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RECEIVED 09 May 2023

ACCEPTED 20 June 2023

PUBLISHED 05 July 2023

CITATION

Adeola AO, Duarte MP and Naccache R (2023), Microwave-assisted synthesis of carbon-based nanomaterials from biobased resources for water treatment applications: emerging trends and prospects.
Front. Carbon 2:1220021.
doi: 10.3389/frcarb.2023.1220021

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Microwave-assisted synthesis of carbon-based nanomaterials from biobased resources for water treatment applications: emerging trends and prospects

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Carbon-based nanomaterials have drawn significant interest as desirable nanomaterials and composites for the adsorptive removal of various classes of pollutants from water owing to their versatile physicochemical properties. The underlying sorption mechanisms serve as the bedrock for the development of carbonaceous adsorbents for various target pollutants. Microwave-assisted synthesis can be regarded as a recent and well-advanced technique for the development of carbon-based nanomaterials, and the use of biobased materials/wastes/residues conforms with the concept of green and sustainable chemistry. For advancements in carbon-based functional nanomaterials and their industrial/field applications, it is essential to fully comprehend the sorption performance and the selective/non-selective interaction processes between the contaminants and sorbents. In this regard, research on the development of carbon-based nanomaterials for the adsorption of chemical contaminants, both organic and inorganic, in water has made considerable strides as discussed in this review. However, there are still several fundamental hurdles associated with microwave-assisted chemical synthesis and commercial/industrial scale-up applications in nano-remediation. The challenges, benefits, and prospects for further research and development of carbon-based nanomaterials/nanocomposites for the purification of water are also discussed.

KEYWORDS

adsorption, carbon-based nanomaterials, chemical pollutants, microwave-assisted synthesis, water treatment

1 Introduction

Over 20 years ago, the concept of “green chemistry” emerged as a result of a shift in emphasis on material research with the move towards the use of waste materials and waste-reduction strategies. The chemical industry as a whole and scientists are directed to follow and promote sustainable and environmentally friendly pathways governed by the twelve principles of green chemistry (DeVierno Kreuder et al., 2017). The seventh principle buttresses the need for designing synthetic procedures with minimal negative impacts on the environment and without the use of hazardous reagents (Kharissova et al., 2019; Forbes, 2021). Thus, using renewable feedstocks and biodegradable raw or waste materials that are

typically rich in carbon, and are available from natural or biological sources, is a more acceptable approach for the environmentally friendly preparation of carbon-based materials from the bulk to the nanoscale.

The synthesis of carbon-based nanomaterials has been facilitated by enabling technologies such as hydrothermal, solvothermal, chemical vapor deposition, microwave-assisted synthesis, and other top-down/bottom-up techniques (Yan et al., 2016; Macairan et al., 2019; Adeola and Forbes, 2021a; Gulati et al., 2023). In comparison to other conventional methods, microwave-assisted synthesis of carbon-based nanomaterials is a non-invasive, easy, quick, clean, and eco-friendly process (Oladipo et al., 2018; Dutta et al., 2022a). The reaction is facilitated and accelerated by the microwave, frequently increasing relative yields. According to reports, microwave-assisted synthesis can circumvent synthesis-related challenges through energy efficiency, simplicity of experimental setup, adjustable process conditions, speedy reactions, and uniform heating/thermal processing. Microwave-assisted synthesis of carbon-based nanomaterials requires low growth temperatures and offers high surface areas, adsorption capacities, and purity while providing energy savings. As such, it presents a promising approach to addressing the challenges for industrial and municipal water treatment (Schwenke et al., 2015; Baghel et al., 2022).

Environmental pollution is unquestionably one of the major challenges facing civilization today. Every day, thousands of tons of dangerous chemicals are released into water bodies (Nassar et al., 2015; Dutta et al., 2019; Tong et al., 2022). Some dangerous contaminants that can be found in the aquatic environment include heavy metals, textile dyes, herbicides, surfactants, microplastics, hydrocarbons, pharmaceuticals, and personal care products, to name a few. To get rid of the contaminants in polluted water, new methods are constantly being researched. While developing carbon-based nanomaterials for environmental remediation, some major factors for consideration include target-specific identification, simplicity of design, affordable production, the toxicity of nanomaterial, biocompatibility, reusability, and the capacity for regeneration after usage. Therefore, new technologies are particularly intriguing because they may hold the key to addressing these challenges. As a result, numerous studies have concentrated on applying green chemistry concepts with the physicochemical surface modification of carbon-based nanomaterials to produce nanomaterials that can mitigate challenges faced during contaminant cleanup (Das et al., 2020; Dutta et al., 2022b; Mohapatra et al., 2023; Osterberg et al., 2023).

Scopus search revealed that over 62% of published data on carbon-based materials in the last decade have focused on design and application in water treatment, and well over 10,000 articles have been published on the subject (Dutta et al., 2022a). Thus, there is a need to constantly review the advances in this field of research, particularly the recent integration of microwave-assisted synthesis in carbon nanomaterials development. The current review assesses the development of advanced carbon nanomaterials (graphene, carbon nanotubes, and carbon dots) for water pollution remediation using microwave-assisted synthesis. The significant aspect of the review includes green nano-synthesis from waste/biobased resources using the microwave method and applications in water treatment, highlighting the merits, challenges, and future prospects.

2 Conventional synthesis techniques of carbon-based materials from biobased resources

Carbon-based nanomaterials have become an essential component of modern technology, with applications in fields such as electronics, environment, catalysis, energy storage, and drug delivery due to their unique qualities, such as high surface area, exceptional mechanical strength, and electrical conductivity (Fathy et al., 2020; Zakaria et al., 2022). Numerous methods are utilized to synthesize these nanomaterials, which can be classified into top-down and bottom-up approaches. The top-down approach consists in breaking down large carbon structures such as graphite, into smaller carbon nanostructures, while in the bottom-up approach, the carbon nanostructures are formed from atoms and molecules (Supplementary Figure S1–S3) (Baig et al., 2021; Abid et al., 2022). Depending on the nature of the precursor and process conditions, some conventional and microwave-assisted techniques can carry out bottom-up/top-down synthesis of carbon nanomaterials.

Arc discharge and laser ablation are among the most used top-down approaches for the synthesis of carbon nanomaterials. These were the earliest methods used for synthesizing fullerenes and carbon nanotubes (CNTs) (Kroto et al., 1985; Iijima, 1991; Arora and Sharma, 2014). In the arc discharge method, a high-voltage electric current is applied between two graphite electrodes in an inert environment/system. An electric arc is formed, generating a high temperature that is responsible for vaporizing the graphite electrodes. Then, the vapor condenses into CNTs or fullerenes (Arora and Sharma, 2014; Deng et al., 2016). On the other hand, the laser ablation technique utilizes a high-power laser to vaporize graphite under an inert atmosphere. The vapor is then carried by the inert gas and condensed into carbon nanomaterials (Deng et al., 2016; Aravind Kumar et al., 2020). Nonetheless, these techniques show several drawbacks including low yield for laser ablation, presence of impurities such as amorphous carbon and residual catalyst, limited control over size and shape, requirement of high temperatures, and consequently high energy consumption, in addition to costly setups. Thus, bottom-up approaches, including chemical vapor deposition (CVD), hydro/solvothermal, and microwave-assisted techniques are considered more promising for the production of carbon nanomaterials on a large scale due to their low cost, high control over size and shape as well as high material yields (Saputri et al., 2020; El-Khawaga et al., 2023).

CVD has been one of the conventional methods most explored for synthesizing different carbon nanomaterials owing to its simplicity and ability to produce high-quality materials (Wang et al., 2018; Fathy et al., 2020; Zakaria et al., 2022). It consists of the thermal decomposition of a carbon source under an inert atmosphere at a high temperature, in the presence of a metallic catalyst. As the carbon source decomposes, the carbon atoms deposit on the catalyst surface, and due to its low solubility in these metallic particles at high temperatures, precipitates are obtained and then carbon nanomaterials are formed (Deng et al., 2016). Although this conventional method is indeed capable of producing high-quality carbon-based nanomaterials, it relies mainly on carbon sources derived from fossil fuels for synthesizing these materials, such as ethylene, acetylene, benzene, methane, ethane, toluene, and xylene

(Goswami et al., 2021; Shi et al., 2023a). In this regard, there has been a rise in interest in a more sustainable and greener synthesis of carbon nanomaterials that focuses on the development of environmentally friendly and sustainable alternatives (Deng et al., 2016; Vivekanandhan et al., 2017; Fathy et al., 2020; Shi et al., 2023a). These alternatives focus on the use of non-toxic, renewable, biodegradable, and abundant precursors, such as honey (Mandani et al., 2017) and juices (Wang et al., 2022), biomass (Zhao et al., 2020), vegetable and animal oils (Duarte et al., 2022), industrial residues (Hu et al., 2019), and agricultural waste (Somanathan et al., 2015). Supplementary Table S1 provides a summary of biobased resources that have been utilized in the synthesis of carbon nanomaterials through conventional techniques.

Owing to its high yield, high purity, and the possibility of morphological and structural control, researchers have focused on developing a more sustainable CVD process. Thus, greener solvents and more environmentally friendly solid carbon sources have been employed as precursors to the synthesis of these nanomaterials (Manawi et al., 2018; Fathy et al., 2020). However, the evolution of toxic gaseous by-products during CVD remains a drawback (Manawi et al., 2018; Ijaz et al., 2020). In this way, pyrolysis, hydro/solvothermal and microwave-assisted techniques have attracted attention due to the non-necessity of the presence of a substrate for the growth of the nanomaterial, in addition to the possibility of using different carbon sources derived from biomass sources, renewable materials, and wastes. As a result, these approaches have been widely utilized in many parts of the world, including developing countries (Wang et al., 2018; Wang et al., 2020a; Devi et al., 2021).

The pyrolysis method consists of the thermal decomposition of a carbon source at a high temperature under an inert atmosphere and has been used extensively for synthesizing carbon dots (CDs) and graphene-based materials (Devi et al., 2021; Asif and Saha, 2023). In the hydro/solvothermal methods, the carbon source is converted into nanostructured carbon materials under high pressure and mild temperatures (<350°C) in a sealed vessel (Byrappa and Adschiri, 2007; Heidari et al., 2019; Gong et al., 2023). In the hydrothermal method, the reaction occurs in an aqueous medium, while in the solvothermal approach, different solvents such as ethanol, acetone, and DMF, are used as the reaction medium. Both methods can produce carbon-based nanomaterials with controlled size and morphology, depending on the synthesis parameters, such as temperature, time, and concentration of the carbon source and solvent (Wang et al., 2020a; Huo et al., 2023).

These methods with the inclusion of microwave technology have proven to be excellent to obtain carbon nanomaterials from renewable and natural sources and residues, rendering this process more sustainable, green, and economically viable. In addition, they provide an alternative to recycling and reusing some residues, not only transforming them into value-added products but also addressing some environmental issues associated with the disposal of these materials. More information about various top-down and bottom-up synthesis approaches for carbon-based nanomaterials, including their merits and demerits are available in recent reviews by Abid et al. (Abid et al., 2022), El-Khawaga et al. (El-Khawaga et al., 2023), Gong et al. (Gong et al., 2023) and Huo et al. (Huo et al., 2023). According to these reports, these methods present some limitations such as the requirements for

high temperatures and pressures, long reaction times, and difficulty in measuring, understanding, and controlling the synthesis/growth process of the nanomaterials (Abid et al., 2022; Huo et al., 2023). Hence, researchers have sought the development of microwave-assisted synthesis methods to address these shortcomings while achieving faster results through better-controlled reactions and product formation (Omoriyekomwan et al., 2019; Omoriyekomwan et al., 2021; Vignesh et al., 2022).

