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Carbon dots derived from biomass and their application towards fluorescence sensing of heavy metals – a review

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ABSTRACT

Environmental pollution from toxic metal ions has become a problem worldwide due rapid industrialization. The release of such toxic metal ions into the environment pose a serious threat to human health and biota. To abate migration of toxic metal ions through the food chain they should be detected and analytical techniques used to detect them are costly and sophisticated. Therefore, utilization of carbon dots as fluorescent-based metal ion sensors compared with the traditional analytical methods is rapidly increasing owing to their low toxicity, tunable fluorescent properties, rapid sensing, simplicity and inexpensive preparation methods. Carbon dots are able to detect metal ions based on the direct interaction between the metal ions and carbon dots which results in the change in the fluorescence signal of carbon dots. They can be synthesized from any carbon-containing source; however, research has shifted towards employing more environmentally friendly precursors such as biomass. As such, this review presents an overview of biomass derived carbon dots (BCDs) that have been employed for the fluorescence detection of environmental metal ion pollutants. Different synthetic methods of BCDs, i.e., top-down and bottom-up synthesis, are briefly discussed, followed by a summary of the possible mechanisms through which BCDs can undergo to enable the detection of analytes in different media. Furthermore, insights on recent advancements in the use of BCDs for metal ion detection are shared. Lastly, this review will conclude with future perspectives and shortcomings thus far.

KEYWORDS

Carbon dots, bottom-up synthesis, top-down synthesis, nanotechnology, contaminants

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INTRODUCTION

Over the years, the interaction of heavy metals and the environment has become more prevalent as research continues to draw attention to the growing rate of heavy metal contamination in the environment. Uncontrolled concentrations of these inorganic contaminants pose a serious threat to the environment and surrounding life, thus making it the focal point of detection. Fortunately, extensive research has been conducted to introduce more simplistic and cost-effective methods of heavy metal detection via nanotechnology instead of traditional instrumental techniques.[1] Through nanotechnology, several types of nano-scale materials of different particle sizes that typically range between 1-100 nm have been produced in efforts of environmental safety.[2-4] Amongst these are a form of fluorescent-based nanomaterial known as carbon dots (CDs), which can be defined as small sized zero-dimensional particles of size less than 10 nm that exhibit a quasi-spherical shape.[5-7] CDs were first discovered in 2004 by Xu and his associates during the purification of single walled carbon nanotube (SWNT) fragments.[8] Following that discovery, Sun and coworkers were the first to synthesize photo luminescent (PL) quantum CDs via laser ablation and surface passivation of a mixture of graphite powder and cement.[9] CDs have received much research attention owing to their many benefits: low cost, easy synthesis methods, and good biocompatibility, thus making their application multifaceted, ranging from bioimaging and sensing, photo-catalysis, drug delivery, energy conversion, analyte sensing, and anti-counterfeiting applications.[10-18] Their usage in versatile applications has shown significant potential in comparison to other quantum dots, such as traditional organic dyes and inorganic/semiconductor quantum dots (QDs) (e.g., QDs: CdSe, ZnO-PbS) because organic dyes tend to easily photo bleach. In contrast, semiconductor quantum dots are prone to blinking.[19] Additionally, QDs tend to be associated with metal toxicity, therefore limiting their application due to their high heavy metal composition. However, CDs make up for these shortcomings as they have high photostability and biocompatibility, good water solubility, are environmentally friendly due to their low toxicity, and can be easily functionalized to improve sensitivity for detection of several analytes.[20] Furthermore, CDs can be synthesized from any carbon containing materials that are inexpensive such as agricultural waste (watermelon peels,[21] kiwi fruit peels,[22] or potato peels[23]), industrial waste (sago waste[24]), livestock waste (cow manure[25]), to highlight just a few hence their environmentally friendly nature. CDs derived from biomass possess advantages in that it is available in abundance, low cost, excellent biocompatibility and highly soluble in water. Biomass derived CDs do not require further surface passivation or doping due to abundance of a variety of elements present in biomass.[26] The existence of a wide array of elements in biomass waste precursors give rise to different functional groups on the surface of CDs which makes them applicable in fluorescence sensing of a wide variety of toxic elements.[11] Fluorescence sensing of metal ions by CDs takes place through fluorescence enhancement or quenching of CDs upon interaction with metal ions and this characteristic of CDs has been exploited by scientists and researchers for fluorometric metal ion sensing.[1] It is important to note that the exact mechanism of metal ion sensing by CDs is not yet fully understood and as a result various possible mechanisms have been suggested such as static and dynamic quenching, complex formation, photoinduced electron transfer, Forster resonance energy transfer, inner filter effect and intramolecular charge transfer.[1,11] Therefore, CDs obtained from biomass have shown promise as low cost metal ion fluorimetric chemosensors.

Herein, this short review summarizes the most recent developments in metal ion sensing using biomass-derived CDs (BCDs). First, the various methods of preparing carbon dots derived from biomass are highlighted, followed by a brief discussion of mechanisms utilized for fluorescence-based sensors. Lastly, a review of the various metal ions detected recently over the past few years using BCDs is presented. A summary of BCDs synthesized through top-down and bottom-up approaches for detecting metal ions is also presented in this review work.

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PROPERTIES OF CARBON DOTS

The unique photoluminescence (PL) and physical properties of CDs have been leveraged in their application as fluorescent sensors. In biological applications, their small size allows for easy penetration of cells and plays a crucial role in the properties of CDs. A study by Sun et al (2021) indicated that the size of CDs directly impacted their antibacterial activity against *E. coli*.[27] Larger sized CDs were found to show decreased antibacterial activity which was attributed to possible differences in the cell uptake and distribution of the CDs in the membrane emphasizing the importance of particle size. Moreover, CDs possess good biocompatibility and minimal toxicity enabling their continual use in biological applications.

In addition to these properties, CDs also exhibit tunable fluorescence emission which is advantageous in the improvement of the sensitivity and selectivity of fluorescent sensors. For instance, Diao and coworkers illustrated how different reaction conditions in the synthesis of *Syringa obatata* Lindl CDs resulted in tuned fluorescence emission.[28] Two types of CDs were synthesized: CDs in the absence and presence of NaOH. CDs synthesized in the absence of NaOH emitted blue light under UV light (QY of 12.4%, emission peak at 425 nm under excitation at 340 nm) whereas those synthesized with NaOH emitted green fluorescence (QY of 6.5%, emission peak at 520 nm under excitation at 450 nm). Both CDs were used for the detection of Fe^{3+} with blue CDs showing better selectivity and sensitivity towards sensing of Fe^{3+} as the green CDs showed no response to Fe^{3+}. However, their fluorescence was quenched in the presence of Hg^{2+} and Pb^{2+} further illustrating that CDs can be tailor-made for target analyte analysis.

The stability of CDs has also proven to be a valuable feature for their use as sensors. Wang et al (2015) illustrated the benefits of stable CDs that were synthesized through the surface passivation of glucose CDs with glutathione via a hydrothermal approach.[29] The stability of the resultant CDs was investigated in the presence of multiple ions, under different xenon irradiation times and prolonged storage time. The findings of this study revealed that the presence of various cations caused no significant changes in the fluorescence intensity of the CDs and under a 2hr irradiation time of the CDs by xenon light, only an 8% reduction in the intensity was observed. Similarly, prolonged storage time did not have any influence on the fluorescence intensity as it remained unchanged therefore signifying their good stability. However, under different pH and temperature conditions, fluorescence quenching and red shifts in their emission was observed with increasing pH and temperature indicating their potential as fluorescent sensors for both parameters. Furthermore, this study also indicates how CDs can easily be functionalized to modify their properties. In summary, CDs possess multiple desirable features that validate their suitability as fluorescent sensors.

SYNTHESIS OF CARBON DOTS FROM BIOMASS

Biomass is commonly used as a renewable energy source; however, its use for CDs synthesis has been widely applied over the years owing to its eco-friendly and low-cost nature and its wide distribution. Biomass waste is commonly used during synthesis of CDs as it is a good carbon source and inexpensive. Furthermore, utilization of biomass waste is advantageous to environmental causes such as waste minimization as underutilized waste can be recycled and converted to transform into materials of high significance hence the high interest in biomass derived CDs. Various preparation methods of biomass-CDs (BCDs) have been developed since their discovery in 2004 and are classified into two categories: bottom-up approach and top-down approaches (Figure 1).[30] The top-down approach typically involves the disintegration of bulky carbon material such as graphite and active carbons into nanosized particles through chemical and physical techniques such as laser ablation, arc discharge, chemical oxidation, and electrochemical oxidation.[31]

On the other hand, the bottom-up approach converts small-sized materials to nanoparticles via carbonization through techniques such as microwave irradiation, thermal pyrolysis, and hydrothermal/solvothermal.[32] Bottom-up methods have gained much interest as they are the most utilized methods employed to fabricate BCDs. They are less cumbersome and cost efficient, do not make use of harsh reaction conditions during synthesis, hence are a greener option and allow for easier functionalization.[33,34] Furthermore, top-down approaches require the use of large carbon materials, thus limiting application in BCDs, however, some of the top-down methods will be briefly discussed as well.

