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Evaluating occupational exposures to the graphene family of nanomaterials: Implications for worker safety and health

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ABSTRACT

Graphene, a two-dimensional carbon-based nanomaterial, has garnered significant attention due to its exceptional physical and chemical properties, leading to its widespread application in various industries. However, the increasing utilization of this class of materials, also referred to as the graphene family of nanomaterials (GFNs), raises potential concerns regarding occupational exposures and health risks for workers. This study aimed to evaluate occupational exposures to GFNs across 11 primary and secondary manufacturing facilities operating in the U.S. A total of 44 workers participated in the study, with paired personal air samples collected for elemental carbon (EC) analysis at both the respirable and inhalable aerosol size fractions. The results revealed exposures with respirable EC concentrations ranging from <0.01 to 1825.23 $\mu\text{g}/\text{m}^3$ with a geometric mean (GM) of 2.01 $\mu\text{g}/\text{m}^3$ and inhalable concentrations from 0.01 to 6327.10 $\mu\text{g}/\text{m}^3$ with a GM of 12.63 $\mu\text{g}/\text{m}^3$. Notably, 38% of respirable samples exceeded a suggested occupational exposure band (OEB) of <10 $\mu\text{g}/\text{m}^3$ established from a recent publication for GFNs, while 53% of inhalable samples exceeded the OEB. Primary manufacturing facilities that produce GFNs generally exhibited higher inhalable exposure levels compared to secondary manufacturers, likely due to the handling of larger quantities of dry powdered materials. Additionally, the use of engineering controls and personal protective equipment varied widely among facilities, impacting exposure levels. This study represents a crucial step in understanding any potential occupational risks associated with U.S. GFN exposures.

KEYWORDS

Graphene; nanomaterials; exposure assessment

Introduction

Graphene is a two-dimensional (2-D), carbon-based, nanomaterial that demonstrates exceptional physical and chemical properties such as electrical conductivity, mechanical strength, and thermal stability (Mbayachi et al. 2021). The past two decades have witnessed an extraordinary increase in graphene research and innovation since the material was isolated in 2004. Graphene's unique properties have facilitated its use in a variety of manufacturing applications, including semiconductors, composites, sensors, energy storage, functional inks, polymer additives, tires, coatings, electronics, and filtration (Novoselov et al. 2012; Randviir et al. 2014).

Graphene is often referred to as the graphene family of nanomaterials (GFNs) since it is a class of

materials based on their surface properties, number of carbon layers, and size. GFNs can include few-layered graphene (FLG, 2–5 layers), multi-layered graphene (MLG, 2–10 layers), graphene nanoplatelets (GNP, stacks of graphene sheets > 10 layers), as well as materials based on oxygen content, which include graphene oxide (GO) and reduced graphene oxide (rGO, <5% oxygen content) (Niang et al. 2025). These materials can be considered pristine, which is their unaltered form, or functionalized, where they undergo surface modifications to increase solubility and reactivity (Georgakilas et al. 2012; Chowdury et al. 2024). There is a growing consensus on GFN characterization and classification as recent standards have been adopted (ASTM Standard E3220 2020; ISO 2025).

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The worldwide graphene market size was estimated at \$0.76 billion USD in 2023 and is expected to expand at a rate of 40.4% over the next 5 years, reaching \$4.13 billion USD by 2028 (Research and Markets 2024). Additionally, surveys conducted in 2016 and 2019 found that graphene was one of the most reported carbonaceous nanomaterials produced or used by U.S. facilities (Babik et al. 2018; Neu-Baker et al. 2022). With the growing production and application of GFNs, the likelihood of workplace exposures throughout their life cycle is expected to rise. Despite the fast pace of growth that promises further innovations and applications in various fields, the number of workers exposed to GFNs in the U.S. is largely unknown.

Currently, there is limited information regarding potential human health effects associated with occupational exposures to GFNs. To date, only one human clinical research study has been published. Andrews et al. (2024) conducted a controlled acute inhalation (2 hr) study that aerosolized 200 $\mu\text{g}/\text{m}^3$ of GO to expose human participants and reported no changes in respiratory or cardiovascular health and a limited effect on blood coagulability. In the absence of other human data, information on the potential effects of graphene has largely been assessed by animal toxicity studies.

Reviews of acute and sub-chronic animal studies have shown the potential to cause non-cancerous adverse lung effects, including inflammation, fibrosis, or granuloma formation following inhalation, intratracheal instillation, or pharyngeal aspiration in rats and mice (Kong et al. 2024; Niang et al. 2025). Other *in vivo* studies, which used various routes and dosing methods, suggest that pristine and functionalized GFNs may exhibit potential pulmonary, systemic, reproductive, developmental, and genotoxic outcomes in laboratory mammals. (Ema et al. 2017; Rhazouani et al. 2021; Niang et al. 2025). However, the toxicological data remain relatively limited, especially when considering the many different types of GFNs and their potential surface modifications.

Several carbonaceous materials, such as synthetic and natural graphite, carbon black, coal dust, and carbon nanotubes, have occupational exposure limits (OELs) in the U.S. that range from 3.5 to 15 milligrams per cubic meter (mg/m^3) at the total/inhalable aerosol size fractions and 0.001 to 5.0 mg/m^3 at the respirable aerosol size fraction (See Supplemental Table S1; NIOSH 2013a; NIOSH 2019a, 2019b, 2019c, 2019d). Inhalable particles ($D_{50} = 100 \mu\text{m}$) are those that may enter the mouth or nose, while respirable

particles ($D_{50} = 4 \mu\text{m}$) may penetrate the unciliated or gas-exchange regions of the respiratory system (Vincent et al. 1999).

There are currently no international OELs published by authoritative organizations to ensure the safe handling of GFNs in workplaces. However, several independent research groups have conducted risk assessments that derived preliminary OELs of 18 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) and 212 $\mu\text{g}/\text{m}^3$ from different types of GFNs (Lee et al. 2019; Spinazzè et al. 2019). Additionally, researchers have also used the NIOSH Occupational Exposure Banding process on GFNs. Results of the banding process indicated that graphene exposures may have potential health consequences at air concentrations $<10 \mu\text{g}/\text{m}^3$ (NIOSH 2019e; Niang et al. 2025).

To date, there is limited occupational exposure data for GFNs captured outside of small-scale research and development settings, and no published exposure assessment research has been conducted within U.S. workplaces to determine exposures to GFNs. Therefore, the primary objective of this study was to conduct an industry-wide exposure study of graphene at representative primary manufacturing facilities and downstream secondary manufacturing facilities. The aim was to (1) demonstrate the feasibility of using facility background corrected elemental carbon (EC) measurements as a marker for assessing GFN exposures, (2) understand the range of occupational exposures within U.S. workplaces, and (3) inform risk assessors and toxicologists on realistic exposure information and material characteristics of GFNs.

