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Optimizing Carbon Dots for Enhanced Lead Removal from Aqueous Solutions: A Novel Approach Using Hydrothermal Synthesis with Tartaric and Oxalic Acids

Alaa R. Said[1] , Mohamed M. Ghobashy[2*], Ahmed Siddiq[3], Ahmed A. Zaher[4,5], Sadeek A. Sadeek[1]

[1]*Department of Chemistry, Faculty of Science, Zagazig University, Zagazig, Egypt*
[2]*Radiation Research of Polymer Chemistry Department, National Center for Radiation Research and Technology (NCRRT), Egyptian Atomic Energy Authority (EAEA), Cairo, Egypt*
[3]*Department of Chemistry, Faculty of Science, Al-Azhar University, 71524, Assiut, Egypt*
[4]*Main Chemical Laboratories, Egyptian Army, Egypt.*
[5]*Chemistry Department, Faculty of Science, El-Mansoura University, El-Mansoura 35516, Egypt.*

Abstract

This study explores the synthesis of carbon dots (CDs) using a novel hydrothermal method with tartaric and oxalic acids as precursors. Three distinct samples, CD-1, CD-2, and CD-3 were synthesized with varying precursor ratios (1:1, 1:2, and 2:1) to investigate their impact on the CDs' physicochemical properties and lead ion removal efficiency. Fourier-transform infrared (FTIR) spectroscopy revealed significant surface functionalization, with CD-3 displaying the highest O-H stretching intensity at 3400 cm^{-1}. Transmission electron microscopy (TEM) confirmed quasi-spherical CDs with sizes ranging from 4 to 8 nm, with CD-1 showing the most uniform distribution (5-6 nm average). Raman spectroscopy indicated varying degrees of graphitization, with CD-3 exhibiting the lowest I_D/I_G ratio of 0.85, suggesting a more ordered carbon structure. Notably, CD-1 demonstrated a superior lead ion removal efficiency of 77.2%, significantly higher than CD-2 (27.5%) and CD-3 (71.2%). The optimum precursor ratio was determined to be 1:1 (tartaric acid: oxalic acid) in CD-1, balancing surface functionalization and structural order, thereby enhancing adsorption properties. This study underscores the importance of precursor ratio optimization in tailoring CD properties for environmental remediation applications.

Keywords: *Hydrothermal synthesis; Carbon dot; Lead removal; Characterization.*

1. Introduction

Carbon dots (CDs) have emerged as a fascinating class of carbon-based nanomaterials, garnering significant attention in the scientific community due to their unique physicochemical properties and versatile applications [1-3]. Since their serendipitous discovery during the purification of carbon nanotubes in 2004, CDs have rapidly evolved into a distinct research field, offering promising solutions in areas ranging from bioimaging and sensing to catalysis and environmental remediation [4-6]. The allure of CDs lies in their exceptional characteristics, including strong photoluminescence, excellent biocompatibility, chemical inertness, and facile synthesis methods. The defining features of CDs include their small size, typically below 10 nm in diameter, and their quasi-spherical morphology. This nanoscale dimension endows CDs with quantum confinement effects, largely responsible for their unique optical properties [7-9]. The photoluminescence of CDs, characterized by high quantum yields, broad excitation spectra, and tuneable emission, has been the subject of intense research. Unlike traditional semiconductor quantum dots, the luminescence mechanism in CDs is more complex, involving both core states and surface-related emissive traps. This complexity offers opportunities for fine-tuning optical properties through careful control of synthesis parameters and post-synthesis modifications [10-13]. One of the most attractive aspects of CDs is the diversity of synthesis methods available for their production. Among these, the hydrothermal method has gained prominence due to its simplicity, scalability, and ability to produce high-quality CDs with controlled properties. The hydrothermal approach involves carbonizing organic precursors in a sealed autoclave under elevated temperature and pressure. This method offers several advantages, including eco-friendly precursors, the ability to conduct one-pot synthesis and functionalization, and the potential for large-scale production [14, 15].

