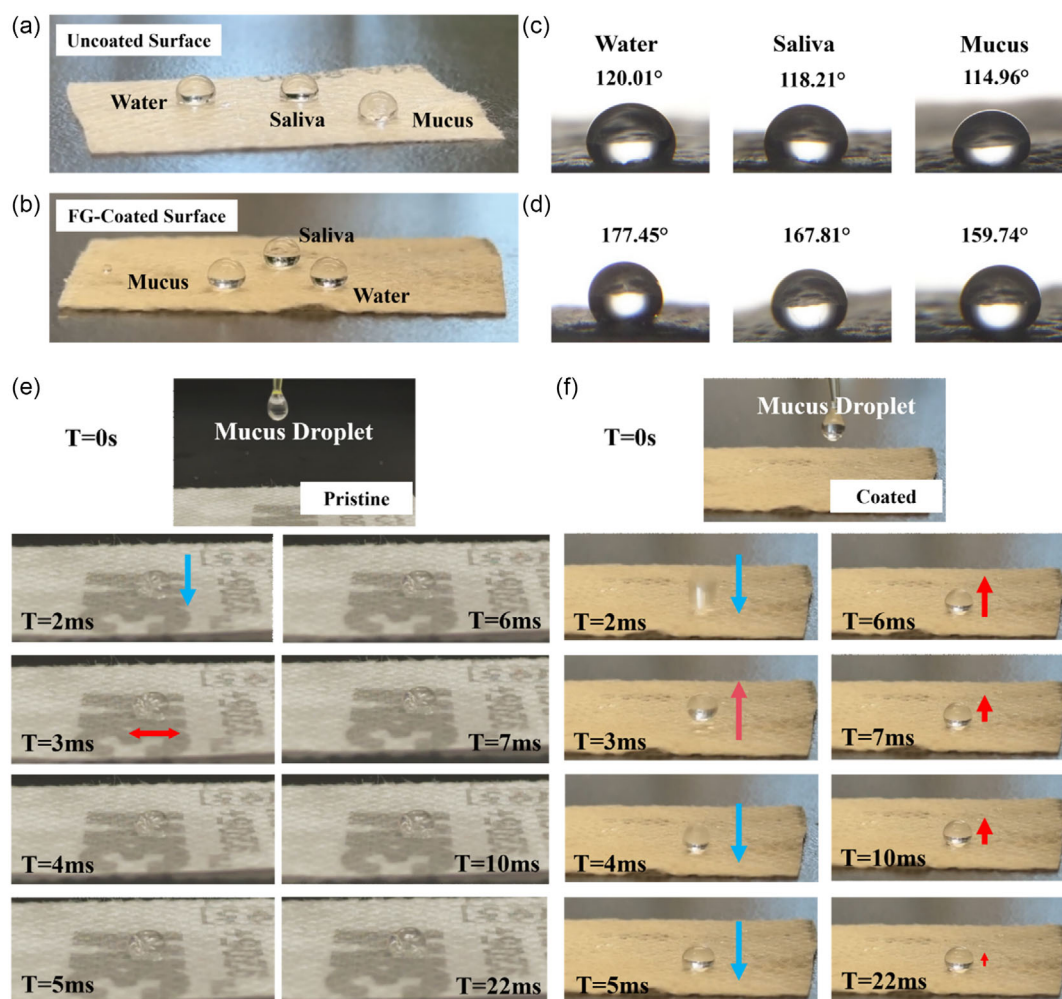


Figure 4. Droplets of mucus, saliva, and water on a) the surface of pristine respirator and b) the FG coated respirator's surface; contact angles on c) the pristine respirator's surface and d) the FG coated respirator's surface; time-lapse snapshots of a mucus droplet falling onto e) the pristine respirator's surface and f) the FG coated respirator's surface, respectively.

off the coated surface (Figure 3d), suggesting that there is a low adhesion between the water droplet and the coated respirator's surface. This enhanced superhydrophobicity is attributed to the synergistic effect between the low surface energy of the highly FG and the increased surface microstructure caused by the FG sheet coating. The wetting performance of our coating onto

face masks, along with that of other coatings reported in the literature is listed in Table S1, Supporting Information.