Methods

Facilities and participants

A total of 11 site visits were conducted between September 2019 and August 2023 at facilities across the U.S. producing or handling GFNs. Companies were recruited from a combination of those who responded to a previous survey or through trade association contacts (Babik et al. 2018). Workers from various departments and job titles who either directly handled the materials or who were potentially indirectly exposed as part of their workday were invited to participate. The study was explained to all workers, and written consent was obtained. Furthermore, this study was reviewed and approved by the National Institute for Occupational Safety and Health (NIOSH) Institutional Review Board.

During each site visit, an initial walkthrough of the facility was conducted to establish potential exposure

sources and possible sampling locations. An overview of facility demographics and the characteristics of GFNs from the 11 facilities is tabulated in [Table 1](#). Facility-level determinants were collected from observations and during an interview with either a site representative or production manager. Collected information included types of GFN handled, material characteristics (carbon layers), observed daily quantities produced or handled, the form(s) of GFN handled, and company workforce size. Brief descriptions of each facility's processes can be found in the [supplemental material](#). Additionally, each facility was categorized into one of two distinct industries, which included:

1. *Primary Manufacturers*—Facilities that produced graphene and subsequently sold their products for research/industrial purposes. Graphene production methods vary but can generally be classified into two classes: bottom-up and top-down (Urade et al. 2023). Top-down production has higher yields but may produce a lesser quality graphene, while bottom-up methods may produce lower yields but generally higher quality graphene materials (Gutiérrez-Cruz et al. 2022).
2. *Secondary Manufacturers*—Facilities that performed additional processing of the graphene materials to create an intermediate or final product. This included a facility that was using graphene in thin films for electronic components in medical equipment, a facility that repackaged and sold GFNs as a distributor, and two facilities that produced functionalized graphene additives for use in thermoplastics and composites.

Personal sampling and data collection

The sampling strategy was adapted from previous NIOSH exposure assessments of other carbonaceous nanomaterials, such as carbon nanotubes (Dahm et al. 2015, 2018). When possible, multiple days of personal sampling data were collected for each participant to better understand daily exposure variations. Worker-level determinants were collected through a combination of observations and a short interview at the end of each sampled work-shift. Collected data included the job task performed throughout the workday (see descriptions in [supplemental materials](#)), direct or indirect exposure durations to GFNs, quantity and form of material handled, as well as engineering controls and personal protective equipment (PPE) used throughout the sampling period.

Personal exposures were assessed using three personal air samplers for each participant. Two samplers collected airborne elemental carbon (EC) at the respirable and inhalable aerosol size fractions, while a third was used to confirm airborne GFN exposure via transmission electron microscopy (TEM) analysis collected at the inhalable aerosol size fraction. All three collected personal samples were co-located, with sample inlets positioned together to sample the same air space in the personal breathing zone of the worker.

Direct reading instruments for area air sampling were co-located to measure particle number (CPC 3007; TSI Inc., Shoreview, MN, USA), mass (DustTrak DRX 8533, TSI Inc), and black carbon (microAeth MA200, AethLabs, San Francisco, CA, USA). The results from these instruments will be discussed in a separate manuscript (Niang et al. [forthcoming]).

Elemental carbon analysis

Personal samples were collected for the airborne mass concentration of elemental carbon (EC) at the respirable and inhalable aerosol size fractions to assess GFN exposures. Personal respirable aerosol collection for EC was performed by using a 25-millimeter (mm) cassette with quartz fiber filters (QFF) attached to a GK 2.69 cyclone (Mesa Labs Inc., Lakewood, CO, USA) and an Airchek TOUCH sampling pump (SKC Inc., Eighty Four, PA, USA) operating at the cyclone specified flow rate of 4.2 liters of air per minute (lpm). Customized adapters for the GK 2.69 cyclone (BGI Inc., Waltham, MA, USA; catalog number 3503) were used to fit the 25-mm cassettes. Additionally, personal inhalable samples were collected using 25-mm QFFs (SKC Inc.) within a Button Aerosol Sampler (SKC Inc.) and Airchek TOUCH sampling pump (SKC Inc.) operating at the sampler specified flow rate of 4.0 lpm.

The airborne mass concentration of EC was analyzed using the NIOSH Manual of Analytical Methods (NMAM) 5040 (NIOSH 2006), a thermal-optical analysis technique for organic and elemental carbon (OC-EC). Bulk samples of GFN were collected from each visited facility, when available, and analyzed to obtain their thermal profiles and used for manual assignment of the OC-EC “split” (Birch et al. 2011; NIOSH 2013a; Dahm et al. 2015). The reported limit of detection (LOD) for NMAM 5040 ranged from 0.1 to 1.0 μg EC per square centimeter ($\mu\text{g}/\text{cm}^2$) of filter deposit, with an average of 0.4 $\mu\text{g}/\text{cm}^2$. Based on the total mass of respirable/inhalable EC collected on the filter and the

Table 1. Facility and GFN material characteristics.

Facility ID	Industry	Total Workforce Size on Site	Employees Handling Graphene	GFN Type	Carbon Layers [†]	Daily Quantity Handled	Primary Form(s) of Material	General Job Tasks	Respiratory* Protection	Engineering Controls
1	Primary (Top-Down)	30	12	GNP	> 25	1 - 100 kg	Dry Powder; Dispersed in Liquid	Graphite loading, Production, Sonication, Wet cake filtration, Maintenance, Post-processing, Packaging, Shipping, OW	Half-Face w/ P100 Cartridges	HEPA Ventilated Enclosure, Enclosed Process, LEV
2	Primary (Top-Down)	20	15	GNP	> 10	> 100 kg	Dry Powder	Graphite loading, Production, Post-processing, Maintenance/Equipment cleaning, QA/QC, Packaging, OW	Half-Face w/ P100 Cartridges	HEPA Ventilated Enclosure, Unventilated Enclosure
3	Secondary-Electronics	5	2	GO	2-5	< 100 g	Dispersed in Liquid; Thin Films	Wet cake filtration, Sonication, QA/QC, Other lab work, OW	None	Chemical Fume Hood
4	Primary (Top-Down)	300	15	rGO	3-5	101 g – 1 kg	Dry Powder	Graphene oxide reduction, Sonication, Post-processing, QA/QC, OW	Half-Face w/ P100 Cartridges	Chemical Fume Hood, Ventilated Enclosure
5	Secondary-Distributor	20	8	GO	NA	1 - 100 kg	Dry Powder	Post-processing	Half-Face w/ P100 Cartridges	None
6	Secondary-Composites	500	4	GO	NA	1 - 100 kg	Dispersed in Liquid; Dried/Wet Cake	Wet cake functionalization, Drying	Half-Face w/ Acid/Gas Cartridges	Ventilated Enclosure
7	Secondary-Composites	8	5	GO	NA	101 g – 1 kg	Dispersed in Liquid; Dried/Wet Cake	Wet cake functionalization, Sonication, QA/QC, Spray coating, Drying, Packaging, OW	Full-Face w/ Acid/Gas Cartridges	Chemical Fume Hood
8	Primary (Top-Down)	100	10	GNP	> 10	> 100 kg	Dry Powder	Production, Sonication, QA/QC, Packaging, OW	Half-Face w/ P100 Cartridges	Unventilated Enclosure
9	Primary (Top-Down)	5	3	FLG	2-5	1 - 100 kg	Dry Powder; Dispersed in Liquid	Graphite loading, Production, Post-processing, Drying, Maintenance, Packaging, Other lab work, OW	Half-Face w/ P95 Cartridges	Unventilated Enclosure
10	Primary (Top-Down)	4	2	MLG	3- > 10	101 g – 1 kg	Dry Powder; Dispersed in Liquid	Production, Sonication, Post-processing, OW	N95 filtering facepiece respirator	None
11	Primary (Bottom-Up)	5	4	FLG	3-9	1 - 100 kg	Dry Powder	Production, Wet cake filtration, Post-Processing, Maintenance, QA/QC, Packaging, Shipping, Other lab work, OW	N99 filtering facepiece respirator	HEPA Ventilated Enclosure

GNP = graphene nanoplatelets; GO = graphene oxide; rGO = reduced graphene oxide; FLG = few layered graphene; MLG = multi-layered graphene; NA = data not available; kg = kilograms; g = grams.