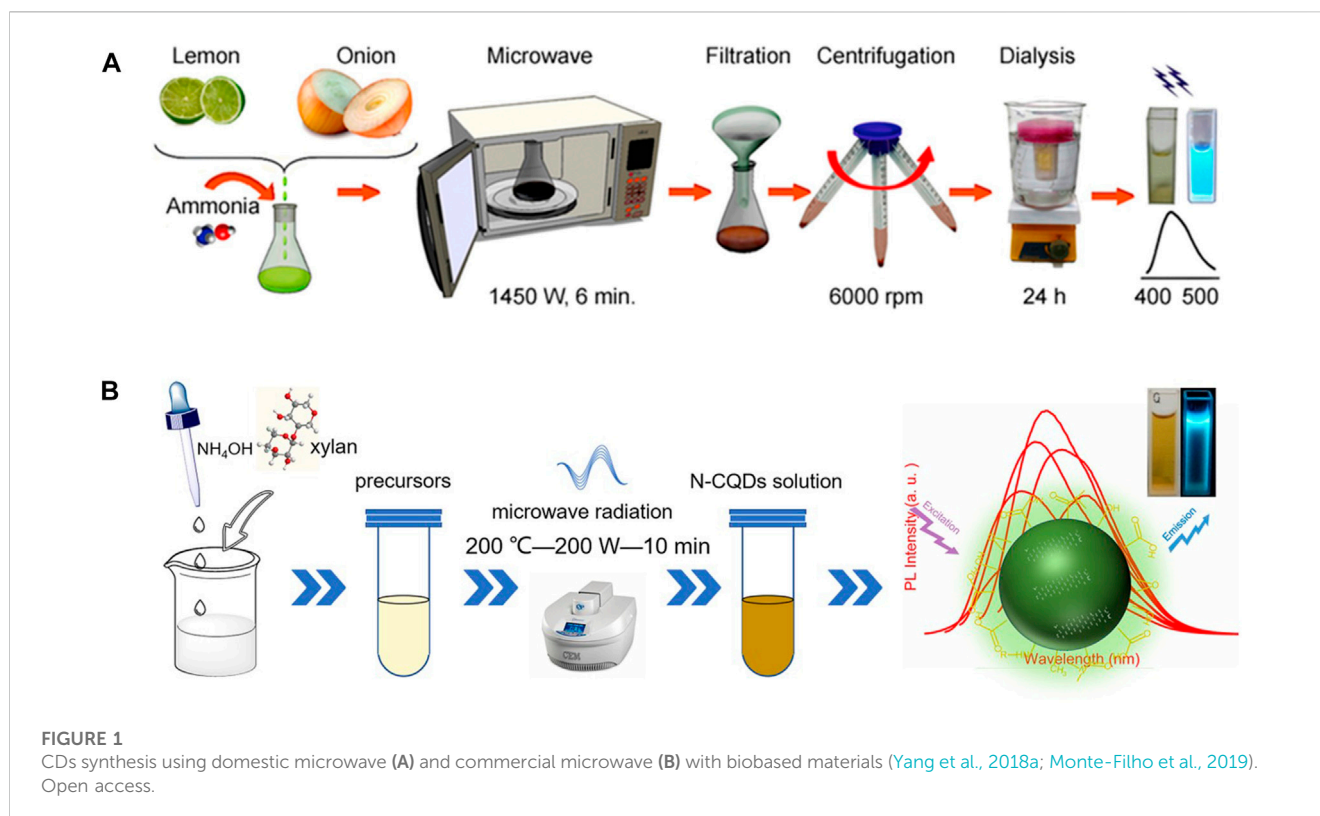
3 Microwave irradiation

Gedye et al., in 1986, and Mingos and Baghurst in 1991, reported an unprecedentedly quick organic reaction under microwave irradiation (Gedye et al., 1986; Mingos and Baghurst, 1991). These were two of the earliest reports on the use of microwaves in chemical synthesis. Prior to these reports, Randall and Booth invented the magnetron, a device that generated fixed microwave frequencies, ushering in the era of microwave-assisted chemical processes (Cole and Cole, 1941; Kaul, 1989). As soon as scientists realized the magnetron's amazing capacity for rapidly heating water, they began to investigate the mechanism at play, and subsequently, its potential for domestic, laboratory, and commercial applications (de Medeiros et al., 2019; Singh et al., 2019).

Domestic microwaves were used during the development phase of microwave-assisted chemical synthesis in research laboratories and unfortunately are still in use in developing countries across the globe (Monte-Filho et al., 2019). Although advanced microwave reactors currently exist (Figure 1), the use of more affordable kitchen microwaves for research has not been completely phased out. Some prestigious publishers reject manuscripts that include household ovens as heating sources. The reason why more advanced microwave equipment has been developed includes safety features offered by modern microwave reactors for chemical synthesis. Furthermore, the main benefit of using microwaves for chemical synthesis is superheating in an air-tight reaction vessel that can withstand high temperatures and pressures, since the Arrhenius rule allows for significantly faster reaction times.

Advanced microwave reactors function as highly practical autoclaves that can swiftly and effectively heat reaction mixtures to 300°C and 80 bar while allowing the reaction medium to stay sealed throughout the duration of the reaction. In contrast, domestic microwave ovens do not offer reaction parameter control and it is almost impossible to monitor the temperature and pressure of the reaction in real-time, with IR sensors for reaction temperature control, pressure sensors to keep track of the reaction pressure in the closed vessels, and a magnetic stirrer to facilitate agitation (Figure 2). Electromagnetic radiation between 0.3 and 300 GHz is referred to as microwaves. To prevent interference with telecommunications equipment, most typical microwave equipment emits electromagnetic radiation at 2.45 GHz.

The type of material being heated by microwaves and the amount of interaction between the sample and the radiation are key determining factors in how a reaction proceeds since conductors, dielectric materials, and insulators all have different characteristics. Following exposure of a material to microwave radiation, three different types of mechanisms have been described in the literature (Mishra and Sharma, 2016; Kahraman



et al., 2020), namely reflection, absorption, and transmittance. Metals and other electrical conductors act as microwave radiation reflectors (Rafael Zamorano et al., 2019) while PTFE and quartz are two substances that permit microwave radiation transmission. Because insulators do not absorb microwave radiation, they are unable to be heated via this technology (Motasemi and Ani, 2012). In what concerns the reaction medium, or the solvent, for these reactions, polar liquids are excellent at absorbing microwaves. As such, they heat up quickly following microwave irradiation because of dipolar interactions and ionic conduction (Hu et al., 2021).

Polar molecules' capacity to absorb microwave radiation and transform it into heat through a process known as dielectric heating is the key to the effectiveness and success of microwave-assisted chemical reactions (de Medeiros et al., 2019). When exposed to microwave radiation, polar molecules with electrical dipole moments rotate continuously in an effort to align themselves with the electric field. This phenomenon is caused by dielectric polarization and conduction loss. Through dielectric loss and molecular friction, the molecules reverse direction when the field alternates, thereby generating heat. The loss factor ($\tan \delta$) is a measure of a solvent's capacity to transform electromagnetic energy from microwaves into heat. According to Table 1, solvents are frequently categorized as strong, medium, or weak microwave absorbers. A solvent's microwave heating process is quicker and more effective with a higher value of $\tan \delta$. A low $\tan \delta$ value, on the other hand, suggests ineffective and slow heating operation under microwave irradiation (de Medeiros et al., 2019).

Figure 3A demonstrates how microwave heating differs from traditional heating in that the center of the material is heated to a greater temperature than the medium allowing for a quick and

efficient reaction. In contrast, under conventional heating, the vessel must be heated first followed by heating of the medium and eventually the heating of the sample. Under microwave heating, the molecules' dipoles attempt to align with the applied electric field during irradiation in microwave-assisted synthesis (Figure 3B). Higher frequency field oscillations cause the dipoles to continually realign with the alternating electric field, which generates heat through molecular friction and dielectric loss. No heating occurs if the dipole doesn't have time to realign (at much higher frequencies), or if it reorients too quickly (during low-frequency irradiation) (Sun et al., 2016; Jiang et al., 2023). Ionic conduction can also contribute to the heating mechanism. Under the influence of the microwave energy field, charged particles that are dissolved into the sample, in ionic form, bounce back and forth and subsequently produce heat by interacting with nearby molecules or atoms (Gabriel et al., 1998; Singh et al., 2019). This technique has been used to prepare different types of nanomaterials including metallic nanoparticles (Zhu and Chen, 2014; Tsuji, 2017), semiconductor quantum dots (Xuan et al., 2015), metal-organic nanomaterials (Thi Dang et al., 2020), polymeric nanomaterials (An et al., 2006), carbon-based nanomaterials (de Medeiros et al., 2019; Thakur et al., 2023), and many others.

4 Microwave-assisted synthesis of carbon-based nanomaterials

Attention has been drawn to the synthesis of next-generation nano-carbon materials, including heterostructures and nanocomposites with improved performance and



FIGURE 2

Microwave reactors with cutting-edge design features include the following: CEM Discover SP with a single-mode cavity (top left). Microwave Research and Applications BP-210 processing MW with reaction temperatures up to 1800°C (top right). CEM Liberty Blue with a critical reagent delivery system (bottom left). CEM MARS6 with a high throughput capacity and a fiber-optic temperature sensor (Bottom right). Adapted from (Chin et al., 2021). Open access.

multifunctionality (Speranza, 2021). To determine the feasibility of prolonged utilization of microwave technology for developing advanced carbon-based nanomaterials like CDs, CNTs, and graphene-based materials (GBMs), the availability of carbon sources is crucial. These nanomaterials have so far been produced using carbon sources including citric acid, acetylene, benzene, methane, toluene, xylene, and other natural hydrocarbon resources (Liu et al., 2011; de Medeiros et al., 2019; Macairan et al., 2019; Sajid et al., 2022; Gulati et al., 2023; Liu et al., 2023). These hydrocarbons contribute to the release of CO₂ which may leak into the atmosphere, leading to global warming. Methane and other volatile hydrocarbons are greenhouse gases that act as more powerful heat-traps than CO₂ and contribute more to climate change than CO₂, which they release following complete combustion. Furthermore, these hydrocarbons are fast depleting and valuable carbon-based nanomaterials may experience a shortage issue in the future due to their reliance on these non-renewable resources. Hence, a more practical, affordable, and environmentally benign method and resources are required to sustainably prepare advanced carbon-based nanomaterials.

Microwave irradiation, which causes polymerization and carbonization, has been used to prepare carbon-based nanomaterials (Alarfaj et al., 2018). Carbon-based nanomaterials absorb electromagnetic energy during the microwave irradiation process and transform it into heat energy (Yang et al., 2018a). The molecular rotation of polar solvent molecules, caused by the interaction of electrical dipole moment with microwave energy, produces thermal energy. During microwave-assisted heating, electromagnetic radiation transfers thermal energy to the carbon precursor (Singh et al., 2019).

The preparation of carbon-based nanomaterials from biobased resources is essentially a top-down approach, which involves the break down of large carbonaceous materials, such as agricultural wastes, into nano-sized carbon structures (de Medeiros et al., 2019; Thakur et al., 2023). The microwave-assisted synthesis is effective for producing carbon nanomaterials and has the added benefits of rapid volumetric heating, high reaction rates, precise control over size and shape of nanomaterials by reaction parameter adjustment, and energy efficiency. Additionally, the homogeneous nucleation and growth conditions created by the homogenous heating of the

TABLE 1 Various solvents and their loss factor value ($\tan \delta$) were determined at 293 K (de Medeiros et al., 2019).

Solvents	$\tan \delta$
<i>Low ($\tan \delta$ less than 0.1)</i>	
Hexane	0.020
Toluene	0.040
Dichloromethane	0.042
Tetrahydrofuran	0.047
Acetone	0.054
Ethyl acetate	0.059
Acetonitrile	0.062
Chloroform	0.091
<i>Medium ($\tan \delta$ range between 0.1 and 0.5)</i>	
Chlorobenzene	0.101
Water	0.123
1,2-Dichloroethane	0.127
dimethylformamide	0.191
acetic acid	0.174
N-Methyl-2-pyrrolidone	0.275
Dichlorobenzene	0.280
2-Butanol	0.447
<i>High ($\tan \delta$ greater than 0.5)</i>	
1-Butanol	0.571
Nitrobenzene	0.589
Methanol	0.659
formic acid	0.722
2-Propanol	0.799
dimethyl sulfoxide	0.825
ethanol	0.941
Ethylene glycol	1.350

precursors during microwave synthesis reduce thermal gradients and result in the development of nanomaterials with a uniform size distribution (Bacon et al., 2014).

4.1 CNTs and doped CNTs

In recent years, multiple studies have targeted the production of CNTs from waste materials to counter the more expensive and time-consuming synthesis procedures that were typically used (Liu et al., 2019). CNTs (1D nanocarbon) may now be easily grown using microwave radiation, opening up a new method for their versatile and economical synthesis. Both domestic and commercial microwave ovens have been utilized as practical plasma reactors for the quick, easy, energy-efficient, and solvent-free growth of

CNTs (Jašek et al., 2006; Baghel et al., 2022). In addition to enabling the rapid growth of high-density CNTs in a matter of seconds, the unique heating mechanism of microwaves also eliminates the requirement for an expensive boiler and a source of combustible gaseous carbon (Liu et al., 2019). Under microwave irradiation, very localized heating, close to the catalyst nanoparticles, can be observed (Chin et al., 2021).

It has been reported that rice husk was successfully used to synthesize CNTs using microwave-induced plasma irradiation with a 600 W and 2.45 GHz power and frequency, respectively (Asnawi et al., 2018). The procedure was carried out at a 750°C temperature for 40 min. The pressure in the reaction tube decreased from 3 to 1 mbar as CNTs grew. The biosynthesized CNTs revealed a twisted and web-like network structure and Raman spectra showed that the I_D/I_G ratio was 1.013. The high I_D/I_G ratio between the D and G band intensities is an indication of the lower quality of the sp^2 hybridized carbon nanomaterial (Figure 4). The lower quality may be due to structural defects and the amorphous nature of the carbon-based nanomaterial. This experiment demonstrates the possible use of waste biomass as a precursor for microwave-based CNTs synthesis. In addition to turning waste biomass into valuable carbon nanostructures, this would help address the environmental problems brought on by the vast amount of agricultural biomass.

Similarly, Hidalgo-Oporto et al. reported the use of biochar derived from wheat straw, oats hull, rapeseed cake, and hazelnut for the preparation of CNTs (Hidalgo et al., 2019). A carbon-rich porous material is produced when biomass is subjected to a thermochemical process and can serve as a precursor for CNTs synthesis (Figure 5). A combination of biochar derived from agricultural waste and ferrocene was heated with a microwave to create CNTs at 400°C and 600°C. The findings showed that the biomass pyrolysis temperature had an impact on the physicochemical characteristics of CNTs. Higher CNTs concentration and lower hydrodynamic diameter were obtained by 600°C biochar. Moreover, CNTs prepared from biochar derived from wheat straw and hazelnut hulls had a higher degree of wall graphitization, indicating superior CNTs quality.