Because the transmission of respiratory disease viruses, including SARS-CoV-2, is through contact with body fluid, and the surface tension of body liquid is much lower than that of water, the results of water droplets are not sufficient to

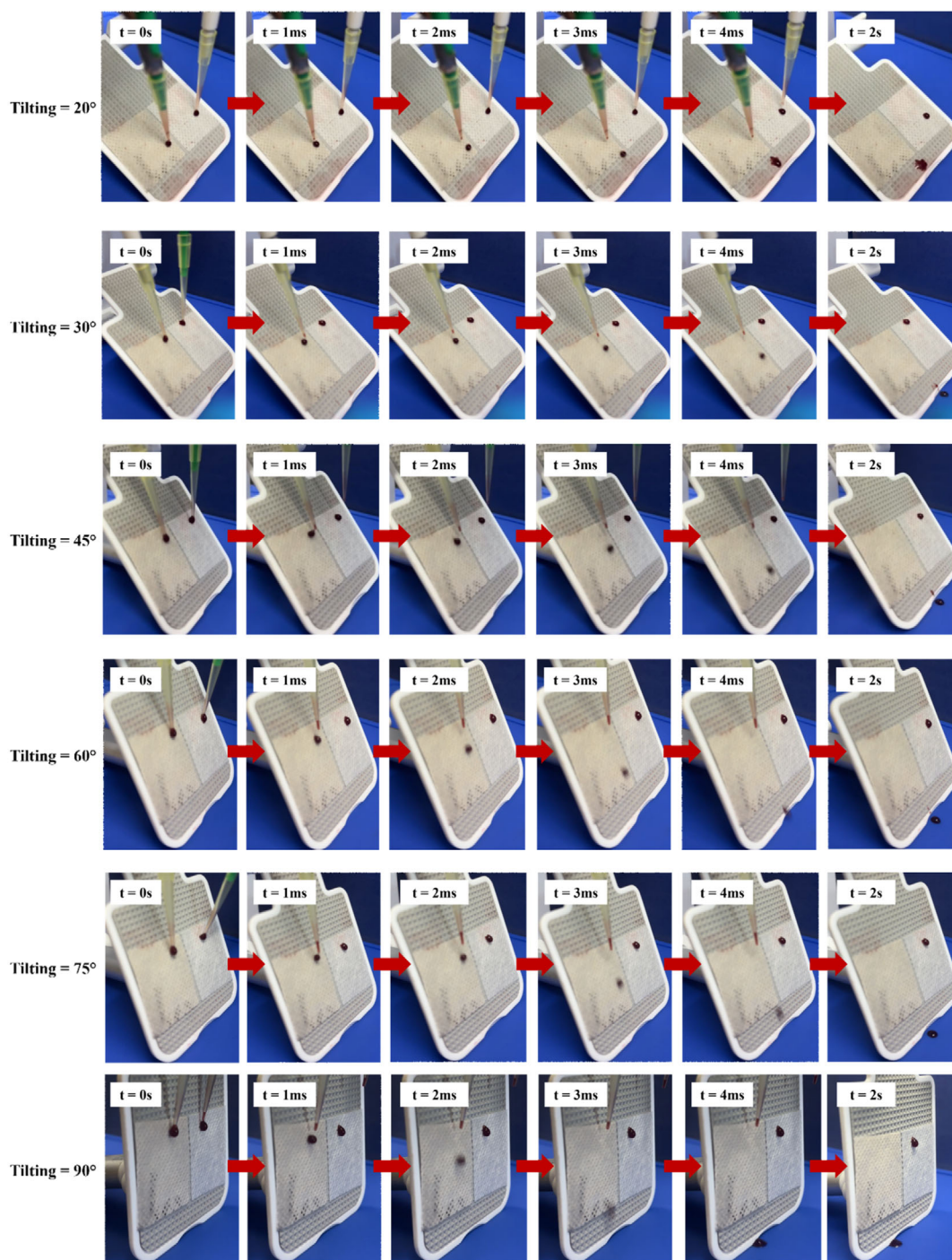


Figure 5. The rolling behavior of mucus droplets on the surface of the pristine respirator (right piece) and the FG-coated respirator's surface (left piece) at different tilting angles.

demonstrate that the superhydrophobic coating is capable of impeding virus ingress. Therefore, it is important to examine the repelling performance of the coating on droplets from body fluids. In this study, we chose saliva and mucus as two representative body fluids, since they are closely related to the transmission of respiratory diseases via coughing and sneezing.