OW = office work; QA/QC = quality assurance and quality control; LEV = local exhaust ventilation; HEPA = high-efficiency particulate air.

*All facilities used nitrile gloves and safety glasses as standard personal protective equipment. †As reported by the company.

sample-specific collected air volume, respirable/inhalable EC mass concentrations were calculated and reported as micrograms per cubic meter ($\mu\text{g}/\text{m}^3$).

The measurement of EC mass using NMAM 5040 is a nonspecific marker for GFN exposure but has been successfully used to assess exposures for other carbonaceous nanomaterials (Debia et al. 2016; Dahm et al. 2018; Canu et al. 2020). Consequently, to account for potential interferences, daily outdoor and/or indoor area background measurements for the airborne mass of EC at the respirable and inhalable size fractions were collected using the same methods as previously stated.

Interferences in NMAM 5040 can be caused by anthropogenic sources such as diesel exhaust, emissions from coal or oil-fired power plants, and the seasonal burning of biomass (Schauer 2003). Locations for area background sampling were selected based on professional judgment and knowledge of each facility's ventilation. Most area background samples were collected in an office or conference room under a fresh air supply that was separated from the main GFN production/processing area. The EC personal measurements were background-corrected to adjust for anthropogenic sources of EC by subtracting the facility-specific area background concentration, by type of sample (respirable or inhalable), as discussed in Dahm et al. (2015 and 2018).

Electron microscopy analyses

Personal inhalable samples were collected using 25-mm mixed cellulose ester filters (SKC Inc.) within a Button Aerosol Sampler (SKC Inc.) using an Airchek TOUCH sampling pump (SKC Inc.) operating at 4.0 lpm. Scanning electron microscopy (SEM) was performed to understand particle loading and distribution on the filter. Transmission electron microscopy (TEM) was performed to characterize particle morphology. Each filter was processed for SEM/TEM analysis following the modified NMAM 7402 (Birch et al. 2017). SEM analysis was performed using a Phenom XL SEM (Thermo Fisher Scientific, Waltham, MA, USA) with a backscattered electron (BSE) detector in the low-vacuum mode. The loading level and particle distribution of each sample were determined by analyzing an auto-montaged image stitched from 16 tiles of SEM images. TEM analysis was performed using a JEOL 2100F TEM (JEOL USA, Inc., Peabody, MA, USA). Each TEM grid was analyzed in TEM and scanning TEM modes.

Statistical analyses

Eight-hour, time-weighted average (TWA) values are reported throughout, as the average sample time was 440 min, and <6% of samples were collected for <240 min. Unsampld time periods were assumed not to accumulate any EC exposure. Descriptive statistics for the airborne mass concentration of EC at both the respirable and inhalable size fractions were calculated, and median, arithmetic mean (AM), standard deviation (SD), geometric mean (GM), geometric standard deviation (GSD), and 5th and 95th percentiles are reported. For non-detectable airborne mass concentrations, values were imputed using the multiple order value imputation method recommended by Pleil (2016).

Marginal regression models were fitted using generalized estimating equations (GEE) with an exchangeable working correlation structure to account for the statistical dependence among personal air samples from the same worker and among workers within the same facility (Chen et al. 2024). The dependent variables in the models were EC mass measured at the respirable and inhalable aerosol size fractions. The distributions of these concentrations were found to be right-skewed (Figure 1).

Consequently, a logarithmic transformation was applied to normalize the data before analysis. The independent variables evaluated in the models included facility-level and worker-level determinants. Regression parameter estimates were obtained to examine the magnitude and direction of differences in mean airborne mass concentrations collected from personal samples across various determinants. All analyses were performed in R version 4.4.1 (R Core Team, Vienna, Austria) and the *marlode* package (Chen 2025). Statistical tests were two-sided at the 0.05 significance level.

Results

All four secondary manufacturing facilities that participated in this study processed or handled GO (Table 1). Of the seven primary manufacturing facilities visited, three produced GNP, two produced FLG, and one each produced MLG and rGO. The range of employees directly handling graphene at each facility was between 2 and 15 workers. Ten of the 11 facilities visited had the capability to produce or handle kilogram (kg) quantities during the workday. The maximum quantity handled was at two primary manufacturers of GNP that handled over 100 kg per day. Facility personnel, except in one facility, were

observed to use respiratory protection for at least some of the tasks sampled, and all but two facilities used some form of engineering control.

General exposure findings

In total, 44 workers were enrolled in this exposure assessment study. Subsequently, 78 respirable and 77 inhalable filter-based personal air samples (77 paired samples) were collected and analyzed for EC at the inhalable and respirable aerosol size fractions, with an additional 78 samples undergoing a confirmatory TEM analysis (Table 2). One inhalable sample was unable to be analyzed due to a sampling error. All personal samples at the inhalable aerosol fraction for EC yielded airborne mass concentrations above the LOD, while 70 samples at the respirable aerosol fraction (90%) had concentrations above the LOD. Additionally, 14 respirable and 26 inhalable samples collected from Facilities 1, 2, and 9 contained large amounts of EC that exhibited incomplete combustion, which likely led to an underreporting of EC mass.

As shown in Table 2, the overall personal background-corrected, respirable EC TWA GM was $2.01 \mu\text{g}/\text{m}^3$ (GSD: 36.51). Detectable TWA respirable air concentrations ranged from <0.01 to $1,825.23 \mu\text{g}/\text{m}^3$. At the inhalable size fraction, the overall background-corrected EC personal air TWA GM was $12.63 \mu\text{g}/\text{m}^3$ (GSD: 14.80). Background-corrected inhalable exposures ranged from 0.01 to $6,327.10 \mu\text{g}/\text{m}^3$ at individual sites. All but eight TEM samples, which were collected from Facility 3, positively identified GFNs. Figure 2 provides representative TEM images of GFNs from personal air samples. Across all facilities, the average respirable to inhalable exposure concentration ratio was 0.50 with a median of 0.24.