Top-down method for synthesis of Carbon dots

Arc Discharge

This is a method commonly used to synthesize materials such as carbon nanotubes; during this method of fabrication, a plasma is produced by the production of an arc between an anode and cathode which, is then used to vaporize the carbon material and hence producing carbon quantum dots in the cathode.[35,36] Synthesis of CDs via arc discharge was first performed in 2004 during the preparation of single-walled carbon nanotubes.[8] A band of fluorescent material was discovered during the preparative electrophoresis of the SWNTs. It was characterized to reveal different colored components under UV light, thus unearthing a class of photoluminescent nanoparticles, i.e., CDs. Recently, Chao-Mujica et al., (2021) produced carbon quantum dots of average size 2.3 nm via a modified version of arc discharge known as submerged arc discharge in water (SADW) with a quantum yield (QY) of 16% being the highest ever as CDs produced from this method are known for their low QYs.[37,38]

Laser Ablation

High temperatures and pressures are typically used during synthesis, and the carbon precursor is vaporized in the presence of high temperature using a pulsed laser until it is converted to a plasma at high laser flux and eventually condenses to form nanoparticles.[39] Laser ablation has been widely used to synthesize CDs because of its efficiency, rapidness, and highly tunable attributes.[40] The use of laser ablation in CDs synthesis is limited because of the low QYs that are usually obtained and poor size control of nanoparticles.[41] However, in 2011, work by Hu and coworkers made a successful attempt to mitigate the demerit of being unable to control the size

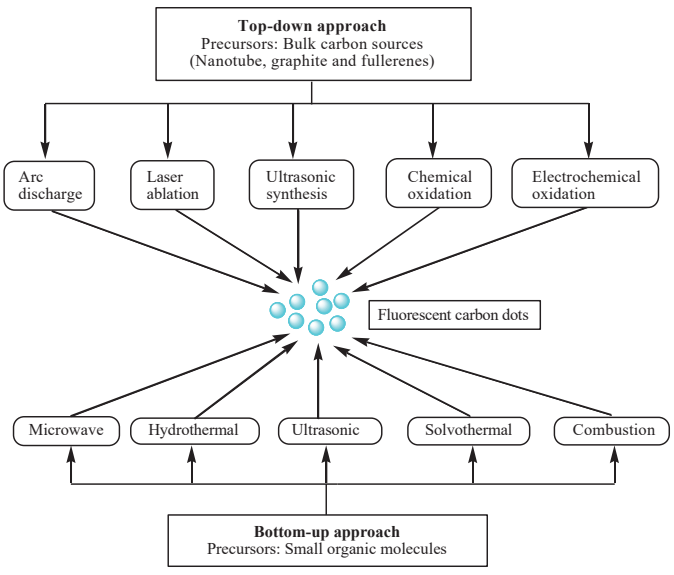

Figure 1: Schematic representation for the synthesis of carbon dots using bottom-up and top-down approaches.

Table 1. Biomass derived carbon dots synthesized through top-down approaches for metal ion detection.

Precursor	Type of Top-down method	Target metal	Limit of detection (LOD)	Reference
Alga (*Dunaliella Salina*)	Chemical oxidation	Hg(II) Cr(VI)	0.018 μM 0.018 μM	[51]
Muskmelon (*Cucumis melo*) fruit	Chemical oxidation	Hg (II)	0.33 μM	[45]
Tomato (*Solanum lycopersicum*)	Chemical oxidation	Fe (III)	0.016 μM	[52]

of the nanoparticle. Their work indicated that controllable sizes are attainable through modifying laser parameters during the preparation of CDs from graphite flakes.[42] Wang et al., (2020) reported a multistep synthesis of soybean derived CDs utilizing a laser ablation in liquid approach to introduce nitrogen groups.[43] CDs that had undergone hydrothermal carbonization and high temperature annealing were ablated using an Nd:YAG laser in the presence of NH_4OH using a pulse energy of 100 mJ for 1 hr to produce nanosized carbon particles with an average size 9.57 nm. Their surface was revealed to possess nitrogen rich functional groups which improved the PL properties of the CDs. The CDs exhibited bright blue emission with a QY of 1.81% in comparison to CDs that were annealed without ablation which showed no emission and hence no measurable QY. Developments in the synthesis process show potential of producing CDs with better PL properties and consistent sizes.

Chemical oxidation

This method involves the treatment of carbonized precursors with oxidizing acids such as nitric acid and sulphuric acid to form CDs.[44] As common as it may be due to its accessibility and provision for large-scale production, this method has been criticized for its use of harsh reaction conditions, multistep processes of purification and neutralization, failure to provide sufficient control over the sizes of CDs and the possibility of contamination of CDs due to residues from oxidizing agents, thus potentially being toxic.[45,46] However, in efforts to provide a more environmentally friendly approach, a study about the green synthesis of blue- and green-, yellow-CDs from tomato (Solanum lycopersicum) for potential application in bioimaging and metal ion detection was reported by Kailasa and coworkers.[47] A well dispersed mixture of frozen tomato slices and H_2SO_4 was heated at 100 °C for 1hr to produce blue emitting CDs. Similarly, green and yellow-CDs, were synthesized by heating a sonicated mixture of frozen tomato and H_3PO_4 at 80 °C for 25 min and 20 min respectively. The three resultant solutions were neutralized with NaOH and then purified through dialysis processes. All three CDs consisted of carboxylic, hydroxyl and amine functional groups with average sizes of 5-10 nm. Furthermore, the CDs were found to have excitation wavelength dependent emission, with QYs of 12.70/%, 4.21% and 2.76% for blue- and green-, yellow-CDs respectively. These properties enabled the successful detection of Fe^{3+} in biofluids and pharmaceuticals samples. Additionally, the CDs showed non toxicity and good compatibility in the bioimaging of HeLa cells. Cheng and associates reported a facile preparation of CDs from a green source via chemical oxidation; in their study, walnut shell-based CDs were synthesized by soaking walnut shells in NaCl and phosphoric acid under ambient conditions.[48] The walnut shells were then dehydrated in an oven and preoxidized under air in a carbonizing furnace in the presence of nitrogen gas. The carbonized walnut shells were then mixed with HNO_3 and H_2SO_4, which was heated in an oil bath and neutralized with NaOH after cooling to room temperature. The solution was then purified via filtration and dialysis. The resultant green fluorescent CDs were found to have a crystalline core with an average size of 3.4 nm, good photoluminescence and photostability and pH-sensitive properties. Furthermore, the CDs were non-toxic and exhibited excellent cytocompatibility, therefore showing significant potential in bioimaging and disease diagnosing. Although reports of green source CDs synthesized via this method have continued, concerns associated with this method are yet to be addressed in future works to reduce its limited application in the synthesis of CDs.[49,50]

Electrochemical synthesis

Known for its convenience, good reproducibility, and simplicity, the electrochemical method is also a novel top-down approach method.[51] Electrochemical approaches offer a facile way to tune properties of CDs such as particle size and photoluminescence by adjusting experimental parameters, hence their wide usage in literature. For example, Borna et al., (2021) reported a study investigating the effect of synthesis method and the effect of current on fluorescent intensity of CDs. CDs were synthesized from two different approaches: hydrothermal and electrochemical synthesis using apple juice and graphite as precursors. In the synthesis of CDs via an electrochemical approach, two graphite electrodes of diameter and length 0.5 cm and 5 cm were used as the anode and cathode, respectively. For the electrolyte, a solution of ethanol and sodium hydroxide was used, and electrochemical peeling was conducted using a DC current source, hence producing CDs of sizes 1-5 nm by stimulating graphic oxidation. The resultant solution was purified and separated by sonification and filtration.

In the hydrothermal synthesis, CDs were prepared from natural apple juice and ethanol, several centrifugation and separation steps were conducted to produce high fluorescing carbon dots. The study concluded that CDs prepared by hydrothermal showed lower fluorescence intensity than those produced electrochemically. The differences in their fluorescence intensities were attributed to their particle size, CDs produced via the hydrothermal method were found to have larger particle sizes ranging from 5-10 nm, compared to those produced via electrochemical oxidation hence lower intensities were obtained. The study also indicated that higher current intensities resulted in weaker fluorescence and larger particle sizes.[52] While it is true that CDs have been widely synthesized electrochemically, most of these are synthesized from chemical sources rather than biomass sources hence, reports of CDs fabricated electrochemically are rare.[53-55]

Bottom-up method for synthesis of carbon dots

Hydrothermal/Solvothermal

Hydrothermal/Solvothermal synthesis of biomass waste derived CDs is the most predominantly used method of production. This method involves the use of elevated pressures and temperatures to fabricate CDs from precursors within an autoclave and typically uses solutions during the synthesis process.[57] Its cost effectiveness, non-toxicity, and ecofriendly processes coupled with production of high QY CDs make it a reliable and efficient option. For instance, Atchudan et al., (2020) synthesized nitrogen-doped CDs derived from dwarf banana peel via hydrothermal method. The resultant CDs were found to have a graphitic structure and a small amount of amorphous carbon with an average uniform size of 4 nm. Furthermore, good water solubility was indicated owing to the presence of functional groups such –COOH and –OH amongst many others. The HN-CDs exhibited excitation-dependent fluorescence with a relatively high QY of 23% and were very stable and possessed good resistance against photobleaching. These excellent properties allowed their usage as fluorescent ink for fluorescent writing and metal ion detection by fluorescence quenching of CDs.[58]

Chung et al., (2020) fabricated CDs/hydroxyapatite (CD-HAP) from sugarcane bagasse char over a wide range of temperatures under

hydrothermal conditions. Synthesis of the CD-HAP was first conducted by hydrothermally heating a mixture of charred sugarcane bagasse and NaOH in an autoclave. Following filtration, the different volumes of CDs were added to readily prepared phosphate mixtures, and each was transferred gradually into solutions of calcium nitrate tetrahydrate in HCl. Characterization of the CDs indicated that the optimum synthesis conditions were at 190 °C, and the size of the CDs was 4.72 ± 1.1 nm; in terms of the structure, sugarcane bagasse CDs were monodispersed whereas CD-HAP exhibited a rod-like shape, but however had the same functional groups. The study concluded that the CDs showed excellent fluorescent properties and enhanced those of the CD-HAP nanocomposite, as well as their drug loading capacity.[59]

Paul and Kurian (2021) synthesized water-soluble nitrogen-doped CDs from two different precursors: jackfruit peel and tamarind peel under hydrothermal conditions for optical and biomedical applications (Figure 2).[60] The jackfruit peel derived N-CDs were found to have an average particle size of 6.4 nm with QY of 13.04%, whilst those derived from tarmind peels were of size 5.3 nm with QY of 6.13%. Both blue emitting N-CDs showed strong photoluminescence at excitation 350 nm owing to the presence of hydroxyl and nitrogen moieties revealed by FTIR analysis.