In a more recent study, Omoriyekomwan et al. reported the use of palm kernel shells to synthesize CNTs using the microwave pyrolysis process at a low temperature of 600°C (Omoriyekomwan et al., 2019). They employed the palm kernel shell (PKS) and used two isolation procedures, namely alkaline-acid and formic acid/acetic acid, to separate the components of PKS, cellulose, and lignin. They claimed that the recovery of monosaccharides in the cellulose's pyrolysis volatiles, which served as a substantial carbon source, promoted CNTs growth. Moreover, the functional group, organic matrix, flaws, and structural quality were all improved. The mechanism of CNTs growth has been postulated to involve the self-extrusion of monosaccharides- and hydrocarbons-rich volatiles during cellulose pyrolysis proceeded by condensation and re-solidification of volatiles on the softened cellulose particles at high temperatures (Figure 6). Although a high ash content in biobased materials or agricultural wastes can operate as a catalyst to accelerate the formation of CNTs, high-purity precursors are still necessary for CNTs synthesis.

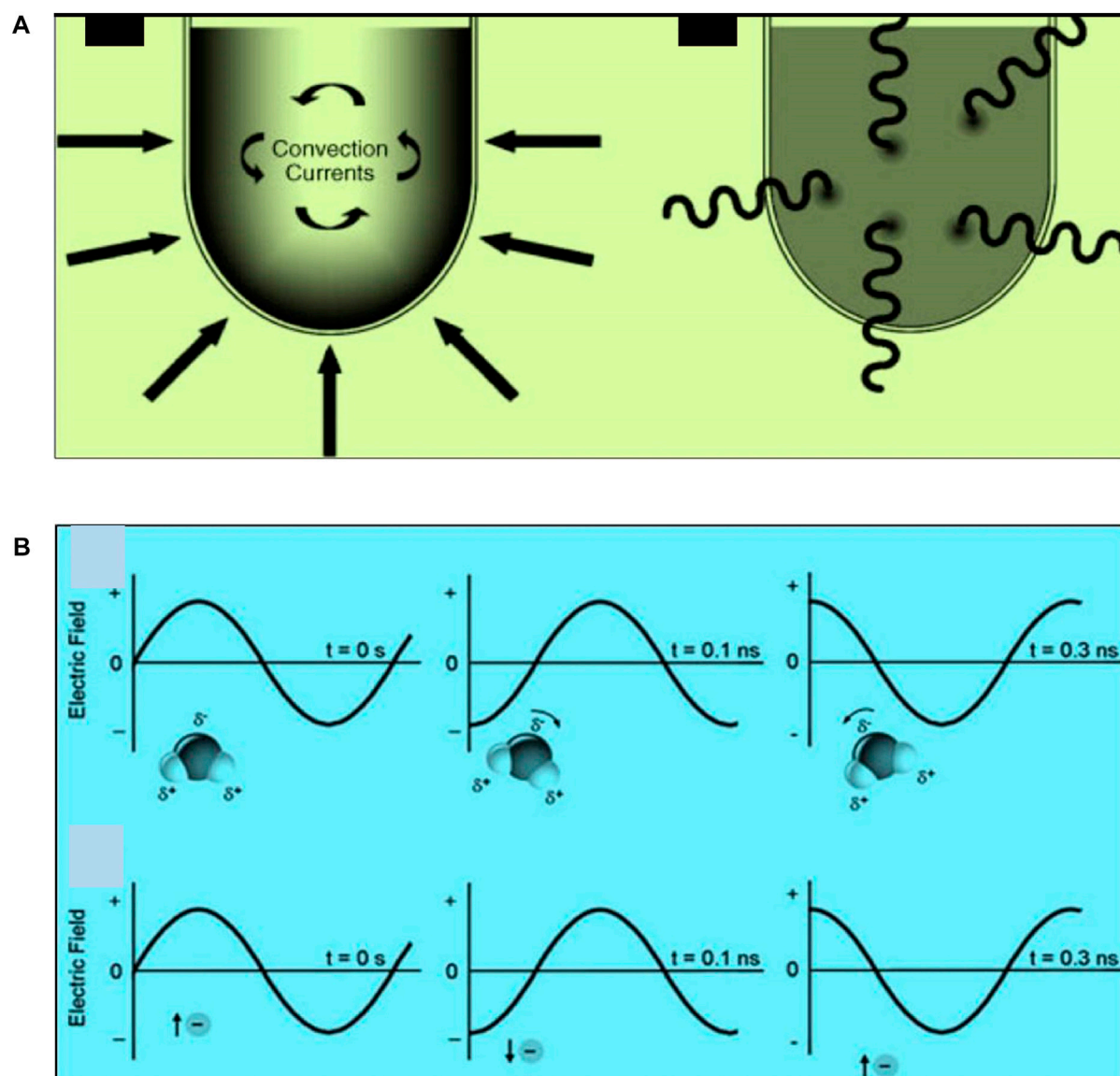


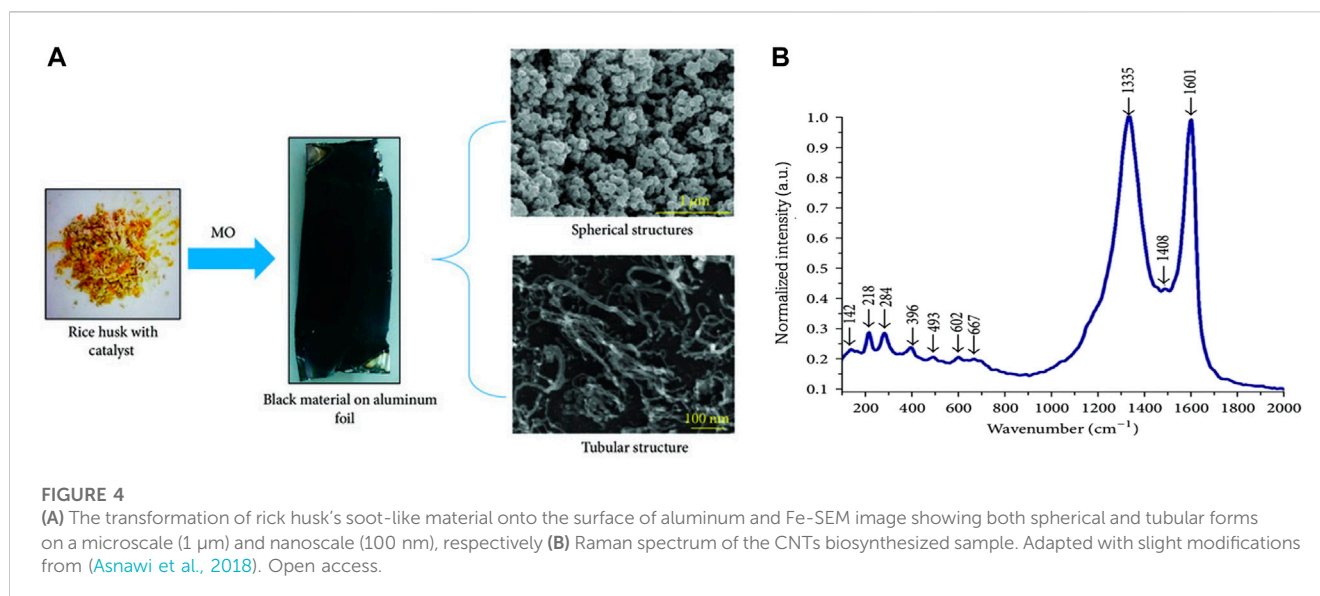
FIGURE 3

(A) Comparison of conventional heating and microwave heating (B) Mechanisms of microwave energy transfers are dipole polarization (top) and ionic conduction (down). Adapted with minor amendments and permission from Wiley (Kappe et al., 2012).

It is noteworthy to mention that while most synthesis protocols for CNTs required hours, the microwave-assisted technique generates CNTs in minutes and seconds. Interestingly, Zhan et al. revealed that in 20–40 s, CNTs can be synthesized using a domestic microwave, howbeit from iron, copper, molybdenum, steel wires, and fibers were used as precursors and not biobased materials. The as-synthesized CNTs had a diameter range of 10–120 nm (Zhan et al., 2017). CNT-based composites have been synthesized in 150 s with the aid of a microwave; they include palladium-graphene oxide-based CNTs (Kumar et al., 2017), nitrogen-iron doped CNTs (Kang et al., 2017) (Figure 7), ruthenium-based single-walled carbon nanotubes (SWCNTs-Ru) (Hemraj-Benny et al., 2020), CNTs/Fe₃O₄ (Li et al., 2023a), etc. Other precursors have been reported in Literature, such as graphite (Algadri et al., 2017; Guo et al., 2017), carbon fiber (Bajpai and

Wagner, 2015), Mxene aerogel (Zheng et al., 2019), graphite and cobalt acetate powder (Ortega-Cervantez et al., 2016), as well as poly lactic-co-glycolic acid (PLGA) coated with polypyrrole (PPy) (Xie et al., 2014).

The affinity of carbon-based nanomaterials for specific types of contaminants in aqueous media is impacted by the different functionalities/moieties on their surface, or their hybridization/combination with other nanomaterials/molecules, which also improves their adsorption efficiency. A few chemical modification techniques and their benefits are summarized in Table 2. Functional moieties may contain oxygen-, nitrogen-, and sulfur-containing groups depending on the type of heteroatom that binds to the carbon in the nanomaterial. Through selective design with appropriate functional groups, the adsorption selectivity of contaminants may improve (Sajid et al., 2022). Typically, related



functional groups are introduced to carbon compounds by oxidation, nitrogenation, and sulfonation.

4.1.1 CNTs-based materials as adsorbents for pollution remediation

Adsorbents composed of CNTs are commonly used to remove chemical pollutants from water and wastewater (Gupta and Saleh, 2013; Yu et al., 2016; Gusain et al., 2020). Due to their large specific surface areas, tunable surface characteristics, porosities, hydrophobicity, hollow and layered architectures, many internal and external binding sites, π -conjugation, as well as simplicity of chemical activation and functionalization, CNTs exhibit excellent adsorption capabilities (Barrejón and Prato, 2022; Pyrzynska, 2023). CNTs-based adsorbents can interact with inorganic and organic pollutants through a variety of mechanisms, including complexation, ion exchange, electrostatic interaction, covalent bonding, precipitation, and van der Waal's interaction, among others (Figure 8) (Adeola and Forbes, 2021a; Sajid et al., 2022). Chemical bonds between organic compounds and CNTs have occasionally been described as a mode of interaction (Indrawirawan et al., 2015; Duarte et al., 2022).

The removal of PFAS and other persistent and endocrine-disrupting pollutants with CNTs-based materials has been reported (Vu and Wu, 2022). As the C-F chain length increased, more perfluorinated compounds (PFCs) with the same functional group were able to bind to MWCNTs. The removal efficiency of the CNTs with hydroxyl and carboxyl groups was significantly lower than that of the pristine CNTs (Deng et al., 2012a). Hydrophobic interactions dominated the mechanism of adsorption. With an Langmuir maximum adsorption capacity of 46.2 mg/g, magnetic MWCNTs coated with chitosan were utilized to treat water containing Bisphenol A (BPA), another endocrine-disrupting substance (Mohammadi et al., 2020). The residual BPA concentration was determined using high-performance liquid chromatography (HPLC).

Pharmaceuticals present in different aquatic systems can cause serious health problems in people, including inflammation, vomiting, headaches, dizziness, and other minor and major

systemic dysfunction. The main problem is that these pollutants cannot be fully removed or degraded by traditional wastewater treatment facilities (Adeola and Forbes, 2021b; Ihsanullah et al., 2022). A nanocomposite of CNTs and alumina was employed to adsorb carbamazepine and sodium diclofenac from water. The residues of diclofenac and carbamazepine were analyzed using UV-vis spectrophotometer. The CNTs were used for adsorption while the alumina improved the hybrid's physicochemical properties and regenerability (Wei et al., 2013). In several additional instances, SWCNTs and MWCNTs were used to remove amoxicillin and SWCNTs displayed much better adsorption capacity for the selected pharmaceutical drug (Mohammadi et al., 2015; Balarak et al., 2016). Residual amoxicillin was analysed using HPLC. In a related investigation, batch experiments were used to examine the ciprofloxacin's adsorption behavior from an aqueous solution onto MWCNTs/ Al_2O_3 (Balarak and McKay, 2021). The spontaneity and endothermic nature of the sorption process were shown by thermodynamic studies. According to the Dubinin-Radushkevich isotherm model, the estimated mean free energy, which ranged between 0.316 and 0.707 kJ/mol, supports a physisorption process. It was established that MWCNTs/ Al_2O_3 are potentially useful for the elimination of antibiotics from contaminated water.