Facility-level exposures and determinants

At individual facilities, TWA GMs at the respirable and inhalable aerosol size fractions ranged from <0.01 to $304.90 \mu\text{g}/\text{m}^3$ and 0.66 to $1135.58 \mu\text{g}/\text{m}^3$, respectively (Table 2). Top-down, primary manufacturing facilities 1, 2, 8, 9, and 10 all had a respirable or inhalable GM concentration over the suggested OEB ($<10 \mu\text{g}/\text{m}^3$) or OEL of $18 \mu\text{g}/\text{m}^3$, while the remaining two primary manufacturers (4 and 11) and secondary manufacturers (3, 6, and 7) were below. Figure 3 displays the range of TWA EC results for both primary and secondary manufacturing facilities at both the respirable and inhalable size fractions. The median EC concentrations for both size fractions were

generally higher in the top-down primary manufacturing facilities compared to secondary manufacturing facilities and the bottom-up primary manufacturing facility.

Marginal regression modeling revealed that working at a secondary manufacturer in the electronics and functionalization industries significantly decreased a worker's exposure potential at the inhalable size fraction compared to a worker at a primary manufacturer ($p = <0.01$ and $p = 0.02$, Supplemental Table S2). Additionally, facilities handling more than 100 kg of GFNs daily were found to significantly increase respirable ($p = 0.02$) and inhalable ($p = 0.0001$) EC concentrations compared to those handling less than 100 g.

The material form handled at the facility-level also significantly increased inhalable EC exposures when facilities handled dry powder GFN ($p = 0.01$). Also, facilities handling GFNs in a solid form, such as a composite or dried cake material, significantly decreased exposures ($p = 0.01$). Workforce size was found to modestly decrease inhalable exposures as the number of employees increased ($p = 0.04$). Additionally, when the results from a subjective categorical assessment, similarly used in Dahm et al. (2019), were found to be inadequate or semi-adequate, a significant increase was found in respirable and inhalable EC exposures compared to those rated as adequate (Supplemental Table S2).

Worker-level exposures and determinants

As seen in Table 3, 59% of workers were observed to handle GFNs in a dry powder form, which resulted in the highest respirable and inhalable GM EC exposures, while fewer than half of participants used GFNs in a liquid (42%) or solid (31%) form. The most common mass quantity of material handled by participants was between 1 and 100 kg of material (37%). The other mass quantities were handled by only 10–17% of participants. Engineering controls observed to be in use while workers were handling GFN included ventilated or unventilated enclosures (33%), fume hoods (18%), and local exhaust ventilation (5%). Additionally, 45% of participants were observed using a NIOSH-approved respirator.

The regression model found few worker-level determinants that significantly increased exposure, while no determinants were found to significantly decrease exposure. Participants who spent more time directly handling GFN, had a confirmatory TEM sample of GFN exposure, or used a ventilated or unventilated enclosure were found to significantly increase

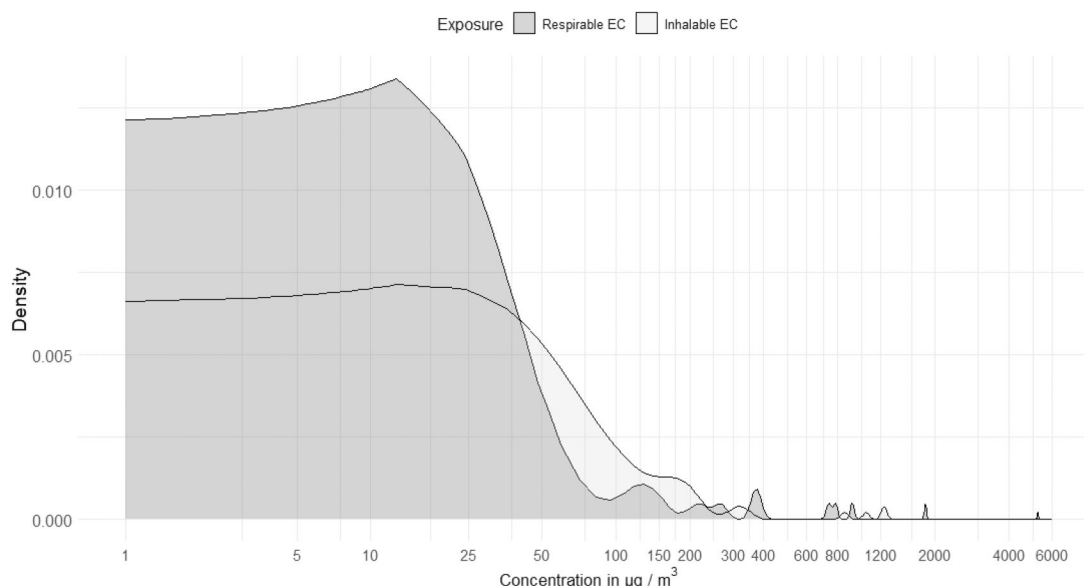


Figure 1. Density plot showing the distribution of personal respirable and inhalable EC GFN exposures.

Table 2. Elemental carbon time-weighted average personal exposures by facility.

Facility ID*	Size Fraction	<i>n</i>	Median (µg/m ³)	AM (µg/m ³)	SD	GM (µg/m ³)	GSD	5th %	95th %	Resp/Inhal Ratio
Site 1 [†]	Resp	14	4.20	66.88	196.62	4.67	10.37	0.31	352.71	0.50
	Inhal	14	26.04	50.00	53.28	30.55	2.81	8.94	147.90	–
Site 2 [†]	Resp	14	25.96	88.65	203.10	31.40	3.57	5.76	358.65	0.52
	Inhal	14	75.81	105.66	80.27	83.86	1.99	36.35	242.50	–
Site 3	Resp	8	0.19	0.97	1.27	0.28	7.39	0.03	2.94	0.86
	Inhal	8	1.32	1.42	0.93	0.79	6.17	0.27	2.70	–
Site 4	Resp	5	<0.01	<0.01	<0.01	<0.01	1.22	<0.01	<0.01	NA ^{††}
	Inhal	5	1.12	1.35	0.88	1.17	1.78	0.66	2.55	–
Site 6	Resp	4	0.02	0.07	0.10	0.04	3.62	0.01	0.20	0.04 [‡]
	Inhal	4	0.43	1.24	1.71	0.66	3.38	0.29	3.32	–
Site 7	Resp	8	0.69	1.94	3.68	0.87	2.94	0.41	7.51	0.50
	Inhal	8	1.56	7.08	15.72	1.53	6.97	0.28	30.93	–
Site 8	Resp	5	17.65	34.11	46.34	3.72	31.03	0.08	97.30	0.54
	Inhal	5	184.00	140.60	138.80	39.10	11.14	2.43	299.90	–
Site 9 [†]	Resp	7	369.18	570.23	618.73	304.90	4.25	77.27	1553.34	0.28
	Inhal	7	1225.80	2286.33	2443.95	1135.58	4.79	293.79	6002.08	–
Site 10	Resp	4	9.48	8.54	5.57	6.19	3.04	2.16	13.61	0.21
	Inhal	4	63.21	64.86	53.32	34.30	5.57	9.75	122.29	–
Site 11	Resp	8	0.30	1.13	2.29	0.36	4.25	0.08	4.72	0.64
	Inhal	7 [§]	0.73	1.80	2.07	0.97	3.44	0.27	4.96	–
Total Samples Collected	Resp	78	2.90	82.27	261.66	2.01	36.51	<0.01	432.64	0.50
	Inhal	77	12.51	250.21	946.77	12.63	14.80	0.29	1085.32	–

n = total number of observations; µg/m³ = micrograms per cubic meter; Resp = respirable aerosol size fraction; Inhal = inhalable aerosol size fraction; AM = arithmetic mean; SD = standard deviation; GM = geometric mean; GSD = geometric standard deviation; % = percentile; NA = data not available.