Determination of chlortetracycline (CTC) in pork samples was carried out by Zhang et al., (2021) using N-doped CDs. A one-step hydrothermal approach was used to synthesize N-CDs from hawthorn fruit and diethylenetriamine (DETA) as the doping agent under optimum conditions. The resultant CDs were quasi-spherical shaped with an average size of 3.8 nm with a good QY of 22.96% and excitation-independent fluorescence. Existence of hydrophilic groups on the surface of N-CDs indicated successful doping, improving their fluorescent properties and successful rapid detection of CTC with good recoveries of 93.62–103.18% via fluorescence quenching.[61] This facile bottom-up method continues to be proven as the most preferred synthetic method for BCDs as growing reports of hydrothermally synthesized CDs with higher quantum yields are recorded.

Microwave assisted synthesis

This approach utilizes microwave energy to fabricate CDs and has also gained favorability over other bottom-up methods due to its quicker reaction times.[62] In a minute, eggshell membrane derived CDs were synthesized via microwave irradiation by Jusuf et al., (2018) for the degradation of methylene blue.[63] One-step synthesis was carried out from charred eggshell membrane in different indicated that smaller sized CDs of 3.88 nm ± 0.56 were obtained in NaOH solution while those in water solution had a larger average size of 4.46 ± 0.77 nm. The shapes of the CDs varied as well; CDs in NaOH were found to be spherical, whereas those in the water were irregular but however, had the same functional groups. The optical properties of CDs in NaOH solution were investigated and the resultant CDs showed pH-dependent photoluminescence and good photocatalytic abilities. Successful degradation of low concentrations of methylene blue and other pollutants under sunlight was performed.

Another one-step microwave-assisted synthesis of blue fluorescent CDs from Sesame seeds, was developed by Roshni and Divya (2017).[64] QY and size of CDs were determined to be 8.07% and 3-10 nm, respectively. Furthermore, the sesame seed derived CDs possessed good photostability as their fluorescence intensity was retained at 95%, and good aqueous solubility as well as high photoluminescence were also observed. Owing to these excellent properties, their application in optical sensing, specifically for ferric ions in this case, was carried out and selectivity towards Fe^{3+} was observed through fluorescence quenching. The limit of detection (LOD) was determined to be 2.56 µM.

Ang et al., (2020) illustrated a microwave-assisted conversion of palm kernel shell (PKS) to blue-emitting luminescent CDs for investigation of the effect of the reaction medium.[14] In this study, CDs were synthesized through a 1-5 minute irradiation time from a mixture of PKS with ultrapure water (UPW), UPW with chitosan,

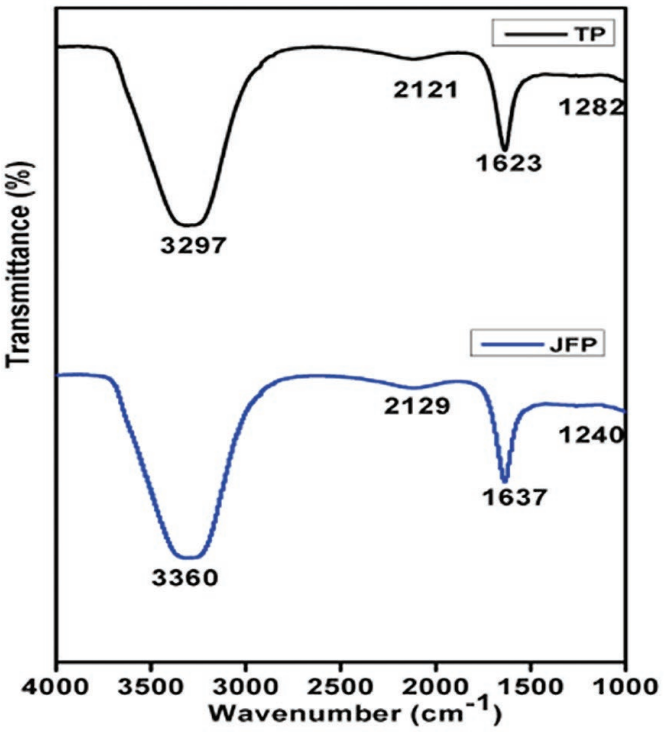

Figure 2: FT-IR spectrum of nitrogen doped CDs from jackfruit peel (JFP) and tamarind peel (TP). Reproduced with permission from Paul and Kurian et al., (2020).

and diethylene glycol (DEG). The resultant CDs all displayed an amorphous structure with sizes ranging between 6.00 nm -7.00 nm and similar functional groups such as hydroxyls and alkenes, but all lacked amino groups. However, the study concluded that CDs synthesized with DEG exhibited overall properties than compared to CDs subjected to different reaction media.

The formation of CDs produced from PKS with DEG had the highest QY and particle size of 44.0% and 7.00 nm, respectively, stronger luminescence under UV light, and displayed better absorbance and photoluminescence intensities. It was also observed that DEG medium allowed for shorter carbonization time, yielding more completely formed CDs. In contrast, CDs formed in UPW and UPW with chitosan exhibited lower QY of 26.3% each and weak fluorescence. No significant difference was observed in the properties of CDs in UPW and UPW with chitosan, thus indicating that PKS and chitosan did not react to form amino-rich CDs, therefore showing the significance in choice of the starting material. All CDs were successfully applied in cellular imaging and detection/removal of metal ions.

A unique solvent-free microwave irradiation method for the preparation of human hair derived CDs was presented by Singh et al., (2020). In detail, pretreated human hair was irradiated with microwaves in 1100 W Panasonic home microwave for 5 minutes. The carbonized human hair was mixed with water, sonicated, and filtered with an aluminum oxide containing column for purification. The prepared CDs were of average size 78 nm and contained 1.21% nitrogen owing to keratin content found in human hair, hence producing N-doped CDs. The CDs exhibited good optical properties such as strong absorbance and emission properties and photoluminescence with a PL QY of 17%. Preparation of CDs from the same precursor was done via a hydrothermal approach in a sealed autoclave. From this, the effect of synthesis approach on the properties of CDs was highlighted. Hydrothermally produced CDs had a high nitrogen content of 8.91% and a higher PL QY of 38%, thus indicating that surface functionalities and other optical properties of CDs can be tuned by employing different synthesis procedures.[65] Although microwave-assisted synthesis presents several advantages compared to other bottom-up methods such as low-cost setups and uniform

Table 2. Biomass derived carbon dots synthesized through bottom-up approaches for metal ion detection.

Precursor	Type of Bottom-up method	Target metal ion	Limit of detection (LOD)	Reference
Oyster mushroom (*Pleurotus species*)	Hydrothermal	Pb^{2+}	58.63 µM	74
Mango (*Mangifera indica*) leaves	Pyrolysis	Fe^{2+}	0.62 ppm	75
Mint leaves	Hydrothermal	Fe^{3+}	374 nM	76
Mexican Mint leaves (*Plectranthus amboinicus*)	Microwave assisted	Fe^{3+}	0.53 µM	77
Dwarf banana peel	Hydrothermal	Fe^{3+}	0.66 µM	58
Finger millet ragi (*Eleusine coracana*)	Pyrolysis	Cu^{2+}	10 nM	78
Prickly pear cactus	Hydrothermal	As^{3+}	2.3 nM	79
Bamboo leaves	Solvothermal	Pb^{2+}	0.14 nM	80
Gingko biboloa	Hydrothermal	Pb^{2+}	55 nM	81
Tea Residue	Solvothermal	Cd^{2+}	2.14 µg/mL	82
Banana (*Musa acuminate*)	Hydrothermal	Cu^{2+}	0.3 µg/mL	83
Coconut coir	Thermal calcination	Cu^{2+} Cd^{2+}	0.28 nM 0.18 nM	84
Cranberry beans	Hydrothermal	Fe^{3+}	9.55 µM	85
Waste tea	Hydrothermal	Fe^{3+} CrO_4^{2-}	0.15 µM 0.81 µM	86
Volvariella volvacea mushroom	Hydrothermal	Pb^{2+} Fe^{3+}	12 nM 16 nM	87
Kentucky bluegrass (*Poa pratensis*)	Hydrothermal	Mn^{2+} Fe^{3+}	1.2 µM 1.4 µM	88
Rice residue	Hydrothermal	Fe^{3+}	0.7462 µM	89
Brewery waste grain	Microwave	Fe^{3+}	0.05 µM	90
Palm kernel shell	Microwave	Cu^{2+}	0.05 mM	91
Radish	Hydrothermal	Cu^{2+}	6.8 µM	92
B. flaellifer (Ice apple) endosperm	Hydrothermal	Fe^{3+}	2.01 µM	93
Seville Orange	Hydrothermal	Fe^{3+}	0.53 µM	94
Gardenia fruit	Hydrothermal	Hg^{2+}	320 nM	95
Pearl Millet Seeds	Pyrolysis	Pb^{2+}	0.18 nM	96
Table sugar	Microwave	Pb^{2+}	14 ppb	97
Broad bean	Hydrothermal	Hg^{2+}	0.96 µM	98
Quince fruit (*Cydonia oblomga*) powder	Microwave	As^{3+}	0.04 **µg** mL^{-1}	99
Oil palm fruit bunch carboxymethylcellulose	Hydrothermal carbonization	Hg^{2+}	0.01 µM	100
Bitter tea tree residue	Hydrothermal	Hg^{2+}	-	101
Coconut water	Hydrothermal	Zn^{2+}	-	102
Pork Bone, bovine bone, and sheep bone	Hydrothermal	Ag^{+}, Cu^{2+}, Hg^{2+}, Fe^{3+}, Pb^{2+}	-	103
Lantana camara berries	Hydrothermal	Pb^{2+}	9.64 nM	104
Poria cocos polysaccharide	Hydrothermal	Cr^{6+}	0.25 µM	105
Water amaranth leave	Hydrothermal	Cd^{2+}	15 nM	106
Ocimum sanctum leaves	Hydrothermal	Pb^{2+}	0.59 nM	107
Bread waste	Hydrothermal	Pb^{2+}	8.9 nM	108
Flax straw	Hydrothermal	Co^{2+} Cr^{6+}	0.38 µM 0.19 µM	109
Kelp	Microwave	Co^{2+}	0.39 µM	110
Starch	Pyrolysis	Ru^{3+}	<100 µM	111
Yeast Cryptococcus podzolicus	Hydrothermal	Ag^{+}	113.57 nM	112