The choice of precursors in CD synthesis plays a crucial role in determining the final properties of the nanoparticles [16, 17]. Organic acids, such as citric acid, tartaric acid, and oxalic acid, have been widely used as carbon sources due to their ability to undergo controlled carbonization and their inherent functional groups that can be incorporated into the CD structure. The rationale behind using tartaric and oxalic acid in the present study stems from their complementary structures and functionalities. With its four carbon atoms and two hydroxyl groups, Tartaric acid can contribute to forming a carbon core while providing abundant surface hydroxyl functionalities. Oxalic acid, the simplest dicarboxylic acid, can act as a strong reducing

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*Corresponding author e-mail: Mohamed_ghobashy@yahoo.com.; **(Mohamed M. Ghobashy).**

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agent and influence the carbonization process, potentially affecting the size and crystallinity of the resulting CDs. The concept of doping CDs with heteroatoms has gained traction in recent years as a strategy to enhance their properties further and expand their application potential. Nitrogen doping, in particular, has been shown to significantly improve the quantum yield and tune the emission wavelength of CDs. Incorporating nitrogen atoms into the carbon lattice creates new energy levels and modifies the electronic structure of CDs, leading to enhanced optical properties [18, 19].

The environmental application of CDs, particularly in heavy metal ion removal, has gained increasing attention due to the pressing need for efficient and sustainable water treatment technologies. Lead contamination, in particular, remains a significant environmental and public health concern worldwide [20]. CDs high surface area-to-volume ratio and abundant surface functional groups make them promising candidates for heavy metal adsorption. The ability to tailor the surface chemistry of CDs through precursor ratio optimization offers a unique opportunity to enhance their affinity for lead ions and improve removal efficiency. Characterizing CDs is fundamental to understanding their structure-property relationships and optimizing their performance in various applications [21-23]. Fourier-transform infrared (FTIR) spectroscopy provides valuable insights into the surface functional groups on CDs, which play a crucial role in determining their solubility, reactivity, and adsorption properties. The analysis of FTIR spectra can reveal the presence and relative abundance of key functionalities such as hydroxyl, carboxyl, and carbonyl groups, which are essential for metal ion binding [24].

Recent research has demonstrated the versatility of photografting polymerization for creating functionalized polymer films with metallic nanoparticles, highlighting the potential of such materials for optoelectronic applications[25]. Complementary studies have explored the integration of active compounds into polymer matrices, showcasing the effectiveness of polymeric systems for controlled delivery applications[26]. Furthermore, polymers with tailored chemical functionalities can significantly enhance performance under challenging environmental conditions, suggesting similar principles may apply to carbon-based nanomaterials for environmental remediation[27] [28].

The synthesis of carbon dots (CDs) for environmental remediation presents challenges in optimizing precursor ratios and surface chemistry to maximize heavy metal removal efficiency. This study systematically investigates the effects of single-doping with organic acids, focusing on controlled functionalization to enhance Pb^{2+} adsorption. Unlike later research exploring dual-doping strategies with nitrogen and rare earth metals for biomedical applications [29], our approach prioritizes environmentally relevant applications by fine-tuning oxygen-containing functional groups. Through hydrothermal synthesis, we evaluate how tartaric and oxalic acid ratios influence the adsorption capacity, graphitic order, and surface charge of CDs. The findings provide insights into the interplay between precursor composition and heavy metal sequestration, highlighting the role of carboxyl and hydroxyl groups in Pb^{2+} binding. This work establishes a foundation for tailoring CD properties for sustainable water treatment, differentiating it from studies emphasizing optical tuning and antimicrobial functionalities through complex doping mechanisms.

2. Experimental

2.1. Material

All chemicals were purchased from Sigma-Aldrich at high purity grades: Oxalic Acid (>99.5%), Tartaric acid

2.2. Preparation of Carbon Dots (CDs) by Hydrothermal/Solvothermal Method

The preparation of Carbon Dots (CDs) detailed in **Table 1** involves a hydrothermal/solvothermal method using tartaric acid and oxalic acid as precursor materials. This method is advantageous due to its simplicity and efficiency in producing CDs with controlled properties. The process begins with carefully weighing tartaric acid and oxalic acid, which are then dissolved in 20 mL of deionized water to form a clear, homogeneous solution. The proportions of these precursors vary across three distinct samples: CD-1 uses an equal mass of tartaric acid and oxalic acid (3g each), CD-2 uses more oxalic acid (2g tartaric acid and 4g oxalic acid), and CD-3 reverses this ratio with more tartaric acid (4g tartaric acid and 2g oxalic acid). Once the precursor solution is prepared, it is transferred into a Teflon-lined stainless-steel autoclave. The autoclave is then sealed and subjected to hydrothermal treatment at 180°C. This temperature is critical as it is high enough to induce the carbonization of the organic precursors, yet below the thermal decomposition temperature of the functional groups that contribute to the CDs' unique properties. The reaction time is also carefully controlled, typically lasting for several hours (commonly two to five hours, depending on the desired outcome), during which the pressure inside the autoclave increases due to the boiling water, creating a superheated environment conducive to the formation of carbon nanoparticles [30, 31]. The high-temperature conditions within the autoclave facilitate the organic molecules' dehydration, polymerization, and carbonization, forming nanoscale carbon dots. The reaction by-products, such as gases and other volatile substances, are contained within the autoclave, preventing contamination and ensuring a high purity of the final product. Upon completion of the reaction, the autoclave is cooled to room temperature naturally, preventing any sudden temperature changes that could affect the structural integrity of the CDs.