*Site 5 was not included as it contained 1 sample, but was included for the analysis of the total samples collected.

[†]Contained samples with large amounts of EC that exhibited incomplete combustion, some EC masses are likely underreported.

^{††}All samples collected at this site had respirable samples under the limit of detection and were excluded from resp/inhal ratio analysis.

[‡]Half of the respirable samples were under the limit of detection and were excluded from the resp/inhal ratio analysis.

[§]One inhalable sample was unable to be analyzed due to a sampling error.

inhalable EC exposures (Supplemental Table S2). The estimated within-facility correlation, which reflects the similarity of exposure measurements among different workers at the same facility, was 0.31 for the respirable fraction and 0.43 for the inhalable size fraction. The within-worker correlation, representing the day-to-day consistency of exposure measurements for the same worker, was 0.65 for the respirable fraction and 0.96 for the inhalable size fraction.

Exposures by job task

The most frequent job tasks observed across the 11 facilities included office work (46%), followed by filtration/functionalization of wet GFN (28%), post-processing of dry GFNs (26%), and GFN production (24%) (Table 4 and see Supplemental Materials for descriptions of job tasks). Of workers that performed these job tasks, GFN production was performed the

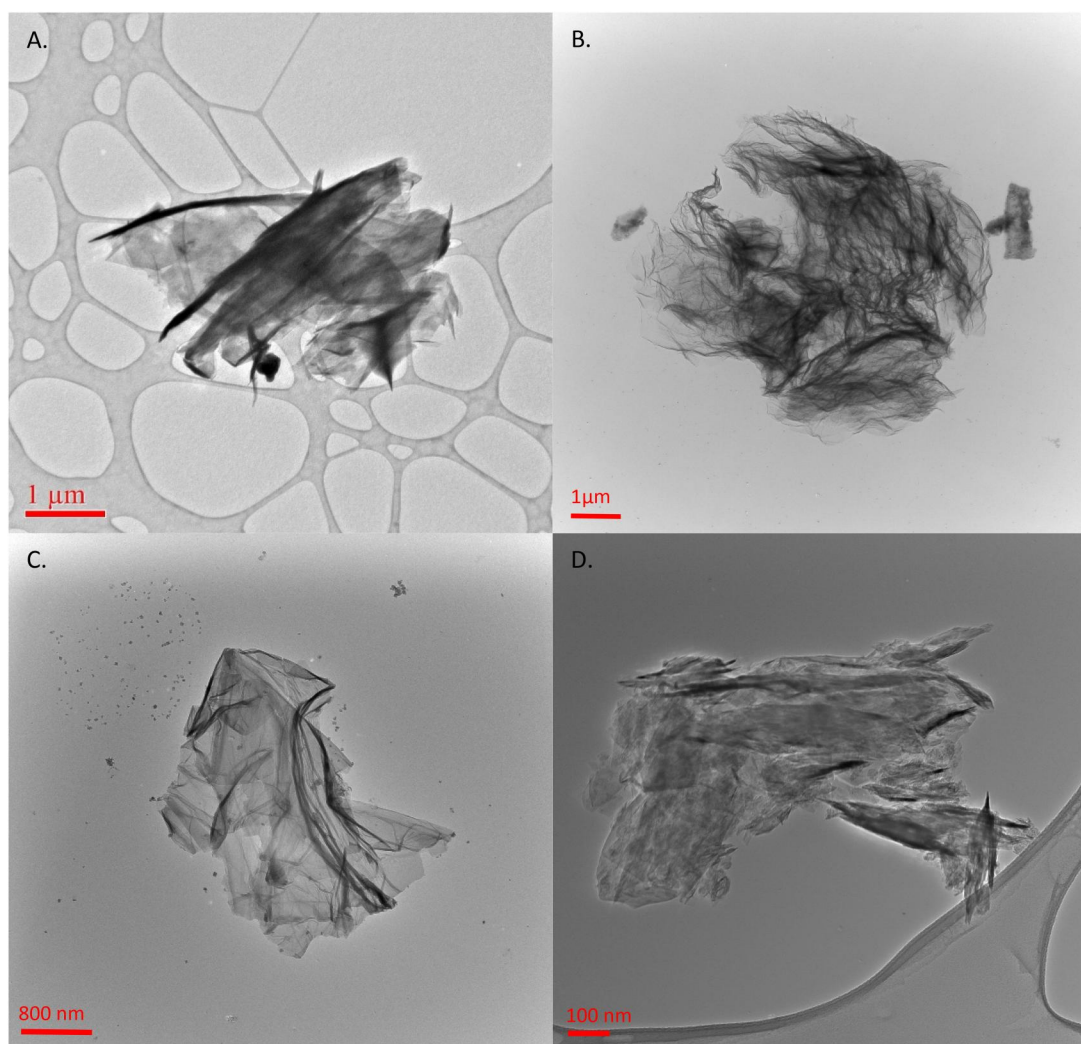


Figure 2. Transmission electron microscopy images of GFNs collected from personal breathing zone samples of workers from (A) GNP from Facility 2; (B) GO from Facility 5; (C) GO from Facility 7; (D) FLG from Facility 9.

longest per sampling period (339 min) on average, followed by office work (299 min), post-processing (248 min), and maintenance tasks (225 min). The highest exposures at both the inhalable and respirable size fractions were associated with job tasks that included packaging final GFN products, drying wet GFNs, loading graphite into reactors to begin top-down production, GFN production, and performing maintenance on equipment.

Few job tasks were significantly associated with decreasing or increasing the exposure potential within the regression model. The job task of loading graphite was modestly associated with a significant decrease in exposure at the respirable size fraction ($p < 0.05$). Conversely, the packaging GFN task was modestly associated with a significant increase in exposure at the respirable size fraction ($p < 0.04$). Additionally, spray coating GFN solutions ($p = < 0.00001$) was

found to significantly decrease inhalable EC TWA exposures, but this task was only completed at one facility (Supplemental Table S2).

Discussion

This study assessed occupational exposures to GFNs for 44 participants from 11 primary and secondary manufacturing facilities located across the U.S. Participating facilities produced or used different types of GFNs with varying amounts of carbon layers, and over half of the facilities produced or used kilogram quantities daily. Additionally, most of the represented facilities were small businesses with fewer than 30 employees.