heating of CDs precursors, production of CDs in larger scales remain a challenge, hence limiting this method for small volume reactions.[66]

Pyrolysis

Pyrolysis typically involves decomposition of organic materials under high temperatures and controlled pressures.[67] Crystalline CDs were synthesized by pyrolyzing cellulose from bamboo leaves by Fahmi et al., (2018) and applied in tumor imaging and therapy after modification with 4-carboxybenzylboronic. The resulting CDs possessed good biocompatibility, multicolor fluorescent properties, and were non-toxic.[68] Aji and coworkers developed a facile pyrolysis approach for fabrication of luminescent nitrogen doped CDs from mangosteen peel and urea.[69] The effect of urea and temperature on CDs features was investigated, and the resultant CDs were reported to contain amine, ketone, and methyl groups, amongst many others, therefore, indicating successful surface passivation by urea. The study concluded that the number of formed CDs could be tuned by varying urea concentration, but no effect was observed in the absorbance or PL properties of CDs. In addition, higher temperatures were found to increase the photoluminescence of the CDs.

Guo et al., (2020) reported a green synthetic approach of turtle shell derived CDs.[70] The highly fluorescent CDs obtained were of an average size of 2.62 nm. They contained a variety of functional groups such as amino groups, hydroxyl, and carboxyl, thus giving them good dispersibility in water. Moreover, the CDs exhibited good fluorescence stability, excitation-dependent PL with QY of 45%, and low toxicity. These remarkable features were attributed to the composition of the precursor: turtle shells, which contain high amounts of collagen. The presence of amino acids in collagen resulted in the formation of CDs decorated with oxygen and nitrogen groups responsible for their PL properties. These were successfully used in applications such as multisignal anti-counterfeiting and optoelectronics by inkjet printing.

In another study CDs were synthesized from agro-industrial residues via pyrolysis; Yerba Mate (YM), Avocado Seed (AS), and Orange Peel (OP) were used as precursors to produce CDs with different physiochemical properties.[71] The effect of precursors on the morphology and fluorescence behavior and other physiochemical properties were discussed in great detail for potential application as emulsion stabilizers and photocatalysts. Carica papaya waste peel extract was used to synthesize CDs via pyrolysis to detect chromium in water.[72] The CDs were successfully prepared by carbonizing the peel extract in a hot air oven and the residue was mixed with deionized water. The CDs were reported to possess absorption-emission characteristics and luminescence properties, as they were found to emit blue fluorescence under UV light. Furthermore, the structural properties of the CDs were investigated and revealed that the CDs were amorphous with spherical-shaped particles of an average size of ~7nm, and their surface was found to have a presence of several carbonyl and functionalities, thus allowing for facile functionalization with ethylene diamine tetra acetic acid (EDTA). The modified CDs were used to effectively detect chromium via a chemo-sensor probe. Like other bottom-up approaches, pyrolysis is also preferred as a synthetic method as it is easy to operate, solvent free synthesis is attainable and economical.[73] However, the use of high temperatures during synthesis is a disadvantage. Thus, it is less popular than other methods such as hydrothermal approach.[48]

Enhancement strategies for photoluminescent properties of carbon dots

The tunability of the photoluminescent properties of CDs is key for improving their effectiveness in different applications. By employing different approaches such as surface passivation, tuning of synthesis parameters and heteroatom doping, the performance of CDs can be enhanced for better sensitivity and selectivity. For example, the effect of heteroatom doping on chitosan derived CDs was investigated using EDTA as a dopant by Gong and coworkers.[113] In this study, two types of CDs were synthesized via microwave irradiation: chitosan-acetic acid CDs (CDs) and EDTA doped chitosan-acetic acid CDs (N-CDs). TEM images showed that the average sizes of CDs and N-CDs were 2.48 nm and 4.27 nm respectively, suggesting that the formation of larger CD particles could be achieved through doping. XPS analysis of the CDs revealed further differences in the composition of both CDs; the elemental content of C, H, O, N of CDs was 37.02, 6.55, 44.68 and 11.75%, whereas in N-CDs 50.60, 7.92, 22.65 and 18.83% was obtained, respectively. These findings indicated successful nitrogen doping as higher nitrogen content was achieved. Moreover, the optical properties of N-CDs showed stronger fluorescence under UV light as supported by the photoluminescence spectra showing higher intensities. The QY of the CDs also increased from 1.6% to 20.02% after doping thus illustrating the benefits of heteroatom doping.

Demonstrating the effect of NH_4OH as a passivant, Liang et al. reported the facile synthesis of passivated Xylan CDs (NCDs) using a one-pot hydrothermal synthesis.[114] A comparison between the non-passivated CDs (BCDs) and NH_4OH passivated CDs (N-CDs) showed that N-CDs exhibited better properties. To illustrate this, the morphology of both CDs was investigated, from the TEM data the average size of BCDs was 19.06 nm. However, the incorporation of NH_4OH to the synthesis process resulted in the formation of smaller sized particles of size 6.92 nm. Additionally, higher nitrogen contents of 6.37% were found in NCDs while BCDs contained no nitrogen content. The presence of nitrogen groups on the CDs resulted in brighter fluorescence emission and higher QY of 16.18% compared to 2.06% of BCDs.

In another study, Monday et al. illustrated how various synthesis parameters affected the properties of CDs.[115] Three types of palm kernel shell derived CDs: undoped CDs, Ethylenediamine doped CDs (CDs-EDA) and L-phenylalanine doped CDs (CDs-L-Ph) were hydrothermally synthesized. The effect of the dopant amount was investigated with both CDs-EDA and CDs-L-Ph showing an increase to higher fluorescence intensities and red shifts in the emission as the dopant quantity was increased from 0.2-1.0 mL for EDA and 80-120 mg of L-Ph. Furthermore, higher temperatures during the synthesis process also led to higher fluorescence intensities for both doped CDs. In addition to other similarities, the particle sizes of CDs-EDA and CDs-L-Ph were of average size of 2.1 and 2.0 nm respectively and both had comparable emission and FTIR spectra. However, differences in their XRD patterns were observed; CDs-L-Ph were reported to have higher crystallinity compared to CDs-EDA. Moreover, varying QYs of 13.7% and 8.6% were obtained for CDs-EDA and CDs-L-Ph, respectively, demonstrating how the dopant influences the properties of CDs.

MECHANISM OF METAL ION SENSING BY CARBON DOTS

CDs can sense and detect metal ions through their interaction together. Their usage in the field of metal ion sensing is attributed to the optical properties that CDs tend to possess and their surface/structural properties, thus making metal ion detection viable following different mechanisms. Detection of metal ions by CDs is usually signified by the enhancement or quenching of fluorescence intensity as illustrated by Figure 3. It is important to note that the mechanisms through which metal ions interact with CDs still need to be fully understood. As a result, several mechanisms have been suggested, such as dynamic and static quenching, Forster energy resonance (FRET), complex formation, aggregation, photo-induced electron transfer (PET), and inner filter effect (IFE), amongst many others. These mechanisms have been used to understand and explain the interaction between metal ions and CDs.[116]

Static and dynamic quenching

Static and dynamic quenching are amongst the most common phenomena used to explain the quenching mechanisms of any