Here, we measured the contact angles of saliva and mucus droplets on the pristine and FG-coated respirator's surfaces, shown in **Figure 4a–d**. For the pristine respirator, all three droplets, including water, saliva, and mucus droplets, adhere to the respirator's surface with a contact angle around 110° (**Figure 4c**). In contrast, the droplets over the FG-coated respirator's surface have a contact angle of over 150°. Not surprisingly, the water droplet has the highest contact angle, and the mucus droplet has the lowest (**Figure 4d**) since the surface tension of water is around 72 mN m^{−1} while the surface tension of the mucus is around 30 mN m^{−1}.^[37]

Similarly, we recorded the mucus droplet dynamics after being released from the pipette on the pristine respirator's surface and the coated respirator's surface, respectively. As shown in **Figure 4e**, when the 20 µL mucus droplet was released from the pipette on the pristine respirator surface, the droplet quickly stick to the surface (<4 ms) without any notable motion. With our coated surface, the 20 µL mucus droplet released from the pipette quickly bounces within less than 4 ms (**Figure 4f**). The saliva droplet dynamics is between the water droplet and the mucus droplet as the saliva surface tension is larger than that of mucus and smaller than that of water.

The contact angle alone is not sufficient to evaluate the surface repellency. Another important factor for surface repellence is the roll-off angle or tilting angle, defined as the angle of inclination of a surface at which a droplet begins to roll off.^[38] To further characterize the repellence of the FG-coated respirator for respiratory

droplets, we recorded the rolling behavior of the mucus droplet on the pristine respirator's surface and the coated surface at different tilting angles. As summarized in **Figure 5**, a 20 µL mucus droplet on the pristine respirator's surface (right side) does not move even when it is tilted to 90°, which is very concerning as respiratory viruses in mucus can stay on the N95 respirator. In other words, the commercial N95 respirators have a risk for virus accumulation, which leads to fomite infection. In stark contrast, the mucus droplets on the FG-coated respirator's surface roll off instantaneously at a tilting angle over 15°. With the tilting angle increased, the roll-off speed of the mucus droplet increases accordingly. Considering that N95 respirators are typically worn with a tilting angle around 45°, the FG-coated respirator can certainly remove mucus droplets effectively and prevent the virus accumulation.

Respiratory viruses are confined in the respiratory droplets for spread.^[23,39] Viruses inside the droplet may decrease the surface tension and increase the adhesion between the droplets and the respirator's surface.^[40] Even upon removal of these droplets from the respirator's surface, there may still be residual viruses on the respirator's surface and cause the transmission of the respiratory diseases. Therefore, to quantify the removal performance of the FG-coated respirator for viruses, we mixed the heat-inactivated SARS-CoV-2 with mucus droplets and deposited them on both the pristine and FG-coated respirators, respectively. After pipetting the mucus droplets up and down in a designated contact area 20 times, we measured the copy number of viruses in the collected fluid using digital reverse transcription polymerase chain reaction (RT-PCR). As shown in **Figure 6a**, more microwells exhibited fluorescence for the FG-coated respirator, indicating a higher viral load in the collected fluid and fewer viruses remained on the surface of the coated respirator. In comparison, fewer viruses were detected in the collected fluid from the