Generally, many of the smaller facilities did not have the resources to have a dedicated occupational and environmental health and safety professional to anticipate hazards, develop worker training, perform

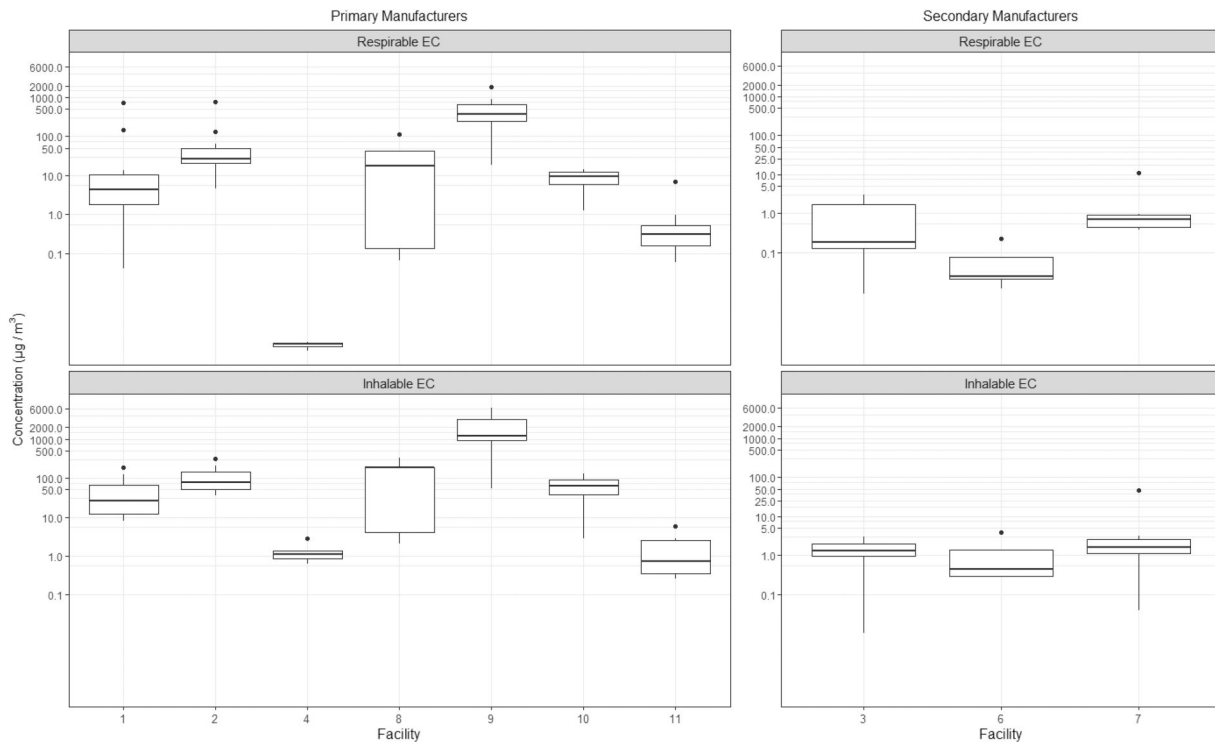


Figure 3. Five number summaries (minimum, first quartile, median, third quartile, maximum, and outliers) of EC respirable and inhalable exposures by facility type. Site 5 was not included as it contained one sample.

Table 3. Percentage and exposure statistics of participants observed handling GFN materials by form and quantity and use of controls and respiratory protection by site.

		Facility ID ^c											Summary Statistics by Category				
	%*	n	1	2	3	4	6	7	8	9	10	11	Overall % [†]	GM Resp. (µg/m ³) ^{††}	GSD Resp.	GM Inh. (µg/m ³) ^{††}	GSD Inh.
Material Form [‡]	Dry	46	50	93	0	80	0	25	100	71	100	63	59	4.28	64.23	31.11	10.58
	Liquid	33	57	0	38	40	100	88	20	57	50	25	42	0.88	42.00	6.95	18.66
	Solid	24	21	0	50	40	100	88	20	43	0	0	31	0.44	54.61	3.72	18.22
Mass Quantity Handled	None	8	21	7	25	20	0	0	0	0	0	13	10	0.25	32.60	4.95	6.03
	< 10 g	13	0	0	75	0	0	25	20	0	0	50	17	0.26	5.06	0.52	5.36
	11–100 g	9	0	14	0	0	0	50	20	0	50	0	12	2.15	6.76	9.04	7.73
	101 g–1 kg	10	7	7	0	80	0	13	0	0	50	13	13	0.04	190.90	4.01	5.06
	1–100 kg	29	57	57	0	0	100	13	0	71	0	25	37	7.94	23.62	39.67	11.92
	> 100 kg	9	14	14	0	0	0	0	60	29	0	0	12	102.60	3.86	256.20	2.40
Engineering Controls Used [‡]	Enclosure	26	21	79	0	40	25	0	60	29	0	50	33	6.65	60.26	50.76	7.61
	LEV	4	7	0	0	60	0	0	0	0	0	0	5	<0.01	243.50	2.41	2.43
	Fume hood	14	7	0	50	80	0	63	0	0	0	0	18	0.05	73.38	1.51	4.79
Respiratory Protection [§]	None	39	64	21	50	20	75	38	40	71	100	50	50	1.67	27.60	10.21	17.78
	Used	35	29	0	0	40	100	100	60	86	100	38	45	3.40	46.01	17.52	21.13

LEV = local exhaust ventilation; n = total number of observations; GM = geometric mean; GSD = geometric standard deviation; Resp. = respirable aerosol fraction; Inh. = inhalable aerosol fraction; µg/m³ = micrograms per cubic meter; g = grams; kg = kilograms.

^cSite 5 was not included as it contained one sample, but was included for the analysis of the overall percent.

*The percentage of participants was calculated by dividing the number of times the form or quantity was observed or reported at each site by the total number of samples at the site, as seen in Table 2.

[†]Personal full shift samples were used to calculate the geometric means for those workers across all sites as task-based samples were not collected. Therefore, the geometric mean could reflect other observations on the same day.

^{††}The overall percentage was calculated using n divided by the total number of samples collected (n = 78).

[‡]Participants could handle more than one material form or use more than one type of engineering control per day.

[§]Respiratory protection included NIOSH approved N95 and P100 filtering facepiece respirators as well as half and full-face respirators with NIOSH approved cartridges.

exposure monitoring, and implement proper engineering controls. However, every facility had at least one employee who worked either full-time or part-time on these issues and expressed awareness of the importance of controlling exposures to GFN.

Most facilities used some combination of exposure control measures and PPE to reduce GFN exposures. However, the extent of their use and capacity to mitigate exposures varied greatly from facility-to-facility. Nearly every facility and 45% of participants were observed using either NIOSH-approved filtering facepiece or elastomeric half-face respirators for respiratory protection. However, participants at two facilities were observed using acid/gas respirator cartridges, which do not protect against GFN or other particulate exposures.

All but two of the facilities had some type of engineering control(s) in place to reduce workplace GFN exposures. Interestingly, workers who used a ventilated or unventilated enclosure were found to have the highest exposures, followed by workers who did not use any engineering controls (Table 3). This was likely attributable to many of the production processes being enclosed, except for short periods when large amounts of material were manually handled or transferred. It should be noted that engineering controls have been proven to be effective at controlling exposures for nanomaterial production and downstream processes (NIOSH 2013b).