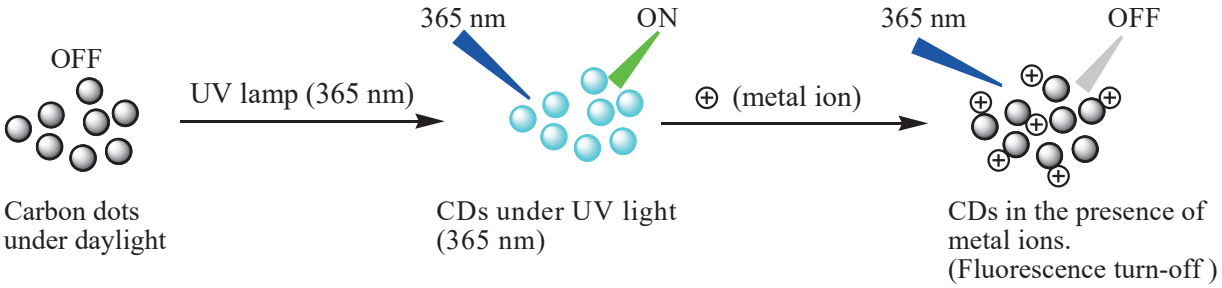

Figure 3: Schematic illustration of the turn on-off mechanism of carbon dots.

fluorescent molecule upon interaction with another analyte. In the case of CDs and their interactions with metal ions, static quenching occurs when complexation between CDs and metal ions occurs in the ground state resulting in the formation of a non-fluorescent complex. This interaction is usually characterized by changes in the absorption spectrum as a result of the ground complex, insignificant changes in the fluorescence lifetimes of the CDs and an increase in fluorescent intensity as the temperature increases.[117] In contrast, dynamic quenching, involves collisions between the metal ion and the excited state of CDs before returning to its ground state.[118] This mechanism exhibits features that are opposite to static quenching: at higher temperatures, the quenching effect by the metal ion increases due to increased frequency of collisions, no changes in absorption spectra of CDs in the absence/presence of the metal ion are observed but the fluorescence lifetime of the CDs changes.[119] Yan et al.,(2019) reported a method of detecting silver ions through the use of yellow–emissive CDs synthesized from anhydrous citric acid and 2,3-phenazinediamine.[120] Detection of Ag^{+} by CDs was proven viable as the fluorescence intensity of CDs decreased upon the addition of Ag^{+}. This was attributed to static quenching as the CD absorption spectrum changes were observed in the presence of Ag^{+} indicating a static mechanism. Furthermore, studies on Stern-Volmer parameters at different temperatures and fluorescence lifetime were conducted to validate detection by this mechanism. No changes in the fluorescence lifetime were observed; a decreasing value of Stern-Volmer quenching constant K_{sv} as temperature increases was reported, thus suggesting static quenching. Zeta potential studies were also carried out to support this.

On the contrary, Hg^{2+} detection in aqueous media via dynamic quenching was demonstrated through using citric acid, ethylenediamine, and methyl blue-derived CDs.[121] With increasing concentration of Hg^{2+}, no changes in the absorption spectra of CDs were seen however their fluorescence lifetime was reported to decrease linearly, thus signifying dynamic quenching. The two mechanisms are typically distinguished through fluorescence lifetime studies, analysis of UV-vis absorption spectra of CDs before and after quenching by the specific metal ion at various concentrations as well as studies of Stern-Volmer parameters.[122,123]

Complex formation

The presence of various oxygenated groups on the surface of CDs and their ability to be chemically modified by functionalization and doping allows for coordination between CDs and metal ions.[124] This occurs through the donation of electron pairs from electron-rich species on the surface of CDs to the metal ion, thus forming a complex.[125,126] Phan and coworkers carried out the successful detection of Fe^{2+} through a nitrogen doped CDs nanoprobe by coordination mechanism.[127] The ferrous ion was reported to have a significant quenching effect on the fluorescence of the NCDs due to the formation of the coordinate bond between Fe^{2+} and oxygen atoms on the surface of the NCDS. Complexation between CDs and metal ions forms the basis of most mechanisms discussed throughout literature as they depend on the interaction between the surface's functional groups of CDs and metal analyte.

Photo-induced electron transfer

The photo-induced electron transfer (PET) mechanism involves the formation of a complex in the excited state by transferring of an electron between an electron donor molecule and an electron acceptor molecule.[128] However, the complex can return to the ground state without releasing a photon. In PET, the metal ion and CDs may act as donors or acceptors depending on the type of PET, i.e., reductive or oxidative electron transfer.[118] In the case of an oxidative electron transfer, the CDs act as the electron donor; an excited electron is donated from the lowest unoccupied molecular orbital (LUMO) of CDs to the lowest occupied molecular orbital (LUMO) of metal ions.[129] In contrast, a reductive electron transfer occurs when the energy level of the HOMO of metal ion is higher than that of CDs therefore resulting in an electron donation from the metal ion to CDs, thus causing fluorescence quenching.[130]

Detection of Hg^{2+} in water was carried out using CDs derived from phenolphthalein and ethylenediamine; a quenching of the fluorescence was observed and attributed to the binding of Hg^{2+} to surface groups of CDs hence causing PET to occur.[131] PET has become a common phenomenon in detecting metal ions by CDs as several works involving the mechanism have been reported.[132-135] Studies involving fluorescence lifetime, cyclic voltammetry, and zeta potential values may be used to confirm PET mechanisms in sensing applications.

Forster resonance energy transfer

Forster resonance energy transfer (FRET) mechanism is a non-radiative energy transfer that occurs between two fluorophores i.e. donor-acceptor pair through dipole-dipole coupling over a short distance.[136] FRET-based systems are commonly used in biological applications to study molecular interactions of living cells but have, however, also proven useful in sensing various analytes.[137] For instance, Chini et al., (2019) designed an efficient FRET-based system for the detection of arsenic and mercury ions in water using graphene quantum dots (GQD) as donor and carbon quantum dots (acceptor) as a suitable donor-acceptor pair. Its efficiency was demonstrated through time-resolved photoluminescence studies and quenching of the FRET signal of the GQD-CD sensor upon interaction with the cations. Moreover, overlapping emission and absorption spectra of the GQDs and CDs were observed, hence indicating a successful FRET-based system.[138] In addition, a donor-acceptor distance of 10-100 Å can be used to prove a FRET based system.[139] The efficiency of this system may be improved by using an acceptor and donor pair that are ideal of which CDs have been proven to be suitable donors/acceptors owing to their unique properties, as discussed.

Inner filter effect

During the inner filter effect (IFE), fluorescence quenching is usually observed due to the reabsorption of emission/excitation energy. Although considered a common problem in fluorometry, IFE has been explored for several fluorescence-based sensing systems. For instance, Cu^{2+} was detected using N-CDs in aqueous samples; fluorescence

lifetime studies suggested an IFE sensing mechanism as the lifetimes remained unchanged in the presence of Cu^{2+}.[140]

Furthermore, the absorption wavelength of Cu^{2+} was observed between the range 180-400 nm whereas the excitation wavelength of N-CDs was in the range 200-400 nm therefore providing sufficient overlapping of the absorption and excitation spectrum of Cu^{2+} and N-CDs. Therefore, fluorescence quenching of CDs by Cu^{2+} via IFE was observed. Similarly, Gedda et al., (2016) used prawn shell derived CDs for the detection of Cu^{2+} in seawater. In the presence of Cu^{2+}, the fluorescence intensity of the CDs decreased hence successfully detected. Through emission and absorption studies, an IFE sensing mechanism was suggested.[141] This non-radiative process is characterized by unchanged fluorescence lifetime of the fluorophore upon interaction with an analyte and overlapping spectra of absorption of analyte and emission or excitation of fluorophore hence is less more complicated than other mechanisms such PET and FRET.[142,143]

Intramolecular charge transfer

Intramolecular charge transfer, ICT, occurs when an electron is transferred from a donor molecule to an acceptor molecule by light excitation forming a conjugated π system.[144] The occurrence of detection by ICT can be recognized by shifts in the emission and absorption wavelengths.[145] Chen et al., (2022) proposed (an ICT) mechanism for the detection of Cr (VI) using N- and S- doped CDs. A fluorescence enhancement was seen during the interaction of CDs and Cr (VI); this was attributed to the possible bonding of Cr (VI) to surface groups present on the surface of CDs, such as –SH. Additionally, the fluorescence lifetime of the CDs also increased and a blue shift in the absorbance wavelength of CDs, thus indicating ICT.[146] Further exploration of ICT-based systems for CDs is yet to be reported, however, these findings contribute to already existing mechanisms involved in fluorescence-based detection and offer potential applications in metal ion sensing.

Other mechanisms

Other sensing mechanisms from different works have also been proposed; Aggregation-induced emission is also one of the other commonly used mechanisms in CD sensors; Chaudhary et al.,(2020) hydrothermally prepared high quantum yield (32%), N, S- co-doped CDs from banana juice for selective detection of Cu^{2+} with a LOD of 0.3 ug/ml over a wide linear concentration range.[83] The fluorescence intensities of the N and S-CDs were significantly quenched by Cu^{2+} in water; as the concentration increased, a redshift in emission peak was also observed. The proposed mechanism was aggregation and charged transfer between N, S-CDs, and Cu^{2+}. Detection via other emerging mechanisms, such as twisted intramolecular charge transfer (TICT) and chelation-enhanced photoluminescence (CHEP), has yet to be explored further in CDs. However, the existing mechanisms have been proven to successfully allow CDs to detect different metal ions with high sensitivity and selectivity. Continued research in understanding and optimizing these mechanisms will further enhance the performance of BCDs.

