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Impacts of reduced graphene oxide modified air purification filters on removal of particulate matter from Ambient Air

Abd Elhakim Ezet[a,b], Ahmed H. Galmed[a], Yasser A. Attia[a*]

[a]*National Institute of Laser Enhanced Sciences (NILES), Cairo University, Giza 12613, Egypt.*

[b]*Air Quality Lab, Cairo University Center for Hazard Mitigation, Giza 12613, Egypt*

Abstract

Particulate matters (PMs) refer to tiny solid or liquid particles suspended in the air and can have a significant impact on human health, contributing to climate change and other environmental hazards. To mitigate the hazards associated with PMs, various techniques are employed for its removal from the air. This necessitates research on improving the efficiency of air purification filters and reducing harmful emissions into the air. This study focused on preparing reduced graphene oxide (r-GO) to modify filters used in air particle monitoring devices. The goal was to investigate how r-GO affects PM adsorption efficiency. Various spectroscopic techniques, including energy-dispersive X-ray spectroscopy (EDX), inductively coupled plasma (ICP), and laser-induced breakdown spectroscopy (LIBS), were used to assess the modified filters' adsorption efficiency quantitatively and qualitatively towards PMs. The results showed that r-GO-modified filters revealed higher PM adsorption efficiency than control filters. The r-GO's exceptionally large specific surface area and pore volume account for its remarkable performance. This novel approach is expected to gain significant attention and contribute to the development of improved air purification technologies.

Keywords: Air pollution control; Particulate matters; Air purification: Nanotechnology; Spectroscopy Techniques; Climate change.

1. Introduction

Air pollution poses a substantial worldwide challenge, exerting detrimental effects on human health, ecosystems, and the climate. It encompasses a range of pollutants, including gases, liquids, and solid particles, originating from both natural occurrences and human activities [1,2]. The impact of air pollutants on the environment and human health is profound. Even slight changes in the atmospheric composition can have a significant impact on climate patterns, disrupt ecosystems, and threaten the survival of many species. Examples of such impacts include acid rain, ozone depletion, and photochemical smog [3]. These pollutants, whether visible or invisible, consist of gases or particles that are not naturally occurring components of the air. While natural sources like pollen, dust storms, and forest fires contribute to the presence of these pollutants, a more pressing concern arises from emissions originating from human activities. The combustion of fossil fuels, the burning of coal, wood, and other fuels in vehicles, homes, and factories all release pollutants into the air, exacerbating the deterioration of air quality [4]. Particulate matter (PM) is a major air pollutant that is made up of tiny particles of solid or liquid matter suspended in the air. It can come from a variety of sources, including the combustion of fossil fuels, industrial emissions, dust, smoke, and fog. PM particles can range in size from a few micrometers to less than a tenth of a micrometer. PM10 is a type of PM that has a diameter of 10

*Corresponding author e-mail: yasserniles@niles.cu.edu.eg.; (Y. A. Attia).
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micrometers or less. PM10 particles are large enough to be inhaled and deposited in the lungs, where they can cause respiratory problems. PM10 is also a major contributor to regional haze, which reduces visibility and can damage vegetation [5-8]. The health impacts associated with PM10 include respiratory and cardiovascular problems. Furthermore, scientific studies have linked exposure to particle pollution to a range of health issues, such as asthma, bronchitis, lung cancer, and premature death. Young children, older individuals, and people with pre-existing heart or lung conditions are the most vulnerable to the effects of PM pollution [9-11].

Nanoparticles are tiny materials with unique properties that make them useful in a variety of fields, including engineering, electronics, and medicine. Nanoparticles have different atomic and electronic structures than larger materials, and their high surface area-to-volume ratio makes them more reactive and gives them new properties [12-15]. The size, shape, and elemental composition of nanoparticles determine their behavior and function. Many experiments have explored how these factors affect the properties and performance of nanoparticles. Nanoparticles show promise for air pollution remediation. They are highly reactive, can efficiently adsorb pollutants, and are cost-effective compared to conventional methods. Nanoparticles have been shown to adsorb heavy metals from the atmosphere, reducing heavy metal pollution. Engineered nanoparticles have also been used to effectively clean indoor air pollutants. Nanotechnology opens new possibilities for tackling air pollution, offering hope for improved air quality and human well-being [16-19].