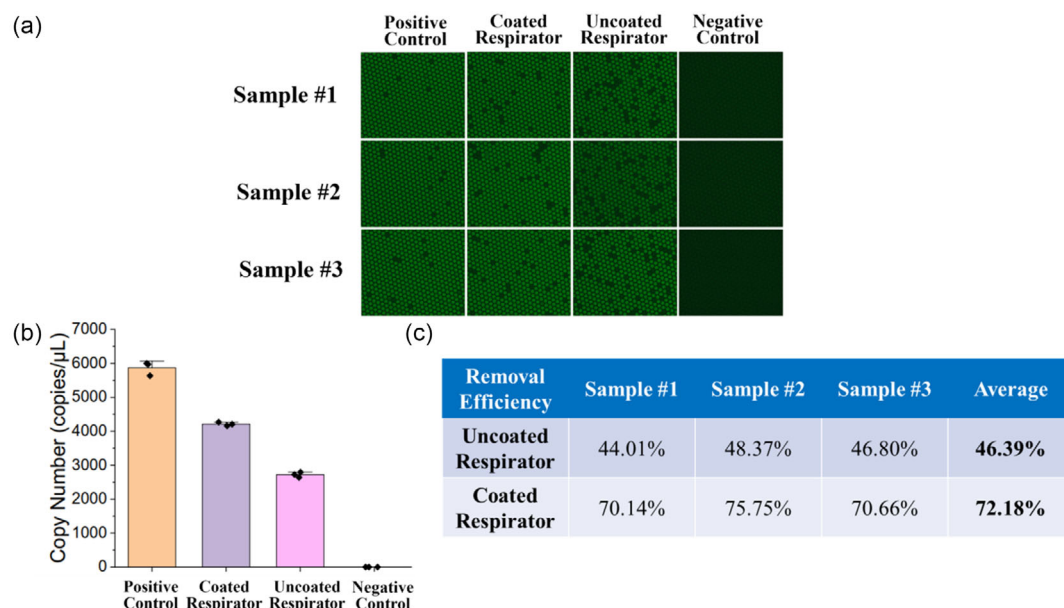


Figure 6. a) Digital RT-PCR for virus detection in the collected spiked samples. Positive control refers to the original spiked sample without processing. Negative control refers to mucus spiked with nuclease-free water; b) the collected number of SARS-CoV-2 virus after direct contact with pristine and FG-coated respirator, respectively; c) the SARS-CoV-2 virus repelling efficiency of pristine and FG-coated respirator.

pristine respirator, with more adhering to its surface. We further quantified the viral loads in the collected fluids (Figure 6b) and calculated the removal efficiency (Figure 6c), with the viral load in the original sample serving as the positive control and standard for comparison. We observed a significant increase around 55.6% in the removal efficiency (72.18%) of viruses after direct contact with FG coated respirator compared with that (46.39%) of the uncoated respirator, suggesting the favorable anti-accumulation capability of the coated respirator. Therefore, the coated respirator can effectively prevent the accumulation of SARS-CoV-2.

3. Conclusions

In summary, we developed a simple one-step approach to spray-coat highly FG on commercially available N95 respirators. The synergistic effect of the low surface energy of FG and the increased surface roughness makes the respirator's surface superhydrophobic. Even more importantly, our tests on respiratory droplets like saliva and mucus suggested that our coating can repel respiratory droplets instantaneously with less contamination when the tilting angle is over 15°. On the contrary, on the pristine N95 respirator's surface, the mucus droplets strongly adhere to the surface and do not move even when the tilted angle is 90°. Finally, our removal experiments with mucus droplets containing SARS-CoV-2 demonstrated that our coated surface can effectively reduce virus accumulation. Since the spray-coating approach is easy to scale-up and there is no need to change any existing respirator production procedures, the developed method has the immediate impact on combating infectious diseases by better protecting the healthcare workers via making the respirators less contaminated. The superhydrophobic coating is not limited to respirators. It can also be coated on other personal protection equipment, like protective shields, to prevent them from contamination to reduce the infection risk for the medical professionals when treating the infected patients. Our work can better protect healthcare workers in terms of preventing the transmission of work-related infectious diseases among workers.

Finally, we would like to comment on the impacts of our coating on human health and environment to show that there are minimum health and environmental concerns for our coating to be implemented in practice. The graphene has been reported to exhibit antibacterial, antiplatelet, and anticancer activities. As a result, graphene and graphene-related nanomaterials have been widely used in the biomedical and biological applications.^[41–45] The solvent that we used was toluene, which has an evaporation rate of 2.4 and can evaporate quickly when exposed to air. In our experiments, we placed the coated N95 respirators into an oven with a temperature of 90 °C for 30 min, then left them in the hood for over 24 h to ensure the complete removal of toluene before further usage. The N95 respirator consists of multiple layers of polypropylene nonwoven fabrics, having a filtration efficiency of 95% for 300 nm particles. The FG used here consists of nanosheet structures with a diameter ranging from 400 nm to 5 µm. Therefore, it is highly unlikely for the coated FG to penetrate inside the N95 respirator. In fact, Figure S3, Supporting Information clearly indicates that there is no FG's presence inside the N95 respirator. Finally, our FG coated N95 respirators are mainly designed to meet the requirements of healthcare

workers in hospitals, especially for those who work in the ICU. After the end of the useful life, those respirators will be treated as special medical waste. Such medical waste must be handled by specially skilled professionals and will be treated by special waste management methods, such as incineration, pyrolysis, and carbonization.

4. Experimental Section

Materials: FG was purchased from XFNANO Materials Tech Co., Ltd (Nanjing, China). Toluene, methanol, and ethanol were purchased from VWR (Atlanta, GA, USA). The N95 respirators used for coating were purchased from the 3M company (Saint Paul, MN, USA). The artificial saliva and the artificial pulmonary mucus were purchased from Biochemazone (Alberta, Canada). All chemicals were used as received without further purification.