Metals are a major part of the group of contaminants that have been researched extensively in CNTs-based adsorption, as they cause numerous health problems in people (such as lead poisoning), animals, and aquatic life (Sajid et al., 2018; Ihsanullah et al., 2020; Sulaiman et al., 2020). The causes of metal contamination are numerous and include both anthropogenic and natural processes and must be removed from the water to pre-determined acceptable limits (Feng et al., 2018; Ore and Adeola, 2021; Adeola et al., 2022a). Mercury was eliminated using MWCNTs that were loaded with CuS. Because of the high surface area of MWCNTs, the amount of CuS dispersed on the surface of MWCNTs increased. CuS addition to the adsorbent was intended to improve mercury affinity. Moreover, the copper can amalgamate with Hg (Wang et al., 2020b). Thus, the CuS/MWCNTs surface purportedly adsorbed mercury as stable HgS, and the

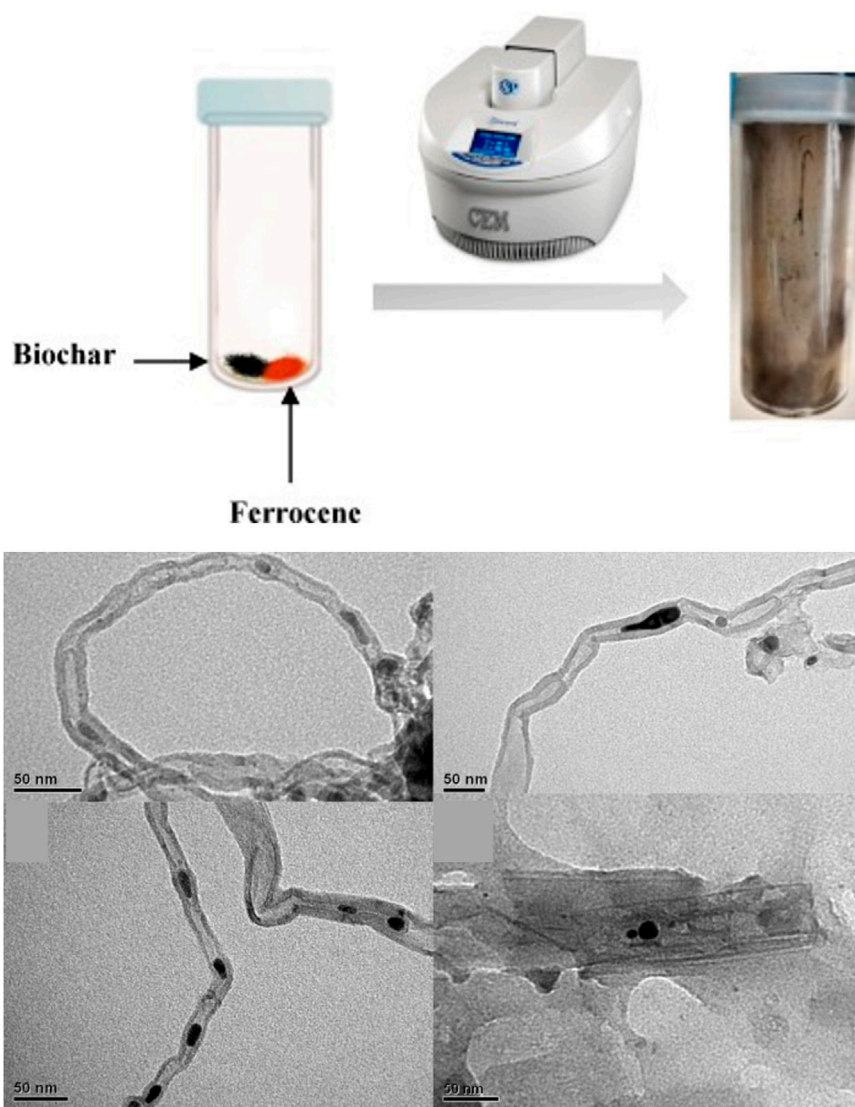


FIGURE 5

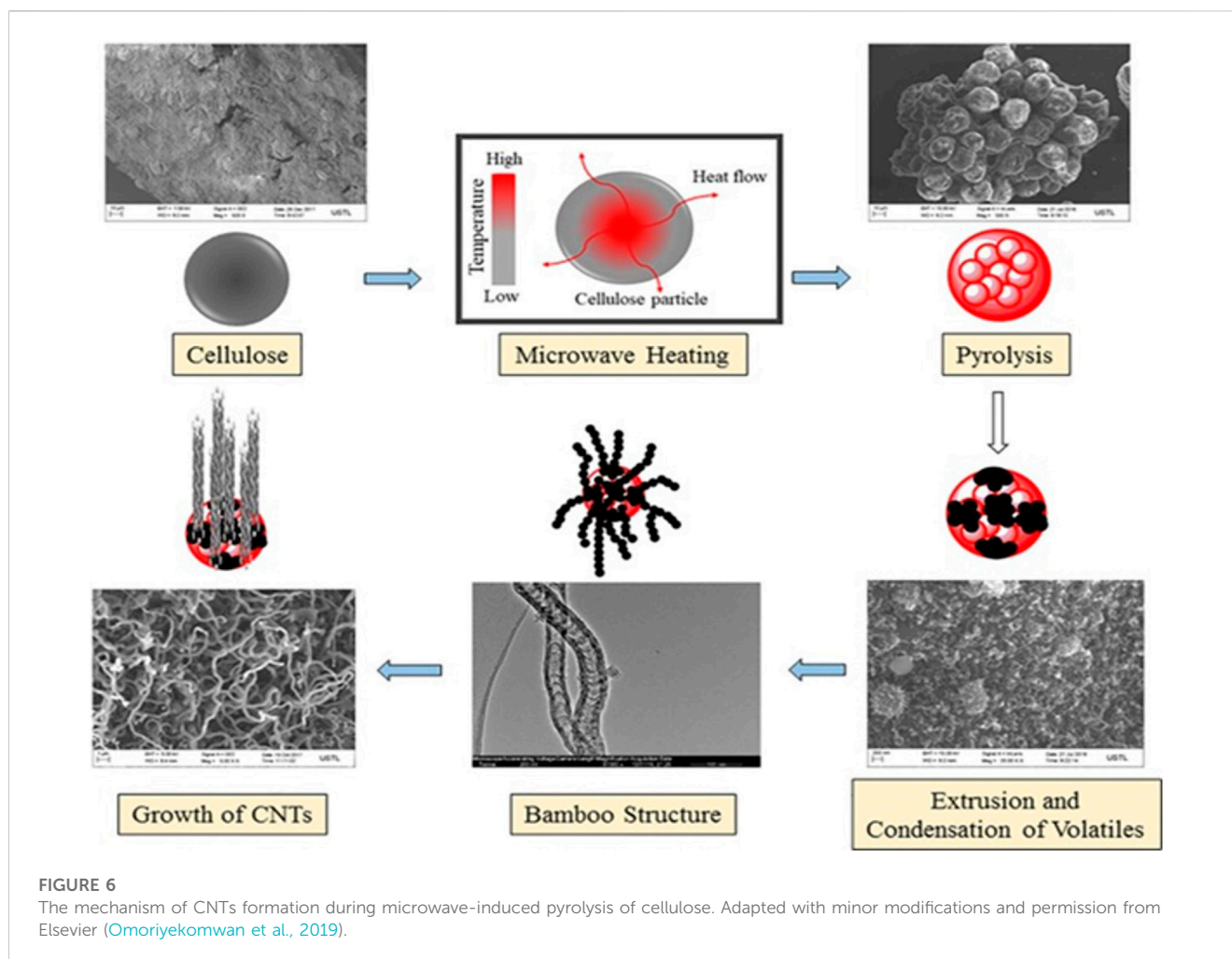
Illustration of microwave irradiation as a tool to develop CNTs from a biochar/ferrocene combination (top). TEM images of CNTs prepared from biochar derived from oat hull, wheat straw, hazelnut hull, and rapeseed cake, respectively (bottom). Adapted with minor modifications and permission from Elsevier (Hidalgo et al., 2019).

sorption process was controlled or facilitated via chemisorption (Liu et al., 2022a). The continuous mercury analyzer (VM3000 Mercury Vapor Monitor) was used to analyze mercury. Recently, a mechanical method (ultra-sonification) and a chemical method (surfactant inclusion) were coupled to stabilize CNTs. The elimination of Cu(II), Ni(II), Pb(II), and Zn(II) ions was accomplished using these CNTs, and residual metal ions were analyzed using Atomic Absorption Spectrometer (AAS) Perkin Elmer 3,300. It was discovered that electronegativity and atomic radius played roles in competitive adsorption. Compared to Ni(II) and Zn(II) ions, a higher percentage of Pb(II) and Cu(II) ions were eliminated (Oliveira et al., 2021). The lead ion is the most electronegative metal with the highest atomic radii among the other cations, which favors its adsorption onto the MWCNTs structure via attractive and hydrodynamic forces.

Table 3 presents a list of CNTs-based nanomaterials that have been used to sorb organic and inorganic compounds from polluted aqueous media. It is noteworthy to mention that various forms of CNTs-based materials have been reported to have Langmuir adsorption capacities as high as 416 mg/g (Cu) for metals, 679.6 mg/g (Rhodamine B) for dyes, 227.3 mg/g (Chipton) for pesticides, 221.2 mg/g (Meloxicam) for pharmaceuticals, and 46.2 mg/g (Bisphenol A) for other compounds, which affirms the affinity of CNTs and their composites for these various classes of compounds.

4.2 Graphene and graphene-based materials

Graphene-based materials (GBMs) have attracted much scientific curiosity in the last decade due to their exceptional



electronic, mechanical, and optical properties which have made them adaptable in several fields of scientific innovations (Sang et al., 2019; Adeola and Forbes, 2021a). Graphene (2D nanocarbon) and its derivatives can be functionalized for use in a wide range of fields, including environmental remediation, medicine, and electronics (Khenfouch et al., 2016; Liu et al., 2020; Papi, 2021; Hashmi et al., 2022). Figure 9 reflects the most common types/forms of graphenic materials, they are graphite, CNTs, reduced graphene oxide, and fullerenes, among others. Because of their unique features, all these graphene-based materials are very different in comparison to one another. Biomass and agricultural waste are useful carbon sources that have been used to produce graphite, which in turn serves as precursor for graphene-based materials (Vivekanandhan et al., 2017; Shi et al., 2023a).

To incorporate hydroxyl, carboxyl, and carbonyl groups on the surface of graphite, harsh chemicals like sulfuric, nitric, and phosphoric acids are typically used in chemical reactions to produce graphene-based materials. When pre-treated graphite is exposed to high temperatures, gases evolve and form graphitic layers (Hernandez et al., 2008). The acidic precursors can intercalate between the graphitic layers because they have two hydroxyl groups on either side. In addition to the emission of toxic gases, the separation of the graphitic layers is frequently haphazard and

ineffective. The microwave-assisted synthesis may address these challenges via rapid heating and better control of the reduction and exfoliation of graphitic layers. The microwave-assisted synthesis of GBMs can be achieved under three categories: 1) chemical reduction of graphite with the aid of a microwave; 2) thermal reduction of graphite with a microwave; 3) simultaneous thermal exfoliation and thermal reduction of graphite with a microwave (Al-Hazmi et al., 2015; Xie et al., 2019).

Microwave-assisted chemical reduction of graphite involves the use of a reducing agent such as hydrazine hydrate under microwave irradiation to expedite the process (Hassan et al., 2009). Hassan et al. examined the reduction process using Raman spectroscopy and observed an I_D/I_G ratio between 0.1 and 0.12 following microwave treatment for 60 s, demonstrating a high reduction degree even after such a brief treatment time. The enhanced thermal stability showed no appreciable mass loss up to 750 °C. Another study by Kumar et al. reported that a 4-h microwave exposure was comparable to a 48-h conventional reaction technique used for the synthesis of GBMs (Kumar et al., 2015). The traditional heating method led to the formation of defects in the graphene basal plane as a result of the evolution of the oxygen functional groups during reduction (Li et al., 2010). This can be addressed by the microwave-assisted thermal reduction approach either with a commercial microwave or a

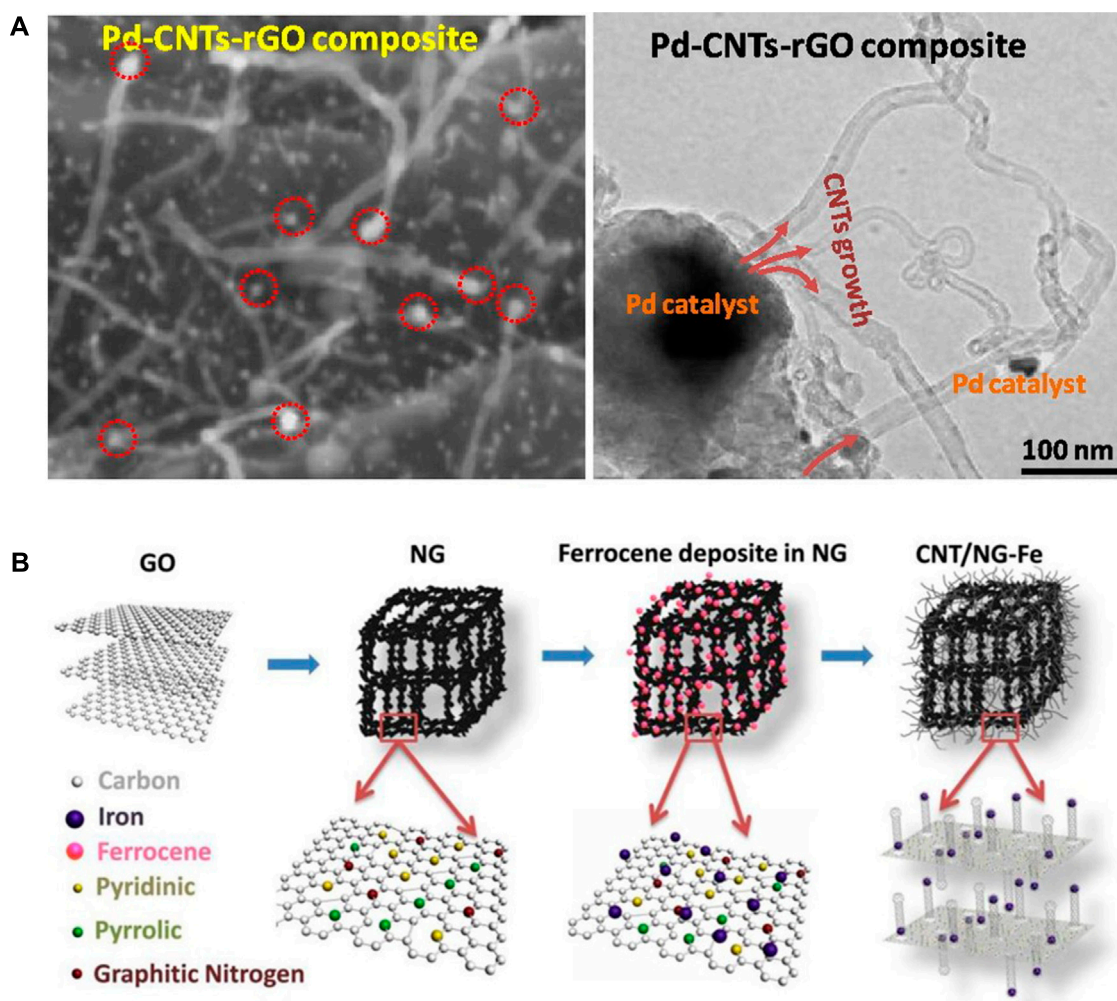


FIGURE 7
 (A) Hierarchical palladium-CNTs-reduced graphene oxide composite produced with microwave (B) 3D nitrogen-incorporated reduced graphene oxide (NG)/iron oxide (CNTs/NG-Fe) CNTs nanocomposite. Adapted with permission from Elsevier (Kang et al., 2017; Kumar et al., 2017).