To date, there is no consensus on exposure assessment or air sampling methods for GFNs. However, exposure assessment methods that utilize elemental carbon (EC) as a marker for exposure, usually collected alongside a confirmatory sample analyzed by electron microscopy, have been developed and validated for other carbonaceous nanomaterials and continue to gain consensus in the literature (NIOSH 2013a; Canu et al. 2020; McCormick et al. 2021). Recent international studies have also used similar methods, which have further demonstrated the feasibility of using EC to assess occupational exposures to GFNs (Lee et al. 2016; Vaquero et al. 2019; Lovén et al. 2021; Storsjö et al. 2024; Poikkimäki et al. 2025).

Lee et al. (2016) collected personal samples to assess EC levels at the respirable size fraction across two research laboratories producing and handling small quantities of GO and rGO and creating graphene thin films. No personal samples collected for EC were found to be above the LOD ($0.3 \mu\text{g}/\text{cm}^2$). Vaquero et al. (2019) assessed GO and rGO exposures at a pilot production plant with the capacity to produce up to 1 kg per day of material, and the only

personal respirable sample collected contained EC levels below the LOD ($1 \mu\text{g}/\text{cm}^2$). Lovén et al. (2021) assessed EC levels at two facilities making ink formulations for surface coatings. EC levels captured in the personal breathing zone ranged from below the LOD ($0.06 \mu\text{g}/\text{cm}^2$) to $5.6 \mu\text{g}/\text{m}^3$. Storsjö et al. (2024) collected total aerosol personal samples from two workers at a graphene production facility that produced less than 100 grams of graphene powder per day and reported EC exposures of 20 and $60 \mu\text{g}/\text{m}^3$ with an LOD of $0.2 \mu\text{g}/\text{cm}^2$. Poikkimäki et al. (2025) assessed respirable EC levels from seven laboratory-scale exposure scenarios handling rGO, GO, and FLG and did not report any EC concentrations above the LOD ($0.31 \mu\text{g}/\text{cm}^2$).

Other studies assessing GFN exposures have focused on exposure metrics such as gravimetric mass analysis and the use of direct reading instruments to assess mass, lung deposited surface area, and particle number concentration (Heitbrink et al. 2015; Spinazzè et al. 2016, 2018; Boccuni et al. 2018, 2020; Iavicoli et al. 2018; Bellagamba et al. 2020, 2023; Tombolini et al. 2021; López et al. 2023). Generally, these studies assessed exposures at facilities producing low volumes of GFNs (<kg). Additionally, previous studies on other carbonaceous nanomaterials have shown that the use of direct reading instruments may not be the most appropriate tools for collecting quantitative exposure assessment data because of the inability to assign gradient levels of exposure (Evans et al. 2010; Dahm et al. 2013).

In this study, workers had a wide range of exposures to GFNs at the respirable (<0.01 to $1825.23 \mu\text{g}/\text{m}^3$) and inhalable (0.01 to $6327.10 \mu\text{g}/\text{m}^3$) aerosol size fractions. The highest exposure at the inhalable size fraction in this study was several orders of magnitude higher than what has been previously reported in the literature for GFN exposures. Generally, the primary manufacturing sites had the highest exposures in this study, a finding that was supported in the regression model, as two of the three types of secondary manufacturers were found to have significantly decreased exposures compared to primary manufacturers. This difference in exposure was likely attributable to many of the primary manufacturers producing and handling larger quantities of dry powdered GFN for longer durations throughout the workday, while secondary manufacturers used GFN materials dispersed in liquids more frequently.

It remains a challenge to place the range of exposures demonstrated in this study within the appropriate context to adequately protect workplace health

Table 4. Average duration, percentage, and exposure statistics of participants performing job tasks by site.

Daily Job Task*	Facility ID											Summary Statistics by Task				
	1	2	3	4	5	6	7	8	9	10	11	Total (n = 78)	GM Resp. ($\mu\text{g}/\text{m}^3$) [‡]	GSD Resp.	GM Inh. ($\mu\text{g}/\text{m}^3$) [‡]	GSD Inh.
Avg. Duration in min., (%) [†]																
Office work	405 (14)	540 (7)	392 (100)	480 (20)	0 (0)	0 (0)	241 (100)	300 (40)	207 (71)	360 (100)	156 (63)	299 (46)	0.98	19.49	4.66	16.61
Shipping	120 (7)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	30 (13)	75 (3)	0.20	9.49	5.16	2.39
Maintenance	390 (36)	210 (21)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	30 (57)	0 (0)	220 (38)	225 (19)	13.60	15.96	57.40	14.88
Post-processing	473 (14)	276 (36)	0 (0)	293 (80)	83 (100)	0 (0)	0 (0)	0 (0)	340 (43)	83 (100)	20 (13)	248 (26)	2.25	132.95	35.16	13.87
GFN production	330 (14)	362 (71)	0 (0)	300 (20)	0 (0)	0 (0)	0 (0)	380 (60)	210 (29)	0 (0)	300 (13)	339 (24)	14.30	22.65	67.36	6.96
Graphite loading	150 (7)	118 (36)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	15 (14)	0 (0)	0 (0)	108 (9)	46.53	7.61	117.92	4.18
Sonication/disp.	80 (14)	0 (0)	20 (38)	240 (20)	0 (0)	0 (0)	82 (38)	30 (20)	0 (0)	60 (50)	0 (0)	71 (15)	0.65	17.81	3.22	4.90
Filtration/funct.	213 (36)	0 (0)	41 (63)	0 (0)	0 (0)	88 (100)	123 (75)	0 (0)	0 (0)	0 (0)	240 (25)	129 (28)	0.54	12.68	2.39	12.94
Packaging	240 (7)	145 (14)	0 (0)	0 (0)	0 (0)	0 (0)	180 (13)	80 (60)	60 (14)	0 (0)	180 (13)	132 (12)	77.48	5.70	146.94	4.31
QA/QC	0 (0)	60 (7)	60 (13)	20 (20)	0 (0)	0 (0)	10 (25)	98 (40)	0 (0)	0 (0)	250 (38)	111 (13)	0.38	22.42	2.08	5.53
Other lab work	0 (0)	0 (0)	55 (75)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	300 (29)	0 (0)	60 (25)	105 (13)	1.27	29.67	2.72	36.23
Drying wet GFN	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	60 (25)	50 (25)	0 (0)	180 (43)	0 (0)	0 (0)	117 (8)	31.50	47.47	138.38	38.09
Spray coating	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	18 (25)	0 (0)	0 (0)	0 (0)	0 (0)	118 (3)	0.76	1.26	1.34	1.09
Avg. = average; min = minutes; GM = geometric mean; GSD = geometric standard deviation; Resp. = respirable aerosol fraction; Inh. = inhalable aerosol fraction; disp. = dispersion; funct. = functionalization;																

Avg. = average; min = minutes; GM = geometric mean; GSD = geometric standard deviation; Resp. = respirable aerosol fraction; Inh. = inhalable aerosol fraction; disp. = dispersion; funct. = functionalization; GFN = Graphene Family of Nanomaterials; QA/QC = quality assurance and quality control; $\mu\text{g}/\text{m}^3$: micrograms per cubic meter.