DETECTION OF VARIOUS METAL IONS BY CARBON DOTS DERIVED FROM BIOMASS

Sensing metal ions, specifically heavy metals, in different media has become critically important due to the growing concerns about environmental contamination and their toxicity to living organisms. Heavy metals consist of metals that have high densities, such as aluminium (Al), lead (Pb), cadmium (Cd), zinc (Zn), iron (Fe), mercury (Hg), chromium (Cr), nickel (Ni), arsenic (As) and copper (Cu). While some naturally exist in the environment and play essential biological roles, increased concentrations or even trace amounts of these metals can result in adverse environmental impacts such as soil and water contamination, thus ultimately posing health threats to biological organisms due to the ease of bioaccumulation in organisms.[147,148] To mitigate these hazardous impacts, scientists continue to design and develop more efficient and less cumbersome ways of detecting metal ions using probes such as CDs. These are commonly used as fluorescent-based or colorimetric sensors; some recent work done towards metal ion detection using CDs derived from biomass is discussed in this review article.

Iron

Iron (Fe) is commonly known for its abundance in the environment and its important role in the transportation of oxygen and the synthesis of DNA in the body.[149] Many CD sensors have been developed in efforts to effectively and efficiently detect iron (Fe^{2+}/Fe^{3+}) as deficiencies/overloads of iron in the body may cause several disorders.[150] As such, monitoring of iron in biological and environmental samples has motivated the development of these low-cost BCD sensors that are discussed below: Kailasa et al., (2019) synthesized three different colored CDs from mixtures of tomato (*Solanum lycopersicum* fruit), H_2SO_4 and H_3PO_4 for the assaying and detection of Fe^{3+} ion in biological and pharmaceutical samples.[47] The resultant CDs were found to be selective towards Fe^{3+} ions in the presence of several other metal ions and anions. This was illustrated by the strong fluorescence quenching of the green-, blue-, and yellow-CDs with increasing concentrations of Fe^{3+} in solution. Fluorescence lifetime and zeta potential studies suggested that the detection of Fe^{3+} was attributed to dynamic quenching through the electron transfer between CDs and Fe^{3+}, resulting in the formation of complexes. Relatively low LODs of 0.065, 0.016, and 0.072 μM for the yellow, blue, and green fluorescing CDs were observed, respectively.

Similarly, Singh and coworkers synthesized bright-colored CDs from mango (*Mangifera indica*) leaves via a green one-pot pyrolysis method for the selective detection of metal ions such as Fe^{2+}.[75] The sensing potential of the blue CDs was investigated using a water sample containing various metal ions; the CDs were found to have strong selectivity towards Fe^{2+}. Further studies indicated significant quenching of the photoluminescence property of CDs by ferrous ions upon increasing concentration. This was attributed to a non-radiative relaxation process and photoluminescence quenching. Hence, effective sensing with an LOD of 0.62 ppm was achieved. Sensing studies were further investigated using a real sample in the form of a Livogen tablet containing ferrous fumarate; the same trend of fluorescence quenching with an increasing amount of Livogen was attained, thus validating the use of CDs as sensors of metal ions.

In the same year, another facile and one-pot hydrothermal synthesis of CDs using Seville orange as a fluorescent sensor for ferric ions in aqueous media was reported.[94] The pH-sensitive and water-soluble CDs exhibited selectivity and sensitivity towards Fe^{3+} in the presence of other anions and cations with an LOD of 0.53 μM. Addition of Fe^{3+} to the CDs resulted in fluorescence quenching attributed to the transfer of electrons between nitrogen-oxygen surface moieties of CDs and the ferric ion. Moreover, different concentrations of Fe^{3+} ranging between 4 μM and 20 μM in tap and groundwater were detected by the as-prepared CDs with good recoveries of 93.6-100 %, thus substantiating their use as a turn-off fluorometric sensor for ferric ions.

Architha and coworkers reported a microwave-assisted reflux synthesis of blue fluorescing CDs from Mexican mint leaves (*Plectranthus amboinicus*) for detecting Fe^{3+} and cell imaging application.[77] The mint leave derived CDs indicated maximum fluorescence quenching upon the addition of Fe^{3+} to the CDs solution compared to other metal ions that were also used, thus suggesting greater selectivity towards the ferric ion. Sensitivity studies of the CDs towards ferric ion illustrated a linear relationship between the fluorescence intensity and concentration of Fe^{3+}; an increasing concentration of Fe^{3+} from 0 μM to 15 μM with a LOD of 0.53 μM

resulted in a decrement of the fluorescence intensity. The mechanism of sensing was attributed to the electron transfer between CDs and Fe^{3+} ions, thus leading to the formation of complexes, hence the high sensitivity and selectivity towards ferric ions. Additional studies showed that the CDs exhibited a turn ON and OFF fluorescence, studies showed that their fluorescence was turned off by ferric ions whereas addition of ascorbic acid (AA) turned it ON. This was supported by the observed color change of the CDs solution from yellow to black upon the addition of Fe^{3+} and black to yellow in the presence of AA. Further works of logic gate implications, cytotoxicity and cell imaging were also reported.

Most recently, Nagaraj et al., (2022) reported a straightforward hydrothermal procedure for preparing CDs possessing good stability with zeta value of 37.3 mV, quantum yield of 19.4%, and PL properties.[93] These were prepared from the endosperm of *B. flaellifer* (ice apple) and used to successfully detect ferric ions in tap water and drinking water with outstanding recoveries of more than 98%. Selectivity studies indicated that the presence of ferric ion significantly quenched the fluorescence of the CDs whilst other cations caused little to no interference, thus indicating selective sensing of ferric ion with a LOD of 2.01 μM. The mechanism of detection was ascribed to the chelation of Fe^{3+} and oxygen-rich groups on the surface of the CDs.

Using another facile hydrothermal procedure, Krishnaiah et al., (2022) synthesized nitrogen doped CDs from *Poa pratensis* (Kentucky Bluegrass) and ethylenediamine as fluorescent probe for the sensing of ferric ion and manganese.[88] The N-CDs possessed several oxygen and nitrogen containing groups, which allowed for the effective sensing of these metal ions owing to the formation of coordination bonds between the surface moieties of the CDs and the metal ions, thus causing quenching of the fluorescence property of the CDs. A significant decrease in the fluorescence intensity was observed upon the addition of Mn^{2+} and Fe^{3+} to the CD solution with a linear relation to increasing concentrations of both metals. At higher concentrations of 25 μM of each metal, the fluorescence of CDs was observed to be turned off, with the fluorescence intensity being nearly zero. Additionally, Mn^{2+} ion was found to have a stronger quenching effect than Fe^{3+} as a visible color change in the CDs solution was observed in daylight, hence implying the use of bluegrass derived CDs as a colorimetric sensor for Mn^{2+} ions. The LOD for Mn^{2+} and Fe^{3+} were reported to be 1.2 μM and 1.4 μM, respectively.

Other biomass precursors such as cranberry bean,[85] waste tea,[86] rice residue,[89] and brewery waste[90] have also been used to synthesize CDs for Fe detection. This shows that the presented BCDs are effective fluorescent-based sensors that offer high sensitivity and selectivity towards iron under different conditions, making them promising in the field of environmental quality monitoring.

Mercury

Mercury (Hg) is often regarded as a non-biodegradable hazardous heavy metal despite its widespread use in the production of thermometers, electrical switches, fluorescent lamps, dental fillings, and cosmetic products.[151-153] Research has shown that exposure to various forms of mercury, Hg^{2+} MeHg and Hg^0, has the potential to cause Minamata disease in the human body that affects several neurological functions.[154] Moreover, Hg contamination is a significant environmental issue in aquatic systems because it readily bio-accumulates in aquatic organisms. As a result, detection of Hg is required, as evidenced by the numerous reports of its sensing employing CDs; some of the most current work in Hg detection is discussed.

Issa et al., (2020) reported a remarkable LOD of 0.01 μM for the detection of Hg^{2+} using N-CDs fabricated from oil palm empty fruit bunch carboxymethylcellulose (CMC) and urea.[100] A "turn off" effect on the fluorescence intensity and luminescence of N-CDs was observed in the presence of Hg^{2+} ions as the concentration increased from 0–50 μM. This was attributed to complex coordination between mercury ions and O- and N- groups on the surface of N-CDs. Application of the sensitive N-CDs as sensors using real water samples was conducted successfully, thus proving their efficiency as probes for mercury detection. In addition, adsorption studies for the removal of Hg^{2+} were explored, and yielded successful removal of mercury under different conditions.

Using broad bean and 1,2-ethanediamine as precursors, Xu et al., (2021) hydrothermally synthesized N-doped CDs for the selective and sensitive detection of Hg^{2+} in water samples with an LOD of 0.96 μM.[99] Interaction between Hg^{2+} and N-CDs indicated that the N-CDs were highly sensitive and selective towards Hg^{2+} as the fluorescence intensity was quenched significantly upon the addition of Hg^{2+} in the absence and presence of other interfering ions, thus indicating good selectivity. Furthermore, sensitivity tests showed a linear relationship between N-CDs and increasing concentration of Hg^{2+} in the range of 0–200 μM the N-CDs with a LOD of 0.96 μM. The mechanism of detection was attributed to static quenching which was suggested through fluorescence lifetime and absorption studies.

Also using static quenching mechanism, Sun et al., (2020) illustrated an efficient detection probe of Hg^{2+} using CDs synthesized from gardenia fruit.[95] The CDs exhibited oxygen, nitrogen, and sulfur containing functional groups, thus presenting several binding sites for the metal ion. During selectivity studies, addition of Hg^{2+} significantly quenched the fluorescence of CDs due to the complex formation of Hg^{2+} and N/S CDs. Absorption and fluorescence lifetime studies were explored to support the proposed mechanism as redshifts in wavelength were observed and no changes in fluorescence lifetime were noticed.