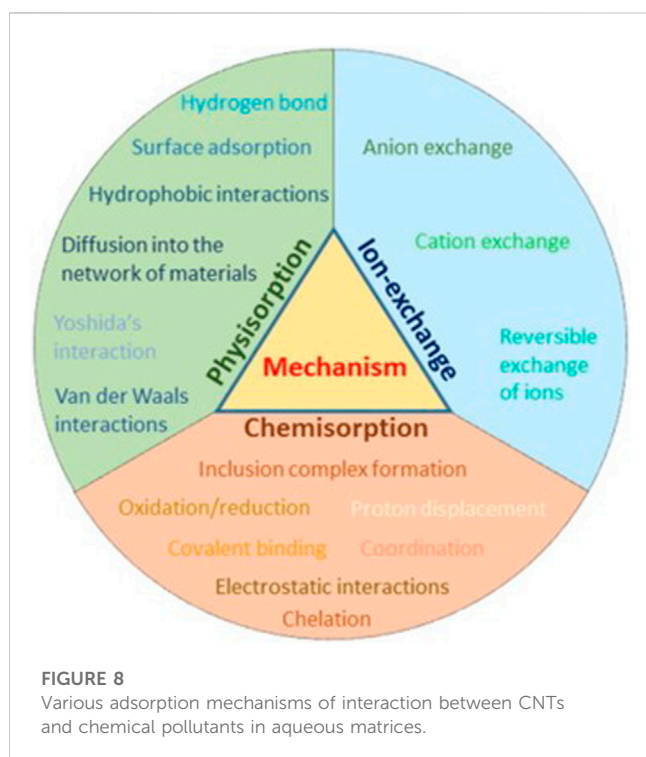
microwave-plasma reactor (Figure 10). Without using any reducing agents or solvents, microwave irradiation's superior thermal action may quickly convert GO into graphene (Voiry et al., 2016; Jiang et al., 2018; Wan et al., 2018). This method offers a viable opportunity to produce graphene on a huge scale. To clarify the underlying process of GBM microwave reduction, more research on the dielectric characteristics of GBMs at various microwave frequencies is necessary.

Due to its poor microwave absorption capacity, graphite oxide itself may not be heated to a temperature that will effectively cause its exfoliation and reduction under microwave irradiation. It is not possible to produce rGO with the same level of purity by simultaneous exfoliation and reduction as it is through the thermal reduction of graphene oxides (Zhu et al., 2010a; Zhu et al., 2010b; Pokharel et al., 2014). However, the reduction degree could be significantly increased when paired with a reducing environment (i.e., H_2) or pretreatment with powerful reducing reagents ($NaBH_4$).

Microwave treatment has been used to synthesize a graphene-based material from used spent tea leaves (Abbas et al., 2020). Due to their distinctive optical characteristics, size, and quantum confinement, graphene quantum dots (GQDs) are zero-dimensional fluorescent materials that have garnered considerable attention. However, one of the associated challenges is their low reaction yields, which contribute to high production costs and restrict their large-scale applications. Using microwave treatment, spent tea was used as a low-cost, environmentally friendly, and renewable biomass resource for the high-yield synthesis of GQDs. The synthesis method uses oxidative cutting and pyrolysis under microwave irradiation to generate GQDs, with an over 84% yield recorded. The size distribution of GQDs was extremely narrow, with an average size of 1.6 ± 0.55 nm. The energy band gap and luminescence emission in the high-energy area increased as a result of the reduced size (Abbas et al., 2020). Similarly, microwave plasma irradiation was used to prepare graphene-based materials from rice husks (Wang et al., 2015a). Plasma and

TABLE 2 Several approaches for modifying carbon nanomaterials in microwave-assisted synthesis.

Modification	Methods	Merits/Effects
Oxidation	Acid treatment; treatment oxidizing agents (KMnO ₄); reacting with a combination of acids and other oxidizing agents	Incorporates alcoholic, carboxylic, and ketonic functional groups
		Improved adsorption performance, particularly for metals
		Metal impurities in the nanomaterial are also removed
		Allows for further functionalization and enhances hydrophilicity for aqueous dispersion
Doping with MOFs, cyclodextrin	Formation of nanocomposite; sol-gel; solvothermal	Carbon nanostructure acts as a support for other adsorbents
		Synergistic effect on decontamination of polluted water
Activation	Treatment with bases, acids, or a mixture of bases or acids	Removal of anionic and cationic contaminants
Magnetic nanomaterials	CVD; sol-gel; pyrolysis; solvothermal/hydrothermal	Easy recovery and regeneration of carbon nanomaterials after adsorption
		Quick dispersion in solution
Doping with polymers	<i>In-situ</i> polymerization; mixing in solution; melt blending	Improved affinity for organics
		Enhanced chemical and thermal stability
Surface functionalization	Nitrogenation; sulfonation	Enhanced selectivity with desired moieties such as thiols for mercury or amines for metals



microwave heating was used to irradiate the powdered rice husks to a temperature of approximately 200 °C while biogas/pyrolysis gas (often CH₄ or C₂H₄) was present (Figures 10, 11). The pressure and temperature utilized in the microwave setup had a significant impact on the dissociation of rice husks and the production of graphitic carbons (Worasuwannarak et al., 2007; Wang et al., 2011; Chen et al., 2014).

4.2.1 Graphene-based materials as adsorbents for pollution remediation

Chemical pollutants are produced by industrial processes, agricultural technology, and synthetic substances like pharmaceuticals and personal care items. Among the main pollutants released by numerous industries are hazardous heavy metals (e.g., Cd, Cu, Hg, Ni, Pb), toxic gases, and organics (e.g., dyes, pesticides, pharmaceuticals) (Alengebawy et al., 2021; Mitra et al., 2022). Industrial growth has a considerable detrimental impact on water resources due to the excessive use of these chemical compounds, which are exceedingly dangerous.

When fossil fuels, coal, and biomass are burnt, polycyclic hydrocarbons are produced that are classified as PAHs (Adeola et al., 2022b). These chemicals are extremely hazardous because they affect human organs leading to tumors, cancer, and chronic cardiovascular problems (Hussain et al., 2018; Song et al., 2021). In addition to PAHs, dyes, surfactants, industrial additives, and pesticides are also worrisome because they interfere with the endocrine system and nerve cells. Another category of organic pollutants, including phenols, bisphenol A, biphenyls, and phenyls, threatens aquatic flora and fauna and poses a health threat to humans (Mohammadi et al., 2020; Tavengwa et al., 2021). Even at very low concentrations (e.g., part per million and part per billion), these organic contaminants are recalcitrant and persistent in the environment. Therefore, it is crucial to remove both organic and inorganic pollutants before the release of wastewater into the environment. Hence the need to develop efficient materials for their removal from aqueous matrices.

There has been extensive research into using graphene and its composites to treat water that contains organic and inorganic contaminants, as indicated in Table 4. To address the United Nations' sustainable development goals (SDG six in particular) and the shortcomings of existing wastewater systems, graphene,

TABLE 3 CNTs-based sorbents for removal of organic and inorganic pollutants in water.

Sorbent	Sorbate	Langmuir adsorption capacity (mg/g)	Kinetics/isotherm	Optimum process parameters [pH; time]	References
<i>Elaeis guineensis</i> /polyvinyl alcohol/CNTs	Dyes: Malachite blue	-	Pseudo-second order (PSO)/Langmuir (monolayer adsorption)	11; 300 min	Zulfiqar et al. (2022)
PANI-MWCNTs	Malachite orange	149	PSO/Langmuir and Freundlich models	7; 90 min	Pete et al. (2021)
4-Aminosalicylic Acid Functionalized MWCNTs	Crystal Violet Dye	440	PSO	5.5; 1 min	Saxena et al. (2021a)
Amine functionalized MWCNTs- porphyrin conjugate	Malachite blue	-	PSO/Freundlich (multilayer adsorption)	5–7; 70 min	Saleh et al. (2021)
Single wall carbon nanotubes (SWCNTs)	Reactive yellow dye 15 and Reactive yellow dye 42	179.9 and 156.1	PSO/Langmuir and Freundlich models	3; 5 and 42 min	Naghizadeh et al. (2022)
SWCNTs	Acid Blue 92	86.91	Pseudo-second order (PSO)/Langmuir	3; 75 min	Balarak et al. (2021)
Sulfonic acid-functionalized-CNTs	Malachite blue	236.5	PSO/Freundlich (multilayer adsorption)	pH 12	Lei et al. (2021)
Hydrogel nanocomposite	Malachite blue	647	PSO/Linear Redlich-Peterson	11; 40 min	Mallakpour and Tabesh (2021)
Multiwall carbon nanotubes (MWCNTs)	Violet 2 R	76.92	PSO/multiple models	4; 120 min	Abualnaja et al. (2021a)
CNTs/MgO/CuFe ₂ O ₄	Methyl violet dye (MVD) and Nile blue dye (NBD)	>35	PSO/Freundlich (MVD), Langmuir (NBD)	8; 50 min	Foroutan et al. (2021)
Poly (Acrylonitrile-co-Styrene)/MWCNTs	Methyl orange	121.95	PSO/multiple models	3; 120 min	Abualnaja et al. (2021b)
WS ₂ /Fe ₃ O ₄ /CNTs-nanocomposite	Amaranth and brilliant blue	174.8 and 166.7	PSO/Langmuir models	3; 5 min	Arabkhani et al. (2021)
Nano-cobalt wrapped by nitrogen-doped CNTs	Rhodamine B	679.56	PSO/Langmuir models	4; 60 min	Yang et al. (2021)
Asparagine functionalized MWCNTs	Malachite green and blue	637 and 500	PSO/Langmuir models	4 and 6; 3 and 5 min s	Saxena et al. (2021b)
Alginate/f-CNTs-CD MFA hydrogel composite beads	Malachite blue	10.26	PSO/Langmuir models	10; 48 h	Mallakpour et al. (2021)
MWCNTs and Ox-MWCNTs	Herbicide/Pesticides: Diuron	39.59 and 48.60	PSO/Freundlich, Langmuir and Polanyi–Manes	Basic and Neutral; 60 min	Deng et al. (2012b)
MWCNTs from plastic waste	Diuron	40.375	PSO/Temkin, Hill, and Koble–Corrigan	240 min	Deokar et al. (2017)
UiO ₆₆ -NH ₂ @MPCA	Chipton	227.3	PSO/Langmuir models	pH 4	Liang et al. (2021)
ZIF-8 and magnetic MWCNTs	Eight Organophosphorus pesticides	2.18–3.89	-	4; 15 min	Liu et al. (2018)
MWCNTs	Malathion	-	-	7/30 min	Dehghani et al. (2017)
Carbon dot modified-magnetic CNTs	Pharmaceuticals: Carbamazepine	65	-	pH 7	Deng et al. (2019)
Polyaniline/MWCNTs	Meloxicam	221.2	PSO/Elovich and Langmuir-Freundlich	2/6 min	Dutra et al. (2018)
Granular CNTs/Alumina	Diclofenac and Carbamazepine	106.5 and 157.4 μmol/g	Langmuir	4/72 h	Wei et al. (2013)
Acid modified-MWCNTs	Diclofenac	24	PSO/Freundlich	7/60 min	Hu and Cheng (2015)

(Continued on following page)

TABLE 3 (Continued) CNTs-based sorbents for removal of organic and inorganic pollutants in water.