Reduced graphene oxide (r-GO) is a wonder material with immense potential in various fields, including air purification. Its unique properties make it a promising candidate for tackling air pollution, which is a major global concern. r-GO boasts an exceptionally high surface area, allowing it to capture and adsorb a wide range of air pollutants, including particulate matter (PM), volatile organic compounds (VOCs), and even some gases. This makes it highly effective in filtering contaminated air [20,21]. The chemical and physical properties of r-GO can be readily modified through functionalization, enabling it to target specific pollutants. This versatility makes it adaptable to different air purification needs. r-GO is incredibly lightweight and can be formed into thin films, making it ideal for creating air filters with low pressure drops and energy consumption. r-GO is a robust material that can withstand harsh conditions and be regenerated for multiple use cycles, reducing waste and maintenance costs [22-24].

This study aims to investigate the potential of r-GO nanostructures in modifying filters used in air particle monitoring devices to enhance the efficiency of particle adsorption. The impacts of these nanomaterials on the filters' performance will be qualitatively and quantitatively analyzed using spectroscopic techniques, including EDX, ICP, and LIBS. The findings from this research will contribute to the development of novel approaches for air pollution remediation, particularly in relation to the removal of heavy metals from the ambient air.

2. Experimental

2.1. Synthesis of r-GO

Graphene oxide (GO) was synthesized using a modified Hummer's method. Six grams of graphite were added to a flask containing 150 mL of sulfuric acid (H_2SO_4) while stirring in an ice bath. After several minutes, the ice bath was removed, and 18 g of potassium permanganate were gradually added to the mixture. The solution turned dark green after two hours of continuous stirring at 35°C. Next, the conical flask was placed in boiling water, and 300 mL of water was slowly added to the reaction mixture. The temperature was maintained for an additional 30 minutes. One liter of distilled water was added to stop the reaction, and the flask was cooled in an ice bath. To convert the remaining permanganate to soluble manganese ions, 20 mL of 30% hydrogen peroxide (H_2O_2) was added. The resulting precipitate was centrifuged, washed repeatedly with 1 M hydrochloric acid (HCl) and distilled water, and then vacuum dried for 24 hours at 60°C. The filtrate was heated at 60°C for 4 hours to produce dry GO powder. To obtain reduced graphene oxide (rGO) from GO, 100 mg of the dry GO powder was placed in an empty beaker. The beaker was then placed on a hot plate set at 350°C for 10 minutes, with an aluminum foil cover with several perforated holes placed over it. The resultingblack powder was

r-GO, which was carefully removed from the beaker [25, 26].

2.2. *Preparation of Nano-modified filters*

Microfiber filters made of pure quartz (SiO_2) are ideal for PM10 testing and air sampling in environments with acidic gases, stacks, flues, and aerosols, especially at temperatures up to 500°C. These filters have minimal sulfate and nitrate artifacts (formed from SO_2 and NO_2) due to their low alkaline earth metal content. The filters, labeled QM-A and numbered according to US Environmental Protection Agency (EPA) standards, are versatile and suitable for a wide range of applications.

Table 1. General description of filter paper.

Grade	**QM-A**
Description	Quartz
Particle Retention in Liquid (μm)	2.2
Air Flow (s/100 mL/in²)	6.4
Typical Thickness (μm)	450
Basis Weight (g/m²)	85

To prepare a filter for air sampling using reduced graphene oxide (r-GO): Weigh 20 milligrams of r-GO and place it in a 100-milliliter measuring flask. Fill the flask to the mark with ethanol and dissolve the r-GO using an oscillator for 15 minutes. This will create a stock solution with a concentration of 200 parts per million (ppm). From the stock solution, prepare any desired concentration by diluting it with ethanol. Weigh a filter and then inject 1 milliliter of the desired concentration of r-GO solution onto the filter using a micropipette. Place the filter in a fume hood with suction for 30 minutes to allow it to dry. Weigh the filter again. The filter is now ready to use with the air sampler instrument. After sampling: Digest the filter according to the manufacturer's instructions. Filter the digested sample using double ring filter paper. Collect the filtrate in a polypropylene conical tube and store it for analysis using inductively coupled plasma (ICP).