Respirator Coating Preparation: The FG with the purity of 98% and 58% fluorine was dispersed in toluene by sonication with 24 kHz at an amplitude of 60% for 60 min. The concentration of the highly FG was optimized to get a dispersion solution with a concentration of 1 mg mL⁻¹, which exhibited both a high concentration and a uniform dispersion. Before spray-coating, the FG dispersion was vigorously stirred for 3 min on a vortex mixer to redisperse it well. The well-dispersed suspension was uniformly coated on the N95 respirators using an airbrush spray-paint gun (Mr. Hobby, PS-309) with an optimal volume speed around 0.1 mL s⁻¹. The spray-coated N95 respirators were then fully dried in a vacuum oven at 90 °C for 30 min and then left in the hood for at least 24 h before further usage.

Morphological Analysis: The surface morphology of the respirator before and after coating was characterized with a SEM (TESCAN, CLARA) at a 2 keV voltage and 30 pA current. The respirators were cut into small pieces and were sputter coated with a thin layer of gold film before the examination. Meanwhile, the surface elemental composition of the coated respirator was also analyzed by the SEM with an EDS detector.

Wetting Property Assessment: To investigate the wetting behaviors of the respirator before and after coating, the sessile droplet method was conducted to assess the droplet contact angle. The respirators were cut and attached horizontally over a glass slide using the double-slide tape. For the contact angle measurements with different fluids (water, artificial saliva, and artificial mucus), a 20 µL droplet was deposited on the tested respirator's surfaces using a micropipette at room temperature (25 °C). For each surface, the contact angle was measured from at least five different locations on the respirator's surface.

Virus Accumulation Assessment: To examine if the FG-coated respirator can effectively prevent virus accumulation, the recovery of the virus from the respirators with and without the FG coating was quantitatively measured. Firstly, we cut the coated and uncoated respirators into small slices of equal sizes, spiked artificial pulmonary mucus with heat-inactivated SARS-CoV-2 (BEI Resources, Cat# NR-52286) at a volume ratio of 6:1, and then thoroughly mixed it. We pipetted 140 µL of the spiked sample onto both the coated and uncoated respirator slices, respectively. Then we pipetted up and down 20 times and collected the liquid on the respirator as much as possible using a new pipette tip. At least three independent experiments were conducted ($n = 3$). Finally, we used the original spiked sample as the positive control and artificial pulmonary mucus spiked with nuclease-free water (NEB, Cat# B1500S) as the negative control. We extracted viral RNA of each sample using QIAamp Viral RNA Mini Kit (Qiagen, Cat# 52904) and performed digital RT-PCR to measure the copy number of each sample. Digital RT-PCR was performed using the QuantStudio 3D Digital PCR System following the manufacturer's instructions. Each RT-PCR reaction contained 1× Reliance One-Step Multiplex Supermix, 900 nM of forward primer (5'-GACCCCAAAATCAGCGAAAT-3'), 900 nM of reverse primer (5'-TCTGGTTACTGCCAGTTGAATCTG-3'), 250 nM of TaqMan probe (5'-6-FAM/ACCCCGCAT/ZEN/TACGTTTGGTGGACC/IABkFQ/-3'), and the extracted samples diluted to one-tenth of the original concentration. The RT-PCR protocol was as follows: 1) reverse

transcription at 50 °C for 10 min; 2) RT inactivation and DNA polymerase activation at 96 °C for 10 min; 3) 40 cycles of annealing/extension at 56 °C for 2 min and denaturation at 98 °C for 30 s; 4) final extension at 56 °C for 2 min; and 5) storage at 10 °C until the chip was read. Once the RT-PCR reaction was complete, the chip was read on either a QuantStudio 3D Digital PCR Instrument (Applied Biosystems) or a fluorescence microscope (Axio Observer, ZEISS).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Fengjie He: data curation (lead); formal analysis (lead); investigation (lead); writing—original draft (lead). **Jiongyu Zhang:** data curation (supporting); formal analysis (supporting); writing—review & editing (supporting). **Shengjie Zhai:** conceptualization (equal); formal analysis (supporting); methodology (equal); writing—review & editing (supporting). **Changchun Liu:** conceptualization (supporting); formal analysis (supporting); funding acquisition (supporting); methodology (supporting); project administration (supporting); writing—review & editing (supporting). **Hui Zhao:** formal analysis (lead); funding acquisition (lead); methodology (lead); project administration (lead); writing—review & editing (lead).

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

anti-virus-accumulation, N95 respirator, spray coating, superhydrophobicity

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