Sorbent	Sorbate	Langmuir adsorption capacity (mg/g)	Kinetics/isotherm	Optimum process parameters [pH; time]	References
MWCNTs	Amoxicillin	22.9	Langmuir	4.6/40 min	Mohammadi et al. (2015)
SWCNTs	Amoxicillin	122.8	PSO/Langmuir	7/45 min	
Acid treated MWCNTs	Other organics: trihalomethanes	0.92–2.41	Freundlich-Langmuir	3–7/150–180 min	Lu et al. (2005)
MWCNTs	CHCl ₃ , CHCl ₂ Br, CHClBr ₂ , CHBr ₃	10.98, 6.85, 6.57, 5.95	PSO/Sips	5–7/45 min	Dehghani et al. (2016)
Magnetic MWCNTs/SiO ₂ /CS	BPA	46.2	PSO/Langmuir	6.2/76 min	Mohammadi et al. (2020)
MWCNTs-KOH@NiNPs	Metals: Pb(II), As(V), Cd(II)	81.0, 440.9 and 415.8	PSO/Langmuir	5.5/30 min	Egboziuba et al. (2022)
As-MWCNTs; OX-MWCNTs	Cu(II) and Zn(II)	364 and 347; 416 and 411	PSO/Langmuir	6/30 min	Egboziuba and Abdulkareem (2021)
β-CD@Fe ₃ O ₄ /MWCNTs	Ni(II)	103	PSO/Langmuir	6/50 min	Lin et al. (2021)
CS/MWCNTs/Fe	Cr(VI)	119	PSO/Langmuir	pH 4	Aslam et al. (2021)
MWCNTs	Pb(II) and Ni(II)	215 and 230	PSO/Langmuir	5.8/60min	Egboziuba et al. (2021)
Waste cooking palm oil (WCPO)-CNTs	Cu(II)	29.22	Langmuir	pH 7	Abu Bakar et al. (2021)
PAAm/FMWCNTs	Cu(II)	320–385	PSO/Langmuir	5/90 min	Abo-Zahra et al. (2022)
Polyvinyl formaldehyde/MWCNTs foam	Pb(II)	3.4	PSO/Langmuir	5/60 min	yosef et al. (2020)
MWCNTs-HAP	Pb(II), Cd(II), Cu(II)	-	PSO/Langmuir	4–6/120 min	Li et al. (2021)

and its derivatives can be combined with a variety of metallic or non-metallic components to create innovative composites that increase the efficiency of removing pollutants in water or wastewater. To effectively treat wastewater, graphene, and its composites may be modified by eliminating, or adding functional groups and facilitating the formation of polymeric membranes or nanocomposites (Wu et al., 2011; Verma et al., 2022a). The process of doping and/or functionalizing/passivation can reportedly amplify the inherent graphene features and add additional stability for enhanced performance or multifunctional activity (Adeola et al., 2021a; Singh et al., 2022). It is noteworthy to highlight that adsorption capacities as high as 1,119 mg/g (Pb) for metals, 2000.1 mg/g (MB) for dyes, 1,667 mg/g (Malathion) for pesticides, 796.8 mg/g (Doxycycline) for pharmaceuticals and 766.58 mg/g (Naph) for aromatic compounds have been reported for various forms of carbon-based materials (Table 4). The removal performance of graphene-based materials is often influenced by the physicochemical properties of the sorbent and sorbate, as well as the solution chemistry encompassing pH, temperature (thermodynamics), salinity, and other process parameters. The adsorption capacities reported for graphene-based materials for different classes of pollutants were generally higher than many of those obtained for CNTs (Tables 3, 4), suggesting that structure and

atomic configurations may also play a role in the binding mechanism and capacity of various carbon-based nanomaterials/nanocomposites.

4.3 CDs and nanocomposites

CDs are zero-dimensional carbon nanomaterials with diameters smaller than 10 nm and have been touted to have minimal toxicity and excellent photostability (de Medeiros et al., 2019; Behi et al., 2022). The majority of CDs include sp² and sp³ hybridization, which are often similar to those in crystalline graphite but lack structural identity (Georgakilas et al., 2015). CDs be prepared as hydrophilic, hydrophobic, and amphiphilic, based on the intended applications (de Medeiros et al., 2019). However, for the adsorption of chemical pollutants in aqueous matrices, hydrophobic CDs with high surface area and porosity are most desirable. Thus, the utilization of relatively water-insoluble carbon, nitrogen, and oxygen precursors is necessary for the preparation of hydrophobic CDs. In addition, CDs and other carbon-based nanomaterials must be recoverable, regenerable, and reusable from a green and sustainable chemistry point of view.

Regarding hydrophobic carbon dot-based nanomaterials for water treatment applications, researchers have reported several

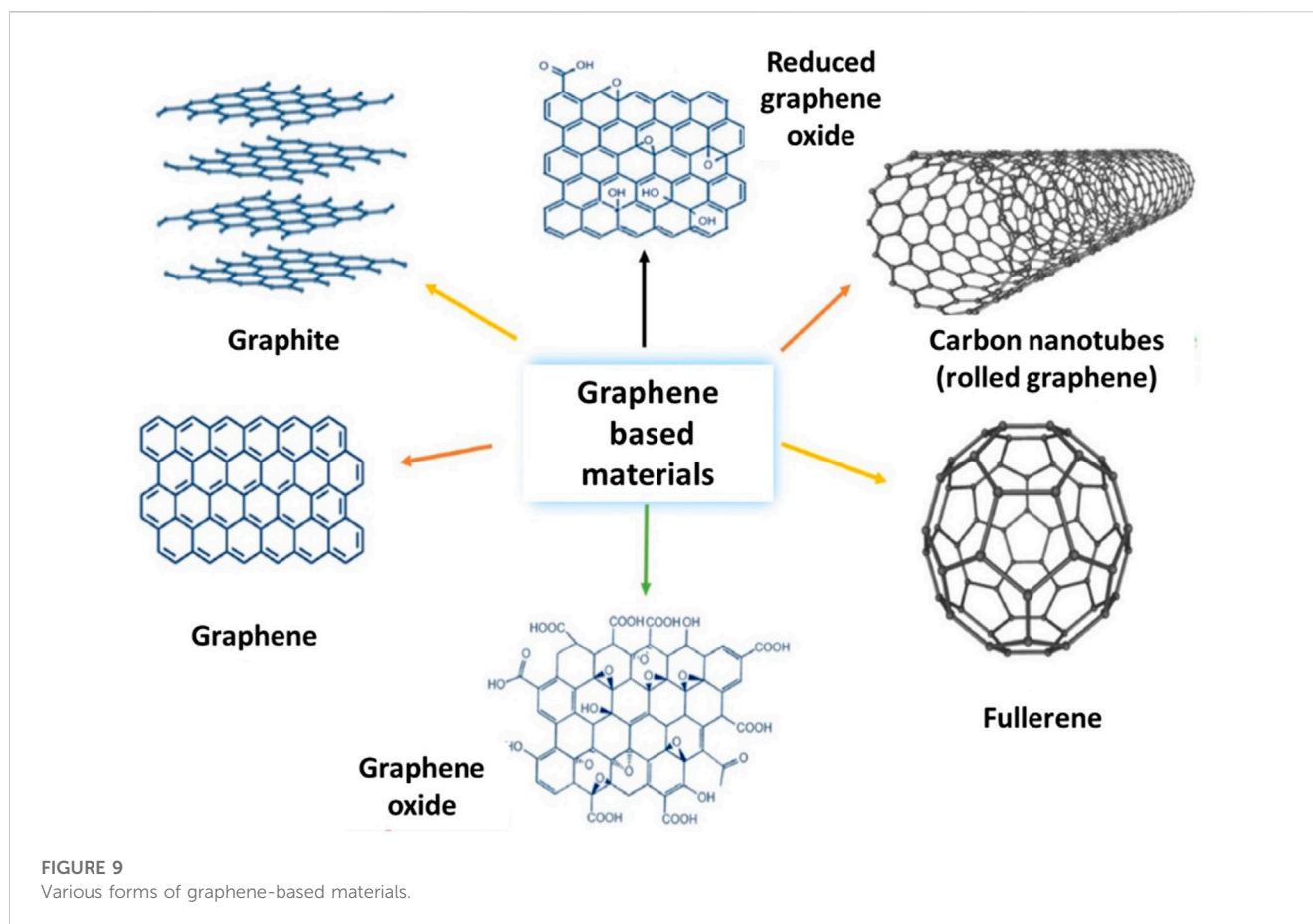


FIGURE 9
Various forms of graphene-based materials.

pristine CDs and composites, i.e., CDs (Yahaya Pudza et al., 2020), CD-modified magnetic nanotubes (Deng et al., 2019), hydrophobic nitrogen-doped CDs (Shi et al., 2023b), carbon quantum dots (Tohamy et al., 2023), and hexadecylamine-functionalized GQDs (Kubheka et al., 2022), among others. CDs and carbon quantum dots have been synthesized using green and sustainable precursors such as agricultural wastes and carbohydrates including cellulose (Yahaya Pudza et al., 2020; da Silva Souza et al., 2018), biowaste lignin (Shi et al., 2019), lemon and onion (Monte-Filho et al., 2019), wood soot (Zhong et al., 2018), dried leaf (Joshi et al., 2018), grass (Sabet and Mahdavi, 2019), broccoli (Arumugam and Kim, 2018), pomelo fruit (Ramar et al., 2018), denature milk (Athika et al., 2019), to name a few. CDs have been used as adsorbents for the removal of both organic and inorganic pollutants in water as summarized in Table 5. For instance, they have been used to monitor dangerous substances in effluents discharged from various chemical industries and adsorbed uranium, cadmium, and benzopyrene from real water samples (Li et al., 2018; Rahmanian et al., 2018; Huang et al., 2019c). Doping carbon quantum dots with nitrogen reportedly enhanced the adsorption of metal ions from wastewater due to their high specific surface area (Tohamy et al., 2023). In comparison to CNTs and graphene-based materials, CDs have not been as widely applied as adsorbents, however functionalized hydrophobic CD-based composites can equally be used as adsorbents and in the fabrication of membranes.

Periodic mesoporous organosilica embedded with CDs has been developed for the elimination of 2,4-dichlorophenol and heavy

metal ions Hg(II), Cu(II), and Pb(II) by Wang et al. (Wang et al., 2015b). The material has a bilayer of CDs, 2D hexagonal mesostructure, high specific surface area, and pore size ($\sim 468.46 \text{ m}^2/\text{g}$ and $\sim 5.50 \text{ nm}$). The $n-\pi$ electron donor-acceptor interaction between O- and N-containing moieties in mesoporous organosilica and the benzene ring in 2,4-dichlorophenol facilitated enhanced adsorption of 2,4-dichlorophenol, while the electrostatic interaction and complex formation between metal ions and amide groups help to increase the efficiency of metal ions removal from contaminated water (Wang et al., 2015b).

Perumal et al. reported the synthesis of nitrogen-doped CDs (N-CDs) and hybrid-spherical-shaped hydrogel particles (CGCDs) from Red Malus floribunda fruits (Perumal et al., 2022). The synthesized CGCDs were utilized as sorbents for the removal of heavy metals from water. The sizes of the CD-based materials ranged from 20 to 300 μm . Approximately $\sim 72\%$ and 99% of Hg(II) was sorbed by CGCDs in single metal ion and multiple metal ion systems. Furthermore, Hg(II), Cd(II), Pb(II), and Cr(III) recorded over 70% removal efficiency in multiple systems by CGCDs (Perumal et al., 2022). This suggests that CD-based materials may serve as a suitable tool for the simultaneous removal of heavy metals from industrial wastewater. In comparison to CNTs and graphene-based materials, CDs have not been as widely applied as adsorbents, however, functionalized hydrophobic CD-based composite can be equally used as adsorbent materials and in the fabrication of membranes.

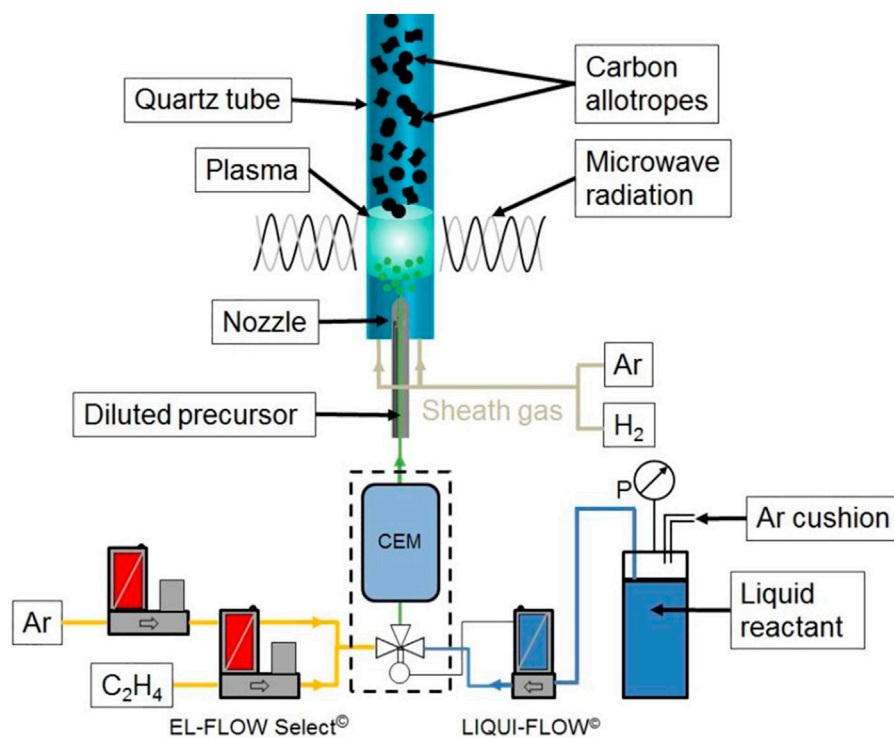


FIGURE 10
Microwave-plasma reactor including reactants supplies and metering. Adapted from (Fortugno et al., 2022). Open access.