2.3. Characterization of nanomaterials

Scanning electron microscopy (SEM) images were obtained using a ZEISS FE-SEM ULTRA Plus microscope equipped with an energy-dispersive X-ray spectroscopy (EDX) analyzer and a Philips CM20 microscope, operating at an accelerating voltage of 200 kV. Sample dispersion was deposited onto an aluminum pin stub and allowed to evaporate at room temperature. X-ray diffraction (XRD) measurements were conducted using a Philips PW1710 X-ray diffractometer with Cu Ka radiation (k = 1.54186 Å). The XRD patterns were recorded within the 20° to 80°2θ range with a step size of 0.020°2θ and a collection time of 10 seconds per step. Fourier transform infrared (FT-IR) spectra were recorded using a Nicolet 6700 infrared spectrophotometer to identify specific functional groups present on the surface.

2.4. LIBS Experimental procedures

To generate plasma, a 50 mJNd:YAG laser with a pulse duration of 5 ns was focused onto the surface of the filter under study. The laser operated at a fundamental wavelength of 1064 nm in air at atmospheric pressure. A quartz optical fiber with a 600-micrometer diameter aperture was used to collect the plasma emission. The fiber was aligned and positioned to ensure that it covered most of the plume emission, minimizing inhomogeneity in the laser-induced plasma. The output of the fiber was connected to an echelle spectrometer coupled to an intensified charge-coupled device (ICCD). This enabled simultaneous spectral analysis in the range of 200 to 700 nanometers with a constant spectral resolution ($\lambda/\Delta\lambda$ = 7500). A delay of 1500 nanoseconds and a gate width of 2000 nanoseconds were chosen to maximize spectral line intensity. The spectrometer's gate width and delay time were controlled by a computer. To optimize signal-to-noise ratio and spectral reproducibility, 10 single spectra were accumulated from different positions on the sample surface. Additionally, to mitigate sample inhomogeneity, the average spectra of five different accumulated spectra were taken for each measurement. To minimize angular dispersion of the plasma emission, measurements were performed at distances from the target surface that were shorter or comparable to the spot dimension. The emission spectra were analyzed using the LIBS++ software. The measurements were repeated under the same conditions for each filter [27,28]. The measurements were repeated under the same conditions for each filter.

3. Results and discussion

3.1. Characterizations of r-GO

The scanning electron microscopy (SEM) image in Figure 1a revealed that the r-GO nanosheets exhibited irregular and folded layer structures. These nanosheets were entangled with each other, and the image also highlighted the presence of numerous wrinkles in the single- or few-layer r-GO nanosheets.

The two-dimensional nature of graphene oxide was diminished in the case of r-GO, as indicated by the absence of the narrow XRD reflection at 2θ = 10.8 Å. Instead, a broad band was observed, suggesting possible changes in the intra-layer spacing, as depicted in Figure 2b. In the Fourier-transform infrared (FTIR) spectrum of graphene, three distinct bands at 1724, 1222, and 1050 cm-1 were observed, corresponding to carbonyl, epoxy, and alkoxide functional groups, respectively, as shown in Figure 1c. Furthermore, the specific surface area (SSA) of the prepared r-GO was determined to be 170.98 m^2 g^{-1}, indicating its considerable surface area for potential applications [25,29].