*More detailed descriptions of each job task can be found in the [supplemental material](#).

[†]The percentage of participants who performed tasks was calculated by dividing the number of times the task was observed at each site by the total number of samples at the site, as seen in Table 2. Workers performed more than one task per day. Therefore, percentages by site may be >100%.

[‡]Personal full shift samples (not task-based samples) were used to calculate the geometric means. The geometric mean could be more reflective of another task performed on the same day.

and safety. First, the potential risks associated with GFN exposures will likely differ by specific material characteristics (e.g., size, carbon layers, impurities), which makes interpreting the growing amount of animal toxicity data challenging. Future study designs could consider using the graphene classification parameters recommended in *ISO/TS 9651:2025 Nanotechnologies—Classification Framework for Graphene-Related 2D Materials* (ISO 2025). Additionally, there has only been one human clinical research study to assess the potential effects of acute inhalation exposures to graphene oxide that found limited health effects (Andrews et al. 2024). While this finding is encouraging, epidemiological information is important to understanding the effects of chronic exposures to GFNs, which are more likely to be experienced by workers (Pelin et al. 2018). Finally, there are no current international OELs published by authoritative organizations to compare exposure sampling results and ensure the safe handling of GFN in workplaces.

Several OELs exist from authoritative organizations for bulk synthetic and natural graphite in the form of OSHA PELs, ACGIH[®] TLVs[®], and NIOSH RELs. These OELs range from 2 to 5 mg/m^3 at the respirable size fraction and 10 to 15 mg/m^3 at the inhalable size fraction (Supplemental Table S1). Graphite is a typical precursor material for a top-down production process of GFNs, which were commonly observed in this study. Multiple facilities were familiar with the OELs for graphite and acknowledged they were trying to maintain exposures below those levels. However, several samples collected from this study for EC were either over the action level (half of the OEL) or approached these OELs. Previous research has suggested that ultrafine (nano) particles may be more detrimental to human health when compared to similar bulk materials (Gupta and Xie 2018). This increase in potential toxicity can be attributed to deeper lung penetration and higher reactivity due to particle size and the increase in surface area (Bakand et al. 2012; Andrews et al. 2024).

Results from this study found that 28% and 10% of the TWA personal samples collected at the respirable size fraction exceeded the suggested OELs of 18 $\mu\text{g}/\text{m}^3$ by Lee et al. (2019) and 212 $\mu\text{g}/\text{m}^3$ by Spinazzè et al. (2019), respectively. Meanwhile, 38% of the respirable personal samples collected were above the suggested OEB of <10 $\mu\text{g}/\text{m}^3$ established by Niang et al. (2025). Similarly, results of personal samples in the inhalable size fraction of 49% and 10% exceeded the suggested OELs of 18 and 212 $\mu\text{g}/\text{m}^3$, respectively, while 53% of

these personal samples exceeded the suggested OEB of $<10 \mu\text{g}/\text{m}^3$.

The discrepancies between suggested exposure limits and exposures observed in this study further emphasize the importance of consensus exposure assessment methods and safety guidance for GFNs. Since there are no consensus exposure assessment methods for GFNs, Niang et al. (2025) suggested sampling for EC at the respirable size fraction due to adverse lung effects from animal toxicity data near the alveolar region. When feasible, collecting samples at both the inhalable and respirable size fractions can be informative, especially if little is known about the material and aerosol characteristics of the GFNs being sampled. A recent study by Evans et al. [forthcoming] examined the dustiness of several GFNs and graphite materials and found that the thoracic aerosol fraction constituted 56% of the inhalable fraction. Similarly, this study found the average respirable-to-inhalable ratio was 50% but varied by site, environmental conditions, and material. This information could be useful to retrospectively convert a value in case future OELs are developed, but the ratio may differ substantially by GFN type.

Additionally, collecting a sample for a confirmatory analysis by electron microscopy could also be useful if resources allow, but it may not always be necessary, especially if the occupational hygienist's professional judgment indicates that exposures are occurring and there are limited organic and elemental carbon interferences in the workplace.

The marginal model for inhalable EC demonstrated higher within-facility and within-worker correlations than the respirable EC model, which still exhibited moderate correlations. These findings underscore the suitability of marginal regression for capturing within-facility variability in clustered exposure data and within-worker variability in longitudinal exposure data. Additionally, the model for inhalable EC also found more significant parameter estimates, most notably for the facility-level determinants. A few of the job tasks were significantly associated with decreasing or increasing exposure potentials. Moreover, a subjective assessment of overall engineering control adequacy was found to be the strongest parameter estimate that increased the exposure potential. An analogous engineering control rating was used in a model to predict carbon nanotube exposures with similar results (Dahm et al. 2019). The variable was categorical and based upon observations with ratings of adequate (appeared to effectively control exposures and used consistently and properly by employees), semi-

adequate (many appeared to be effective however some were likely deficient and were not consistently or properly used by employees), and inadequate (lacked proper engineering controls and/or controls present likely did not effectively control exposures). This may suggest that professional judgment of engineering control adequacy could be useful in future models to assist in estimating occupational exposures. Especially since it has been shown that industrial hygienists who use professional judgment alone to make exposure judgments have a wide range of accuracy (Logan et al. 2009).

Limitations

This study should be considered cross-sectional in nature and may not be wholly representative of exposures that U.S. workers handling GFN may encounter. Additionally, the overall summary statistics for all sites and for several individual facilities are likely an underestimation of the true exposure, as some samples contained large amounts of EC that exhibited incomplete combustion during EC analysis. The standard deviations and geometric standard deviations indicated large variances in the exposures from facility-to-facility and for some job tasks. This was likely due to the wide range of job duties and exposure potentials at each facility. The job tasks in this study were assigned based on similar duties, but many facilities were performing similar tasks in different ways and with varying levels of controls. Finally, the summary statistics displayed in Tables 3 and 4 should be interpreted with caution since task-based samples were not collected. A participant likely performed multiple tasks throughout their workday that contributed to their overall exposure.

Conclusion

In summary, this is the first multi-site investigation conducted in the U.S. to describe the range of occupational exposures for various types of production and manufacturing facilities. There are currently no well-recognized OELs for GFNs, but samples collected from this study for EC were found to either be over the action level or approached OELs for bulk carbonaceous materials, such as graphite. This exposure assessment study, along with other recent studies, demonstrates the feasibility of using EC as a marker to assess GFN exposures and inform risk assessors and toxicologists on realistic exposure information and characteristics of GFNs. The findings underscore

the importance of standardized exposure assessment methods and the establishment of formal occupational exposure limits for the growing family of graphene materials to safeguard worker health.

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Disclaimer

The findings and conclusions in this report are those of the author(s) and do not necessarily represent the official position of the National Institute for Occupational Safety and Health (NIOSH), Centers for Disease Control and Prevention (CDC). Mention of any company or product does not constitute endorsement by NIOSH/CDC.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

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

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