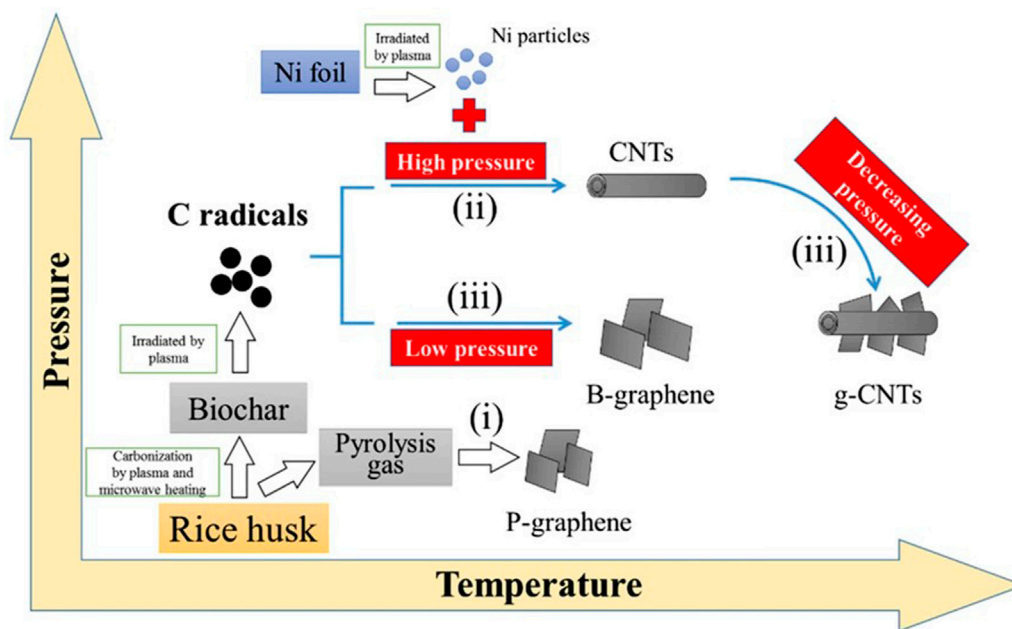


FIGURE 11
A plausible microwave plasma irradiation growth model for graphene, CNTs, and g-CNTs from rice husks Adapted with permission from Elsevier (Wang et al., 2015a).

TABLE 4 Graphene-based sorbents for removal of organic and inorganic pollutants in water.

Adsorbent	Target pollutant	Langmuir adsorption capacity (mg/g)	References
Graphene oxide/ZrO(OH) ₂	Metals: Arsenic (III and V)	84.89; 95.15	Luo et al. (2013a)
Graphene oxide/ferric hydroxide	Arsenic (V)	23.78	Zhang et al. (2010)
Graphene oxide/TiO ₂	Cadmium (III)	65.6	Lee and Yang (2012)
Graphene/c-MWCNTs	Mercury (II)	93.3	Sui et al. (2012)
Graphene oxide/poly (amidoamine)	Zinc (II)	0.2023 mmol/g	Yuan et al. (2013)
Graphene oxide	Copper (II)	117.5	Wu et al. (2012)
Graphene nanoshell/Fe ₃ O ₄	Chromium (VI); Lead (II); Mercury (II); Cadmium (II); Nickel (II)	17.29; 27.95; 23.03; 27.83; 22.07	Guo et al. (2014)
Graphene oxide encapsulated polyvinyl alcohol/sodium alginate	Copper (II); Uranium (II)	247.16; 403.78	Yi et al. (2018)
Graphene oxide passivated with ethylenediamine	Lead	479.0	Madadrang et al. (2012)
Graphene/SiO ₂	Lead (II)	113.6	Hao et al. (2012)
Graphene oxide/magnetic chitosan	Lead (II)	76.94	Fan et al. (2013)
Graphene oxide	Iron (III)	133.3	Han et al. (2010)
Graphene oxide	Gold (III); Palladium (II); Platinum (IV)	108.342; 80.775; 71.378	Liu et al. (2013)
Graphene oxide/Magnetic fungal hyphal	Nickel (II); Cobalt (II)	97.44; 104.34	Chen et al. (2021)
Graphene oxide/MOF/polymer	Arsenic(V)	180.0	Kong et al. (2022)
Graphene oxide	Copper (II); Zinc (II); Cadmium (II); Lead (II)	294.0; 345.0; 530.0; 1,119.0	Sitko et al. (2013)
Graphene oxide/magnetic chitosan	Lead (II)	112.35	Samuel et al. (2018)
Graphene oxide/Nanoscale Zero-Valent Iron (nZVI)-magnetic Fe ₃ O ₄	Chromium (VI)	101.0	Lv et al. (2014)
Reduced graphene oxide/Nanoscale Zero-Valent Iron (nZVI)	Arsenic (III and VI)	35.83; 29.04	Wang et al. (2014)
Graphene/Fe ₃ O ₄	Arsenic (III and VI)	0.452; 0.385	Guo et al. (2015)
Graphene	Europium (III) Thorium (IV)	150.0; 220.0	Huang et al. (2019a)
Graphene oxide/chitosan	Gold (III); Lead (II)	1,076.6; 216.92	Liu et al. (2012)
Graphene oxide/polyaniline nanofibers	Zinc (II)	297.62	Ramezanzadeh et al. (2018)
Graphene oxide	Uranium (VI)	299.0	Li et al. (2012)
Reduced graphene oxide/Magnetic dithiocarbamate	Copper (II); Cadmium (II); Lead (II); Mercury (II)	113.64; 116.28; 147.06; 181.82	Fu and Huang (2018)
Graphene/Fe ₃ O ₄	Selenium (IV and VI)	45.57; 27.12	Fu et al. (2014)
Graphene/Iron-chitosan	Chromium (VI)	83.8	Shan et al. (2021)
Graphene sheets	Arsenic (III and VI); Sodium	121.97; 141.92; 138.79	Mishra and Ramaprabhu (2011)
Graphene oxide/ZnO	Arsenic (III)	8.17	Singh et al. (2022)
Graphene oxide/thiol-Fe-Mn composite	Mercury (II)	233.17	Huang et al. (2019b)
Graphene oxide/manganese ferrite	Arsenic(V)	102.0	Shahrin et al. (2018)
Graphene oxide/PVC/p-Phenylenediamine	Lead (II)	44.8	Khan et al. (2021)
Graphene oxide biopolymer	Lead (II)	504.0	Bloor et al. (2021)
Graphene oxide/ultrathin carbon layer encapsulated magnetite nanoparticles	Silver(I); Lead (II); Chromium (VI); Aluminum (III)	62.9; 125.8; 158.2; 173.9	Wang et al. (2021)

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TABLE 4 (Continued) Graphene-based sorbents for removal of organic and inorganic pollutants in water.

Adsorbent	Target pollutant	Langmuir adsorption capacity (mg/g)	References
Reduced graphene oxide/Polyacrylonitrile	Mercury (II)	325.732	Awad et al. (2021)
Graphene oxide/poly-cyanoguanidine	Mercury (II)	174.7	Lin et al. (2022a)
graphene oxide/EDTA/chitosan	Mercury (II); Copper (II)	324.0; 130.0	Verma et al. (2022b)
Graphene oxide-dicationic ionic liquid composite	Chromium (VI)	271.08	Shang et al. (2021)
Reduced graphene oxide/Fe-Al layered double hydroxide/sodium alginate	Arsenic(V)	190.84	Nithya Priya et al. (2021)
Graphene/MOF	Copper (II)	343.49	Eltaweil et al. (2021)
Graphene oxide/PVC	Strontium (II)	20.07	Huo et al. (2021a)
Graphene oxide/PVA/MnO ₂	Cobalt (II); Strontium (II)	60.3; 26.8	Huo et al. (2021b)
Modified magnetic graphene oxide	Dyes: Acid red	524.2	Tang et al. (2018)
Reduced graphene oxide	Malachite green	476.2	Gupta and Khatri (2017)
Graphene oxide/PD	Methylene blue	2000.1	Dong et al. (2014)
Graphene oxide/ionic liquid-modified	Methylene blue	448.0	Gupta et al. (2022)
Graphene oxide/hollow mesoporous silica	Methylene blue; Methylene orange	476.19; 279.27	Sari Yilmaz (2022)
Graphene oxide/4 A molecular sieve	Rhodamine B	62.81	Liu et al. (2022b)
Graphene oxide/Amine-amide functionalized	Basic blue 41; Methyl orange	199.5; 64.0	Verma et al. (2022a)
Magnetic Nitrogen-doped graphene oxide	Reactive orange 12	333.33	Alsaiari et al. (2022)
Amino-magnetite@graphene oxide@amino-MnO ₂	Congo red	54.95	Mahmoud et al. (2022)
Mesoporous zeolite-A/reduced graphene oxide nanocomposite	Methylene blue	638.0	Farghali et al. (2021)
Metal ferrite-enabled graphene oxide nanocomposites	Methylene blue	25.81–76.34	Bayantong et al. (2021)
Polymeric graphene oxide nanocomposites	Antacid Orange RL	21.43–44.76	Noreen et al. (2021)
Graphene oxide/Konjac glucomannan	Methyl blue; Methyl orange	133.67; 93.50	Gan et al. (2015)
Graphene oxide/Bismuth oxide	Rhodamine B	320.0	Das et al. (2018)
Graphene oxide/magnetic silica nanocomposite	Pesticides: Chlorpyrifos, Parathion, Malathion	25.6; 135; 61.9	Wanjeri et al. (2018)
Graphene nanoplatelets	Chlorpyrifos	140.0	Lazarević-Pašti et al. (2018)
Reduced graphene oxide/silver nanocomposite	Lindane	827.00	Sen Gupta et al. (2015)
Graphene oxide/Fe ₃ O ₄	Endrin; dieldrin	99.0; 1.0	Shrivastava et al. (2017)
Graphene oxide/ β -CD-Fe ₃ O ₄	Thiacloprid; thiamethoxam; imidacloprid	3.11; 0.66; 1.42	Liu et al. (2017)
Reduced graphene oxide/biochar	Atrazine	67.55	Zhang et al. (2018)
Graphene oxide	Chlorpyrifos, Malathion	98; 1,667	Yadav et al. (2019)
Graphene wool	Other aromatic compounds: Phenanthrene; Pyrene	5; 20	Adeola and Forbes (2019)
Graphene oxide	p-nitrotoluene	238.8	Chen and Chen (2015)
Reduced graphene oxide	Pyrene; Anthracene; Naphthalene	198.0; 80.91; 766.58	Sun et al. (2013)
Graphene oxide/Fe ₃ O ₄	1-Naphthol; 1-Naphthamine	389.25; 408.09	Yang et al. (2013)
Graphene coated materials	Phenanthrene	1.74	Yang et al. (2015)
Graphene nanosheets	Phenanthrene	138–151	Apul et al. (2013)

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TABLE 4 (Continued) Graphene-based sorbents for removal of organic and inorganic pollutants in water.

Adsorbent	Target pollutant	Langmuir adsorption capacity (mg/g)	References
Graphene oxide/Fe ₃ O ₄	Aniline	170.4	Zhang et al. (2022)
Graphene/Fe ₃ O ₄	aniline and p-chloroaniline	375.94; 43.86	Chang et al. (2012)
Graphene	Bisphenol A	182.0	Xu et al. (2012)
Graphene nanosheets	Bisphenol A	181.82	Apul et al. (2013)
Graphene oxide/chitosan/organoclay	Bisphenol A/17 α -ethynylestradiol/Triclosan	20; 30; 40	de Almeida et al. (2022)
Graphene oxide/Amine-amide functionalized	4-Nitrophenol	54.1	Verma et al. (2022a)
Graphene nanosheet/biochar	Phthalic acid esters	25.43–45.65	Abdul et al. (2017)
Graphene wool	Pharmaceuticals: Efavirenz; Nevirapine	4.41; 48.31	Adeola et al. (2021b)
Graphene oxide passivated with ethylenediamine	Ibuprofen	240.0	Cai et al. (2016)
Graphene nanoplatelets/Fe ₃ O ₄	Amoxicillin	14.10	Kerkez-Kuyumcu et al. (2016)
Graphene oxide	Tetracycline; oxytetracycline	39.1; 130.4	El Hadki et al. (2021)
Graphene oxide/hydrogel	Doxycycline	15.9	Abdulsahib et al. (2020)
Graphene oxide/cyclodextrin/iron nanocomposite	Oxytetracycline	26.8	Lin et al. (2022b)
Graphene oxide/magnetite	Ciprofloxacin; Norfloxacin	18.22; 22.20	Tang et al. (2013)
Graphene oxide/biochar	Doxycycline	796.8	Xiong et al. (2022)

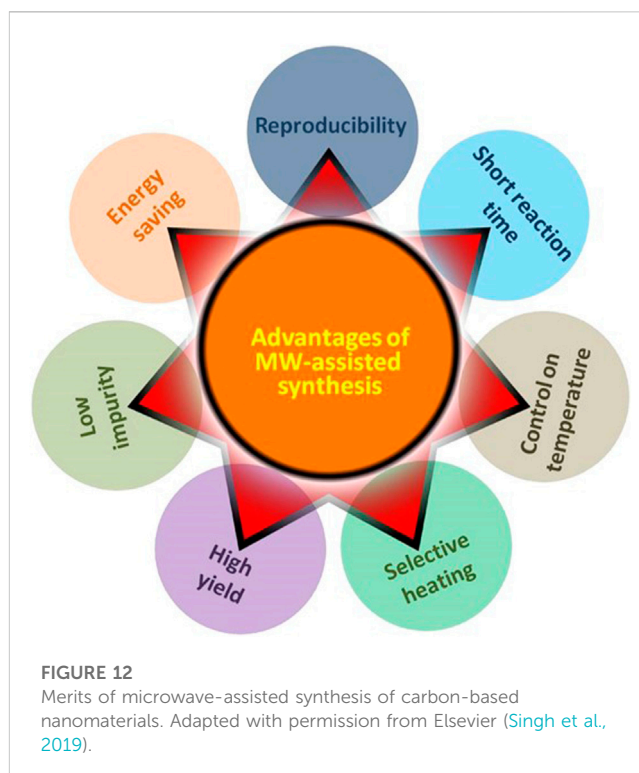
TABLE 5 CDs and derivatives as adsorbents for removal of organic and inorganic pollutants in water.