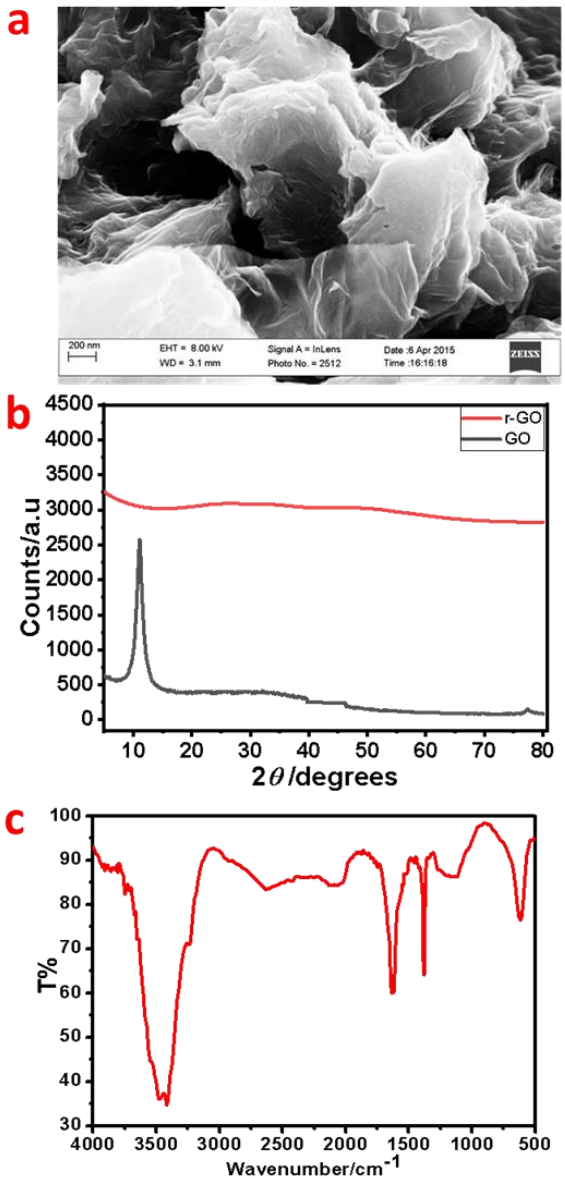

Figure 1: SEM image of the prepared r-GO nanosheets (a), XRD patterns (b), and FTIR spectrum of r-GO.

Spectroscopic measurements were conducted using the filters over a 15-day period. The techniques employed were EDX, ICP, and LIBS.

3.2. EDX Technique results

The morphology of the filter (P) was assessed using SEM images and EDX analysis, as depicted in Figure 2. Analysis of the filter revealed the presence of various elements, including sodium, magnesium, aluminum, silicon, potassium, calcium, and iron. Among these elements, silicon was found to be the most abundant, while titanium was the least abundant (Figure 2a, b). These findings prompted further investigation into the presence of these elements in other filters, with the goal of assessing the impact of treated filters with nanomaterials to adsorb heavy elements and air pollutants. In Figure 2c, d, an increase in the concentrations of potassium, molybdenum, iron, and magnesium elements was observed in this r-GO modified filter compared to the blank filter. The percentage of magnesium, potassium, iron, and molybdenum in the blank filter (1.78, 1.24, 3.76, and 0.95 wt% respectively) increased to (3.1, 4.77, 6.8, and 1.6 wt%) in the r-GO modified filter, respectively. These findings indicate that the efficacy of the filter improves when treated with r-GO, leading to enhanced adsorption of suspended pollutants in the air sample.

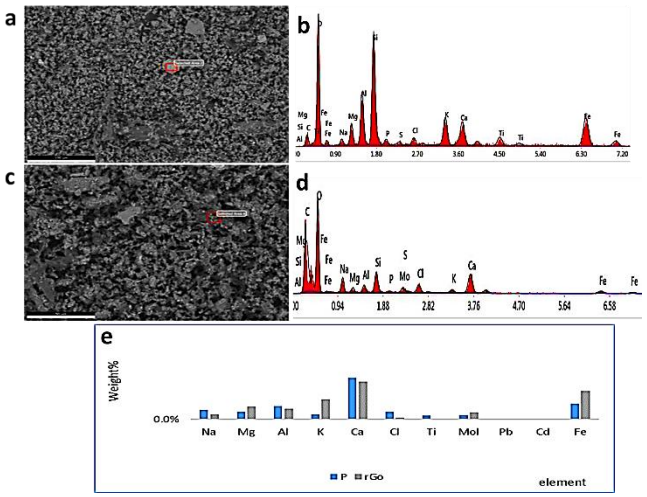

Figure 2: Overall (wt%) of the PMs for blank filter in comparison to the nano-modified filters with r-GO.

3.3. ICP Results

During a 15-day period, the filters were operated at the same location as the initial three filters to observe any variations. Notable differences emerged

when the filters underwent ICP analysis, which is detailed in figure 3. As it can be seen from figure (3) that there is an increase in the concentrations of lead, sodium, potassium, and manganese when using different nanomaterials than that in the case of the blank filter. Zn is not detected by EDS analysis and detected with ICP technique.