Exploring a wide range of Hg concentration, Hu et al., (2021) used CDs derived from bitter tea tree oil residue and Urea as a nitrogen source to detect Hg^{2+}.[101] FTIR spectra and XPS indicated the presence of pyridine and amine groups, providing sufficient nitrogen sites for Hg^{2+} to bind to. A decrease in fluorescence intensity was reported upon interaction with Hg^{2+} due to complexation between NCDs and Hg^{2+}. The CDs were also applied for sensing ferric ions. Based on the presented LOD data, the use of BCDs for Hg sensing has been demonstrated to be practical, effective, and efficient due to their high sensitivity, making them ideal for Hg^{2+} detection in real sample analysis.

Copper

Copper (Cu) is a naturally existing heavy metal that is abundant in nature and is considered one of the most essential metals biologically.[155] It serves as a mineral that supports enzymatic activities in both plants and the body. However excess amounts of it can cause adverse effects on organisms. Subsequently, there is a need to regulate Cu levels in the environment as well as in organisms. Thus, several studies have been undertaken in an effort to broaden the numerous ways of Cu detection. Amongst these sensing methods are CDs, which are becoming increasingly popular in Cu sensing.

Chauhan et al., (2020) developed a turn-on-off sensitive sensor for the dual detection of Cu^{2+} and Cd^{2+} in aqueous media using coconut coir as a precursor for CDs via thermal calcination. In this work, the fluorescence of CDs was quenched by Cu^{2+}, while in the presence of Cd^{2+}, an enhancement in the fluorescence intensity was observed. The differences in fluorescence intensities upon interaction with metal ions were suggested to be due to complexation with oxygen groups on the surface of CDs, thus resulting in an increase in non-radiative recombination of charges in the CDs hence fluorescence quenching. On the other hand, induction of intrinsic radiative combination decay rate was proposed as the cause for enhanced fluorescence. The LOD of Cu^{2+} and Cd^{2+} were calculated to be 0.28 and 0.18 nM, respectively.[84] This method can be efficiently used as a probe for dual detection of Cd^{2+} and Cu^{2+} in wastewater.

Through the PET mechanism, Cu^{2+} ions were detected in water samples using a fluorescent paper-based CD sensor.[92] The CDs were prepared hydrothermally using radish vegetable as a starting material;

the resultant CDs contained various functional groups through which chelation with Cu^{2+} could occur therefore forming a complex and hence causing fluorescence quenching of CDs. A linear relationship between CDs and concentration of Cu^{2+} over concentration range of 10-60 μM was reported with a LOD of 6.8 μM. Applications of the solid-state sensor in real water sample were reported. Further use of this sensor was applied in acetic acid vapor sensing.

Blue fluorescing CDs were synthesized via microwave irradiation using palm kernel shell and urea.[91] The metal sensing capabilities of CDs in aqueous media were explored, and it was observed that the interaction of Cu^{2+} and CDs resulted in the most significant fluorescence quenching of CDs. Detection of Cu^{2+} by CDs was carried out over a concentration range of 0 to 0.5 mM wherein decreasing fluorescence intensity was observed with increasing concentration; this phenomenon was attributed to electrostatic interactions between Cu^{2+} ions and CDs. The LOD was calculated to be 0.05 mM. Further application of these CDs was done in cell imaging in bacteria and fluorescence ink studies. The utilization of BCDs as Cu sensors provides an efficient and reliable way to monitor and regulate Cu levels in environmental and biological systems.

Lead

Lead (Pb) is a highly toxic metal ion that is widely used, resulting in several environmental concerns. Long time exposure to lead in the human body is known to cause harm to several organs including the brain, thus resulting in neurological dysfunctions and death in extreme cases.[156] The health impacts associated with lead are primarily the reason monitoring of lead is critical hence the constant need of development of new sensors. CDs are one of many newly emerging Pb^{2+} ion sensors that offer great selectivity and sensitivity; some of the works reported in Pb^{2+} sensing using BCDs are briefly outlined. A green synthesis of N-doped CDs derived from *Lantana camara* berries for sensing Pb^{2+} in aqueous media via dynamic quenching was reported by Bandi et al., (2018). These hydrothermally prepared CDs were found to possess hydroxyl and carboxyl functional groups on the surface of CDs hence their ability to successfully detect Pb^{2+}. Addition of Pb^{2+} solution to CDs resulted in a significant fluorescence quenching; an inversely related correlation was illustrated between the concentration of metal ions and the fluorescence intensity of CDs. Furthermore, the CDs were reported to have good sensitivity as detection of Pb^{2+} was carried out through a wide range of concentration of 0 mM to 3000 mM. The LOD was determined to be 9.64 mM.[104]

Similarly, Eswaran and coworkers reported a Pb^{2+} detection method through the use of highly fluorescent bread waste CDs synthesized hydrothermally.[108] Detection of Pb^{2+} was carried out over a concentration range of 4.0 to 56.6 μM with a LOD of 8.9 nM. Successful detection by CDs was signified by visible fluorescence quenching upon interaction with Pb^{2+} ions in solution, the fluorescence color of the CDs changed from green to colorless in the presence of Pb^{2+}. Changes in the absorption and fluorescence spectra were also observed, thus proving effective detection. The mechanism of detection was attributed to the aggregation of CDs particles after addition of Pb^{2+} ions caused by the binding of the Pb^{2+} ions to the carboxylate groups of CDs. The CDs were further applied in real sample analysis using river, tap, and drinking water; excellent recoveries of each were obtained, thus confirming the viability of using CDs as fluorescent-based sensors for Pb^{2+} ions.

Using *Volvariella volvacea* mushroom, Zulfarjri et al., (2020) hydrothermally synthesized CDs for sensitive dual detection of Pb^{2+} and Fe^{3+} in aqueous solutions. Selectivity studies showed that Pb^{2+} and Fe^{3+} ions were found to have a more significant quenching effect on the fluorescence of CDs in comparison to other metal ions and amino acids. However, the CDs were reported to have a stronger affinity for Pb^{2+} ions as naked eye changes were observed upon adding Pb^{2+} ions; a solution color change from brown to a white cloudy color was seen. Sensitivity studies were also carried out in the concentration range 1-1000 μM of Pb^{2+} and Fe^{3+} ions with good linearity in the concentration range of 1-100 μM. Higher concentrations of these metal ions resulted in greater fluorescence quenching with a LOD of 12 nM and 16 nM for Pb^{2+} and Fe^{3+}, respectively. Fluorescence quenching by Fe^{3+} and Pb^{2+} was attributed to the formation of a complex chelate between the surface groups of CDs and the metal ions. The CDs were further applied for real water sample analysis.[87]

Illustrating another Pb^{2+} naked eye sensor, Ansi and Renuka (2018) reported the use of table sugar derived CDs for the selective detection of Pb^{2+} ions in water with a LOD of 14 ppb. Addition of Pb^{2+} ions to the CDs resulted in turbidity of the solution hence serving as a remarkable visual detector of Pb^{2+}. Turbidity as a function of Pb^{2+} concentration was investigated, a progressive increase in turbidity observed with increasing concentration of Pb^{2+} ions, thus showing that CDs served as a selective and sensitive sensor. Detection of Pb^{2+} ion was attributed to aggregation of CDs particles in its presence; XRD, TEM, and EDAX data were used to support this mechanism. Application using real water samples was carried out to validate the use of table sugar CDs as an effective Pb^{2+} ion sensor.[97]

Through the use of pearl millet seed, Chauhan and coworkers (2021) demonstrated a colorimetric and fluorometric CD sensor for Pb^{2+} ion in water.[96] In this study, the interaction of Pb^{2+} and CDs resulted in an enhancement of fluorescence intensity of the CDs but interestingly also resulted in a color change of CDs from yellow to colorless, while in the presence of other metal ions, no color change was observed. Sensitivity studies in the range of 1 nM to 30 mM were carried out; a linear relationship of fluorescence enhancement as a function of increasing concentration of Pb^{2+} was observed with a LOD of 0.18 nM. The ability of CDs to serve as an effective sensor of Pb^{2+} was attributed to a charge transfer process and complexation of Pb^{2+} to the functional groups of CDs. Further biocompatibility studies using different biological assays were carried out.

Other biomass precursors such as oyster mushroom,[74] bamboo leaves,[80] *Ocimum sanctum* leaves,[107] and ginkgo biloba[81] leaves have also been used to synthesize CDs for the detection of Pb^{2+} ions. The above BCDs have presented compelling evidence that they can serve as effective and accurate sensors for detecting Pb^{2+} ions in water because of their low detection limits. Furthermore, the visible color changes observed in some other sensors provide a promising future in the rapid and in-situ analysis of Pb^{2+} in aqueous media.

Arsenic, Chromium and Cadmium

Arsenic (As), chromium (Cr), and cadmium (Cd) are toxic metal ions that have significant environmental importance. The prolonged exposure to these may result in carcinogenic effects causing kidney dysfunctions, lung diseases, and severe damage to other organs in the body.[157,158] As such, rapid and effective detection of these ions in the environment is essential. The use of CDs as probable sensors for As^{3+}, Cr^{6+} and Cd^{2+} is discussed. For instance, Cr (VI) was detected via IFE mechanism and static quenching using CDs synthesized from *Poria cocos* polysaccharide.[105] It was shown that Cr (VI) caused a turn-off effect on the fluorescence of CDs as its concentration increased from 1-100 μM with a LOD of 0.25 μM. The green CDs were further applied in real water samples where recoveries of 82% and above for each tested water sample were recorded, thus substantiating their use as a promising sensing probe for the detection of Cr (VI). Cytotoxicity, fluorescence lifetime, and zeta potential studies were discussed.