Adsorbent	Target pollutant	Langmuir adsorption capacity	References
Carbon dots-modified magnetic nanocomposites	Benzo [a]pyrene	76.23 ng/mg	Yang et al. (2019)
Carbon dot-modified magnetic carbon nanotubes	Carbamazepine	23.78 mg/g	Deng et al. (2019)
Hexadecylamine functionalized graphene quantum dots	Phenanthrene	1,377 mg/g	Kubheka et al. (2022)
Ultrathin BiOCl/nitrogen-doped graphene quantum dots composites	Ciprofloxacin	-	Mou et al. (2019)
Nitrogen-doped carbon quantum dots/magnetite nanocomposites	Methylene blue	24.88 mg/g	Tadesse et al. (2018)
Carbon dots modified mesoporous organosilica	2, 4-dichlorophenol	99.70 mg/g	Wang et al. (2015b)
Nitrogen-doped carbon quantum dots	Cadmium (II), Lead (II)	-	Sabet and Mahdavi (2019)
N-doped carbon quantum dot conjugated with Fe ₃ O ₄	Lead (II)	303.03	Mashkani et al. (2018)
Carbon dots-doped hydrogel particles	Lead (II), Cadmium (II), Mercury (II), and Chromium (III)	90.0, 70.2 62.0, 25.0	Perumal et al. (2022)
Fluorescent carbon quantum dots	Lead (II)	10.70–12.77 mg/g	Tohamy et al. (2023)
Magnetic CQDs modified MnFe ₂ O ₄ nanocomposite	Uranium (VI)	194.2 mg/g	Huang et al. (2019c)
Carbon quantum dots/layered double hydroxide hybrid	Cadmium (II)	14.71 mg/g	Rahmanian et al. (2018)
S,N-codoped carbon quantum dots modified chitosan membranes	Cadmium (II)	112.4 mg/g	Jlassi et al. (2020)
Carbon quantum dots	Cadmium (II)	66.68 mg/g	Yang et al. (2018b)

In general, carbon-based nanomaterials interact with organic and inorganic compounds via one or several adsorption mechanisms. Electrostatic interaction: adsorption of charged organic and inorganic pollutants onto the charged surface of carbon-based nanomaterials (Isaeva et al., 2021). Electric potential is created on the adsorbent by the presence of positive and negative charges on the carbon adsorbents as a result of deviation from the pH point of zero charge. Under varying solution pH, organic pollutants become charged as a result of protonation and deprotonation. These electrostatic charges cause adsorption due to electrostatic interactions on both the surface of the organic pollutant and the surface of the carbon-based nanomaterial. When functional groups are protonated or deprotonated, the strength of these interactions also varies (Deline et al., 2020; Isaeva et al., 2021). π -complexation interactions involve electron sharing between contaminants and carbon-based nanomaterials, resulting in the creation of inner-sphere surface complexes. The overlap of π electrons between sorbate and carbon materials also leads to π -complexation or π - π interactions (Maitlo et al., 2019). Hydrogen bonding occurs mostly in compounds containing hydroxyl and carboxyl groups, and their adsorption by carbon nanomaterials is facilitated via this mechanism in aqueous medium (Speranza, 2021). Electron donors with an atom more electronegative than hydrogen and an acceptor atom with an unshared lone pair of electrons combine to produce a hydrogen bond between carbon nanomaterials and pollutants (Ahmed et al., 2022). Hydrophobic interactions: hydrophobic contaminants maybe driven to the surface and pores of hydrophobic carbon nanomaterials (Saji, 2021). The octanol-water partition coefficient (LogK_{ow}) of the pollutants reflects its hydrophobicity, and a partitioning mechanism often drives hydrophobic solutes in an aqueous medium onto the surface of carbon-based nanomaterials (Adeola and Forbes, 2019; Qian et al., 2020). Covalent bonding between adsorbate and adsorbent is often responsible for chemical adsorption (chemisorption). Covalent interaction between sorbates and carbon particles involves electron sharing for bond formation (Sabzehmeidani et al., 2021). On the hand, physisorption (physical adsorption) is driven by weak forces of interactions such as van der Waals interactions (Ion et al., 2021). Physisorption is often reversible and non-specific interaction between carbon nanomaterials and contaminants. It is controlled by competitive adsorption that occurs at varying rates at the heterogeneous surfaces of adsorbents (Agboola and Benson, 2021).

5 Merits and challenges of microwave-assisted synthesis of carbon-based nanomaterials

Significant advancements in microwave instrumentation have aided in the synthesis of materials with improved reaction conditions control, producing materials with more desirable physicochemical characteristics. Commercial microwave reactors are now equipped with fiber-optic probes, magnetic stirrers, and sensors for more precise monitoring of the synthesis conditions, resulting in an enhanced reaction yield with higher reproducibility (Favretto, 2003; Ferguson, 2003; Luo et al., 2013b; Dudley et al., 2015), lower energy requirements, and shorter reaction times, among others (Figure 12). However, pressure sensitivity and thresholds (typically lower than 400 psi), and penetration depth limitations are one of the main drawbacks of microwave-assisted



synthesis (Antonio De La et al., 2011; de Medeiros et al., 2019). The volume needed for commercial scale-up is also a challenge as conventional microwave reactors cannot accommodate large-scale synthesis to this day. There are also some restrictions on the size of the reaction sample for the microwave-assisted synthesis of carbon nanomaterials due to the short penetration depth in particular materials. *In-situ* measurements of the reaction conditions, particularly with metallic substrates, are the other major restriction of microwave-assisted synthesis. Metallic substrates can't be used for material synthesis or processing since they interfere with the electromagnetic field (Bilecka and Niederberger, 2010; Schwenke et al., 2015).

The use of microwave reactors for chemical synthesis and industrial scale-up applications still faces several fundamental difficulties and there are knowledge gaps. Important features like heating homogeneity and rate may change during scale-up, and may significantly impact operating efficiency and product yield, yet these characteristic changes may occur via vaguely understood mechanisms (Sturm et al., 2014; Buttress et al., 2017). Some theoretical tools and predictive models have been developed to model large-scale applications of microwaves; however, the intricacy of the electromagnetic field distribution inside microwave reactors supports only qualitative results from these models (Robinson et al., 2010a; Robinson et al., 2010b; Li et al., 2023b). Despite all of this, these models provide valuable insights in addition to experimental results. A promising method of creating quantitative numerical models is now available thanks to recent developments in commercial software and computational resources (Yang and Chen, 2021). These models can guide the design of advanced reactors by predicting their performance and validating those predictions with experiments and clarifying scale-up behavior

to increase industrial capacity. Techno-economic analysis such as a bottom-up or top-down cost method, can be used to estimate the budget for industrial scale-up, in terms of plant set-up, workers, electricity, waste generation, etc., to assess the technological and economic viability of carbon-based nanomaterials for water treatment (Ghosh et al., 2022; Mpongwana and Rathilal, 2022).

Carbon-based nanomaterials have demonstrated extremely high efficacy in water treatment, particularly when it comes to the elimination of both organic and inorganic pollutants. However, some carbon nanomaterials may not be the most appropriate choice for water treatment, if intended for direct use in membranes (Cao et al., 2022; Guo et al., 2022). Due to the presence of negatively charged groups, most carbon-based nanomaterials rapidly disperse in water, requiring high-speed centrifugation for their recovery (Almanassra et al., 2021; Abboud et al., 2022; Ge et al., 2023; Singh et al., 2023). Membrane fouling remains the main issue in the water treatment industry that can be reduced by utilizing carbon-based nanomaterials (Adeola and Forbes, 2021a; Singh et al., 2023). There is little techno-economic research on nanomaterials made of carbon and thus, challenging to determine the full potential of this class of nanomaterials in water treatment procedures, relying solely on adsorption performance.

In summary, microwave radiation has proven to be a very efficient heating source. It has the following benefits: 1) an effective heating source (superheating) that increases reaction rates and facilitates faster synthesis, 2) the ability to readily adjust instrumental or reaction parameters, 3) size and form control, 4) selective heating based on the idea that various materials react to microwaves differently, 5) due to homogeneous heating and improved process parameter control, chemical reactions are more reproducible under microwave heating than under traditional heating, 6) microwave chemistry may be combined with other established techniques, such as solvothermal and sonochemical techniques. On the other hand, the drawbacks of microwave-assisted synthesis include its lack of scalability (batch sizes are normally limited to a few grams), restricted applications (to materials that absorb microwaves), safety and health risks associated with the use of microwave-heating apparatus and cost of purchase of advanced microwave apparatus. Prior to industrial-scale utilization of advanced carbon nanomaterials synthesized with microwaves, commercial readiness, manufacturing scalability, availability at an economically viable cost, and post-treatment toxicity assessments are required. Furthermore, it will be useful to compare laboratory-scale and industrial site findings that will facilitate the development of carbon-based prototype filters and/or membranes for long-term wastewater treatment.

6 Concluding remarks

Advanced carbon-based nanomaterials, such as CNTs, CDs, graphene, and their composites along with other carbonaceous and non-carbonaceous materials, have been widely researched for the adsorptive removal of organic and inorganic pollutants in water. The development of effective methods for the separation and recycling of these contaminants is vital from environmental safety and process sustainability points of view. However, to facilitate the future design of carbon-based nanomaterials from bioresources via microwave technology and other techniques, it is important to have a

thorough understanding of the bonding behaviors and intermolecular interactions using spectroscopic and microscopic characterization. The need for the techno-economic feasibility study for field-based/industrial-scaled applications of microwave-synthesized carbon nanomaterials from biobased materials cannot be over-emphasized.

The compendium in this comprehensive review (Tables 1–5), reveals that numerous carbon-based nanomaterials have been modified using functional groups, containing fluorine, nitrogen, oxygen, and sulfur, and via composite formation with other nanoparticles such as polymers, chitosan, magnetite, and biobased materials. These modifications create binding sites, large surface areas, and/or magnetic properties for optimal pollutant removal. In the mechanistic study of interactions between contaminants and carbon-based nanomaterials, a variety of approaches have been used, including zeta potential measurement, spectroscopic characterization, imaging techniques, theoretical calculations, and computational modeling. Electrostatics, complexation, π - π interactions, hydrogen bonds, hydrophobic interactions, covalent chemisorption, and van der Waals interactions are a few examples of potential sorbate-sorbent interaction processes that may occur. Future research can focus on several areas to address some lingering challenges or gray areas. The selective adsorption of chemical contaminants in aqueous matrices has not been thoroughly studied, and the principles underlying the selectivity are still not entirely understood.

In conclusion, due to the myriad of functional groups on the nanomaterials/nanocomposite, multifunctional carbon-based sorbents can be used to remove multiple contaminants simultaneously. It is possible to regenerate carbon-based nanomaterials depending on the nature of the sorbates, either by washing with suitable solvents, photodegrading, mechanical discharge, and/or combusting at appropriate temperatures, without inducing any significant damage to the nanomaterial. Physically adsorbed pollutants can be released more easily than chemically adsorbed ones since desorption requires less energy, thus preserving the carbon nanomaterials' capacity for re-use. Recent developments demonstrate that under reasonably benign conditions, microwave technology would be an efficient method for producing pristine and functionalized carbon-based nanomaterials from biobased resources, with several benefits and prospective applications including the treatment of wastewater.

Author contributions

AA: Conceptualization; data curation; formal analysis, writing—original draft; writing—review and editing. MD: Data curation; formal analysis; writing—original draft. RN: Project administration; supervision; validation; writing—review and editing. All authors contributed to the article and approved the submitted version.

Acknowledgments

The authors are grateful for the funding provided by the Natural Sciences and Engineering Research Council (NSERC) through the Discovery Grant Program, and Concordia University for funding through the Research Chair Program.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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Supplementary material

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/frcrb.2023.1220021/full#supplementary-material>

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