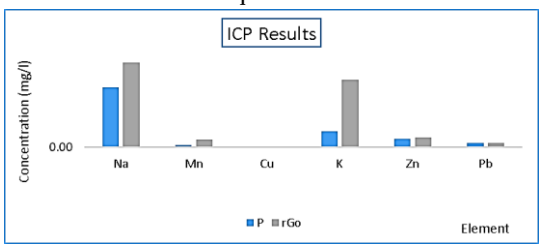

Figure 3: ICP results for the blank filter and their nano-modified r-GO filter.

2.5. LIBS Results

LIBS is an analytical technique that involves using a pulsed laser beam to excite atoms in a sample, creating a plasma plume. The plasma emits light with characteristic spectral patterns, which can be analysed to identify the elements present in the sample. LIBS is versatile and can be used with solids, gases, and liquids, regardless of their conductivity. It requires minimal sample preparation and is partially non-destructive. LIBS can analyse hard materials and allows for simultaneous multi-elemental analysis. However, there are some drawbacks to using LIBS. It can be expensive, and obtaining suitable standards for quantitative analysis can be challenging. The multielement nature of LIBS introduces a matrix effect that can interfere with the accuracy of the analysis. Precision can also be relatively poor, typically ranging from 5% to 10%, depending on factors such as sample homogeneity, sample matrix, and laser excitation properties [21,28]. To analyse the concentration of different elements using LIBS, the spectra of various positions within each filter sample are averaged to obtain a single spectrum for each filter. To minimize experimental fluctuations, the spectra are normalized using the Carbon line at 247 nm. A spectral line is selected for each element of interest, and the intensities of the elemental lines at their respective spectral lines (e.g., Na I at 589.5 nm, Mn I at 403.3 nm, Mg I at 280.2 nm, Fe I at 358 nm, Cu I at 327.3 nm, Cd I at 326.1 nm, Ti I at 506.4 nm, Al I at 309.2 nm, and Zn I at 330.2 nm) are tracked as indicators of elemental concentration in each filter.

The untreated filter (filter sample P) displayed the lowest intensity. It is evident that r-GO was effective carbon nanomaterial in improving the filter's performance. The concentrations of the elements under study (sodium, manganese, magnesium, iron, copper, cadmium, titanium, aluminium, and zinc) were highest in the r-GO modified filter than the untreated filters (refer to Figures 4).

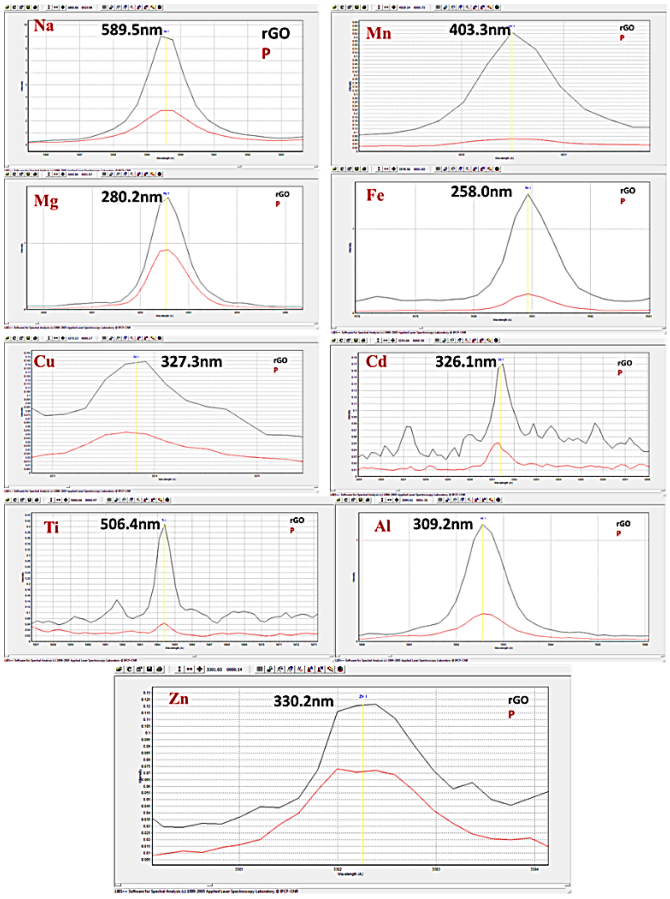

Figure 4: PMs precipitated at the blank and r-GO modified filters with different treatments the intensities of the PMs I spectral line.