In a related study, selective detection of Co^{2+} and Cr^{6+} was illustrated by Hu and associates using flax straw CDs. The hydrothermally prepared CDs consisted of nitrogen and oxygen containing groups giving them the ability to bind to these metal ions.[109] Addition of Co^{2+} and Cr^{6+} to the CDs solution caused the fluorescence to decrease significantly as the concentrations increased from 0-500 μM for both metal ions. Fluorescence quenching of CDs by both metal ions was attributed to IFE as unchanged fluorescence lifetime and overlapping spectra of the absorption spectrum of CD-Co^{2+}/CD-Cr^{6+} and the emission spectrum of CDs was observed. The LODs of Co^{2+} and

Cr^{6+} were calculated to be 0.38 and 0.19 μM, respectively; CDs were successfully applied in real water samples and further studies for detecting ascorbic acid were also reported.

Using water amaranth leaves, CDs modified with pyrene carboxaldehyde, Keerthana et al., (2023) reported a ratiometric detection of Cd^{2+} in wastewater from battery and plastic industries. The modified CDs exhibited nitrogen-rich groups that enabled the interaction with Cd^{2+} through binding, resulting in fluorescence quenching via the FRET mechanism. Increasing concentration from 0 - 70 μM of Cd^{2+} in CDs solution resulted in fluorescent gradual color changes from blue to red under UV light at 365 nm, thus illustrating the capacity of the CDs to serve as a ratiometric sensor for Cd^{2+}. Furthermore, the proposed CDs were found to be highly selective and sensitive towards Cd^{2+} with a LOD of 15 nM.[106]

Similarly, Huang and coworkers proposed a solvothermal synthesis of CDs from tea residue and choline chloride/urea for the detection of Cd^{2+}.[82] Detection of Cd^{2+} was carried out over a concentration range of 0 to 20 μg/mL with a calculated LOD of 2.14 μg/mL. To further validate their efficiency, real sample analysis using artificial lake water was demonstrated wherein good recoveries of 93% and above were achieved, proving viability as a Cd^{2+} ion sensor. The fluorescence sensing mechanism was attributed to chelate formation between surface groups of CDs and Cd^{2+} which resulted in fluorescence quenching.

Radhakrishnan and Panneerselvam (2018) presented a green synthesis of glutathione passivated CDs derived from prickly pear cactus for dual detection of As^{3+} in drinking water.[79] The presence of As^{3+} in CDs solution resulted in fluorescence quenching due to a non-radiative electron transfer process between the two species. The CDs were reported to be selective and sensitive towards As^{3+} with a LOD of 2.3 nM in the concentration range of 0-30 nM. The CDs were also effectively used to detect the ClO^{4-} ion in drinking water, demonstrating their potential for dual sensing for both ions in water.

In the same year, Ramenzani et al., (2018) reported application of quince fruit powder (*Cynodia oblonga*) derived CDs for the detection and cell imaging of As^{3+}. Selectivity studies revealed that As^{3+}, Fe^{3+}, and MnO^{4-} had a strong quenching effect on the fluorescence of microwave synthesized CDs. However, these interferences were removed using a masking agent. Further studies revealed that in the presence of a mixed MnO^{4-} and As^{3+} solution, fluorescence enhancement of CDs occurred due to the recombination of electron pair holes. A linearity between fluorescence enhancement and increasing concentration of As^{3+} in the range of 0.1 to 2.0 μg/mL was established. The CDs were further applied in real water analysis wherein a LOD of 0.04 μg/mL was determined.[99] Overall, the above BCDs serve as versatile and efficient sensors for heavy metal ions owing to their remarkable fluorescence behavior.

Other metal ions

While most research on biomass derived CDs and their sensing applications is mostly focused on sensing Fe^{3+}, Hg^{2+}, Pb^{2+}, and Cu^{2+}, there have been some reports of the detection of other metal ions. Fu et al., (2022) recently reported a method to detect and separate several metal ions in water using three different CDs derived from three types of animal bone, namely pork bone (PBCDs), bovine bone (BBCDs) and sheep bone (SBCDs), using hydrothermal synthesis.[103] From XPS data, all three Bone CDs contained Ca, N, O, S and C in different quantities hence different quenching effects of fluorescence intensity of CDs were shown upon interaction with metal ions (Ag^{+}, Cu^{2+}, Hg^{2+}, Fe^{3+} and Pb^{2+}). Although all metal ions caused a quenching effect of the fluorescence intensity of CDs, Fe^{3+} was shown to have the greatest quenching effect on the CDs, while Ag^{+} was reported to have the greatest quenching effect on SBCDs. The BCDs were successfully applied to completely distinguish these metal ions from one another in water samples using linear discriminate analysis and hierarchical analysis.

Detection of Co^{2+} in water was reported by Zhao and associates via microwave assisted synthesis of CDs fabricated from kelp and ethylenediamine as precursors.[110] Addition of Co^{2+} to the CDs resulted in fluorescent and colorimetric changes; a visual color change from brownish yellow to dark blue under UV light was seen, while the fluorescence intensity also decreased in concentration ranges of 10-200 μM of Co^{2+} with a detection limit of 0.39 μM. The sensing abilities of the CDs were attributed to IFE mechanism as proven by absorption spectra and fluorescence lifetime studies. Moreover, good recoveries obtained from real sample detection validated the use of kelp CDs as reliable sensors for Co^{2+}. In a notable study by Chen et al., (2022), Ru^{3+} was detected using starch CDs prepared via pyrolysis method. Upon interaction with various metal ions, the fluorescence intensity of the starch CDs was significantly quenched in the presence of Ru^{3+} compared to other metal ions. Sensitivity studies of the CDs towards Ru^{3+} showed that the fluorescence intensity of the CDs responded linearly to different concentrations of Ru^{3+} in the range 0-1000 uM with a LOD below 100 uM. The fluorescence quenching of the CDs by Ru^{3+} was attributed to changes in electronic structures of the CDs that occurred during their interaction. [111]

Using yeast *Cryptococcus podzolicus* CDs synthesized hydrothermally, detection of Ag(I) in aqueous media was reported by Ji and team.[112] In the presence of Ag(I) ions, the fluorescence intensity of CDs was seen to decrease with increasing concentration of Ag(I) in the range 0 – 15 uM with a LOD of 113.57 nM. Furthermore, the viability of the CDs as sensors of Ag(I) was tested by analysis in real water samples: tap, waste and river water. Good recoveries of 97.33-99.67% were obtained. Their successful detection was attributed to static quenching as suggested by the UV-Vis spectra, fluorescence lifetime measurements and Stern-Volmer analysis.

In a different study, Zn^{2+} detection in aqueous media using CDs fabricated from coconut water was presented by Jayan et al., (2021). As expected, the fluorescence intensity was quenched upon interaction with the metal. Antibacterial studies were also reported.[102] In conclusion, these studies have collectively illustrated the ability of BCDs to detect a wide array of metal ions through fluorescence quenching or enhancement in real water samples with low detection limits, high recoveries, good selectivity, and dual detection capabilities therefore indicating their practical applicability in the field of metal ion sensing.

SHORTFALLS AND FUTURE PERSPECTIVES

The research work summarized in this review has demonstrated the impressive application of biomass derived carbon dots for metal ion sensing in diverse environments. Their changeable fluorescent properties have paved the way for effective sensing, with low detection limits and, in some instances, the ability to detect multiple ions. While BCDs may show significant potential in the metal ion sensing field, some challenges remain yet to be addressed. Firstly, BCDs are presented as potential sensors for metal ions, offering several advantages, such as ease of use and low-cost benefits over existing instrumental analysis methods. However, their production remains at a small scale; therefore, issues concerning commercialization, and the feasibility of upscale production are still unattended. This leaves a question of their reliability as metal ion sensors in daily real-life applications. Secondly, current literature is focused on specific sets of metals, such as Fe^{3+} and Hg^{2+}, while few reports on other environmentally significant ions, such as cadmium, have been published. Therefore, more research is required to understand the selective interaction between BCDs and metal ions to fabricate target-specific BCDs. Moreover, it is also worth noting that biomass derived CDs are known to have relatively low quantum yields compared to chemically synthesized CDs; improvements in this aspect are also necessary for better performing CDs. However, despite these challenges, biomass carbon dots remain a subject of interest and hold great potential in the detection of metal ions.

CONCLUSIONS

Development of sensitive, selective and cost-effective sensors for metal ion detection is a continuous process for scientists and researchers. This review has highlighted the recent developments in the field of biomass derived carbond dots (BCDs), a new carbon-based nanomaterials, and their continued efforts in the detection of various metal ions. As a natural renewable resource, biomass can play a significant role in the production of carbon dots and their application towards fluorescence sensing of metal ion in various sample matrices. It has been demonstrated that BCDs can be synthesized at low cost because of abundant availability of biomass and applied towards metal ion detection. Therefore, the ease and time saving of their production coupled with their striking reacting ability with a wide array of metal ions make them a suitable candidate for metal ion detection. Although the mechanisms responsible for metal ion detection are not yet understood it is envisaged that carbon dots will create more exciting opportunities for environmental pollution monitoring. Undoubtedly, research in using BCDs for metal ion sensing continues to gain considerable attention, resulting in the development of more efficient and highly sensitive sensors. However, further exploration of key areas such as surface functionalization, selection of precursors of BCD, synthesis methods for target-specific sensing, and mechanisms that govern the interaction between metal ions and BCDs is still required. Such developments will offer interesting avenues for the future of environmental monitoring and may offer solutions to the shortcomings discussed.

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