There is a big difference between the LIBS spectral lines intensities in case of the treated and untreated samples. This may be attributed to, beside the low adsorption of elements in the untreated filters; the LIBS signal is enhanced when used with nanoparticles [28].

2.6. Comparison LIBS Results to the ICP Results

To justify the results obtained from LIBS, a comparison was conducted between the LIBS results and the results obtained from ICP analysis for the elements Mn, Na, Zn, and Cu. Figure 5 illustrates this comparison for the PMs Na, Mn, Zn, and Cu. The comparison reveals a strong agreement between the LIBS and ICP results for Na, Zn, and Cu. However, for Mn, there is some discrepancy between the two

techniques. Both LIBS and ICP indicate a minimum value for the untreated filters. Additionally, LIBS shows that the highest intensity is observed in the filters treated with r-GO.

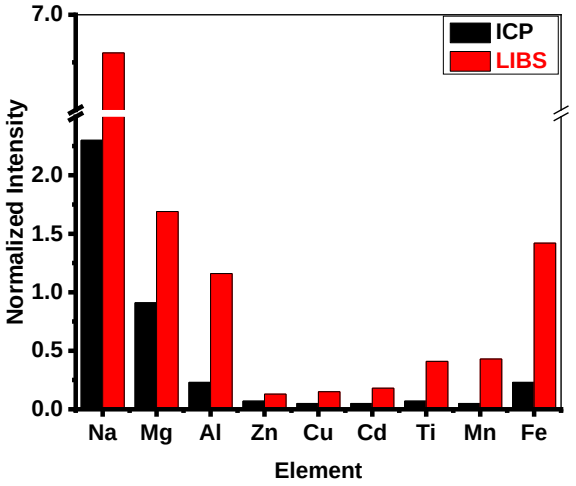

Figure 5: Comparison between LIBS results and ICP results for PMs using r-GO modified filters.

The r-GO modified filter was the most effective at adsorbing heavy metals from the air sample. This was shown by both ICP and LIBS, which showed an increase in filter efficiency when nanomaterials were used. However, LIBS cannot solely demonstrate this change in efficiency because the LIBS signal is naturally enhanced by the presence of nanoparticles [30, 31].

The LIBS and ICP results for PMs elements show that LIBS is a reliable method for analyzing the concentrations of these elements in the filter samples, with a high level of agreement between the two techniques. Both LIBS and ICP show that the untreated filters have the lowest concentration of Mn, while the filters modified with r-GO have the highest concentration of Mn according to ICP. However, LIBS shows a higher value for the filters modified with r-GO compared to those untreated filters, and the highest intensity is recorded in the filters modified with r-GO. These inconsistencies suggest that the accuracy and precision of the LIBS technique may be limited for Mn analysis specifically. Despite this limitation, both LIBS and ICP results clearly demonstrate that the r-GO modified filter is superior at adsorbing heavy elements from the air sample. This finding highlights the potential of r-GO as an effective nanomaterial for enhancing the efficiency of filters in capturing and retaining heavy elements.

Carbon-based particles are becoming popular alternatives for heavy metal removal due to their unique properties, such as exceptional mechanical strength, electrical conductivity, and thermal conductivity. Reduced and oxidized graphene are particularly promising adsorbents for this application. They can be functionalized with groups that enhance metal ion sorption, and their surface atoms can attract adsorbates due to the partial absence of surrounding atoms. This attraction can involve weak van der Waals forces (physisorption) or covalent bonds (chemisorption), depending on the species involved and electrostatic attraction [32-35].

4. Conclusions

This study focused on enhancing the performance of air particle monitoring devices by modifying the filters with reduced graphene oxide (r-GO). The objective was to investigate how r-GO affects the efficiency of PM adsorption. The spectroscopic techniques such as energy-dispersive X-ray spectroscopy (EDX), inductively coupled plasma (ICP), and laser-induced breakdown spectroscopy (LIBS) were used to assess the adsorption efficiency of the modified filters quantitatively and qualitatively towards PMs. The results demonstrated that the filters modified with r-GO exhibited higher PM adsorption efficiency compared to the control filters. This improved performance can be attributed to the exceptionally large specific surface area and pore volume of r-GO. This novel approach is expected to garner significant attention and contribute to the development of advanced air purification technologies.

5. Conflicts of interest

There are no conflicts to declare.

6. Formatting of funding sources

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