Table 1. The detailed process for the preparation of Carbon Dots (CDs) using a hydrothermal/solvothermal method

	Tartaric acid	**Oxalic acid**
CD-1	3g	3g
CD-2	2g	4g
CD-3	4g	2g

2.3. Material Characterization

In this study, a comprehensive characterization of the synthesized CDs was conducted using advanced analytical techniques to elucidate their structural, and morphological, and evaluate their possibility for lead removal from aqueous solutions. The synthesized CDs were characterized using FTIR spectroscopy, scanning electron microscope (SEM), and transmission electron microscopy (TEM).

3. Result and discussion

3.1. FTIR analysis of Synthesized Carbon Dots

Figure 1 presents the synthesized CDs' Fourier Transform Infrared (FTIR) spectra, specifically samples CD-1, CD-2, and CD-3. FTIR spectroscopy is a critical analytical technique used to identify the functional groups on the surface of the CDs, which are crucial for understanding their chemical properties, reactivity, and potential applications. The FTIR spectra provide a detailed overview of the CDs' surface chemistry, revealing the bonds' nature and the types of functional groups that have been formed during the hydrothermal synthesis. The FTIR spectrum of CD-1 shows a broad absorption band centered around 3191 cm^{-1}, corresponding to the O-H stretching vibrations of hydroxyl groups. This peak indicates the presence of surface-bound water molecules or hydroxyl groups that result from the hydration of the CD surface during the synthesis process.

These hydroxyl groups play a significant role in the solubility of CDs in water and contribute to their biocompatibility, making them suitable for applications in biological environments. Further analysis of CD-1 reveals strong absorption bands around 1670 cm^{-1} and 1638 cm^{-1}, corresponding to the stretching vibrations of carbonyl groups (C=O), characteristic of carboxylic acids or esters. The presence of these groups suggests that the surface of the CDs is rich in oxygen-containing functional groups, which can influence the CDs' reactivity, ability to form hydrogen bonds, and photoluminescent properties. These functional groups are typically introduced during the carbonization of the organic precursors and are essential for the CDs' photophysical behavior, particularly in tuning their emission properties.

Comparing the FTIR spectra of CD-1 with those of CD-2 and CD-3, it is evident that while the general functional groups are similar, there are subtle differences in the peak positions and intensities [32-36]. For CD-2, the O-H stretching band is slightly shifted, and there is a noticeable difference in the intensity of the carbonyl stretching bands. This suggests that the surface chemistry of CD-2 is slightly altered, likely due to the higher oxalic acid content, which may lead to a different carbonization pathway and surface functionalization. The FTIR spectrum of CD-3 also shows variations, particularly in the region of 1000-1500 cm^{-1}, where C-O stretching vibrations are observed, indicating the formation of ether or ester groups.

These variations in the FTIR spectra highlight the impact of precursor ratios on the CDs' surface chemistry. The differences in functional group composition and distribution can significantly influence the CDs' optical properties, understanding these differences is essential for tailoring the surface properties of CDs for specific applications, such as in sensing, where surface chemistry plays a crucial role in detecting target molecules.

3.2. SEM analysis of Carbon Dots

Figure 2 presents the Scanning Electron Microscopy (SEM) images of the CDs (CD-1, CD-2, and CD-3), which provide detailed insights into the synthesized nanoparticles' morphology, size distribution, and surface characteristics. SEM is an indispensable tool for characterizing nanomaterials, offering high-resolution images that reveal particles' shape, size, and surface texture at the nanoscale. The SEM image of CD-1 reveals a population of quasi-spherical nanoparticles with a relatively uniform size distribution. The particles are well-dispersed, with minimal aggregation observed, suggesting that the synthesis process effectively prevented the particles from clumping together. This uniformity in size is crucial for applications where consistency in particle behaviour is necessary, such as in drug delivery systems or as fluorescent probes in bioimaging. The average size of the nanoparticles in CD-1 is 5 to 10 nm, typical for CDs and contributes to their unique optical properties, including their size-dependent photoluminescence. In the case of CD-2, the SEM image shows a slightly different morphology than CD-1.

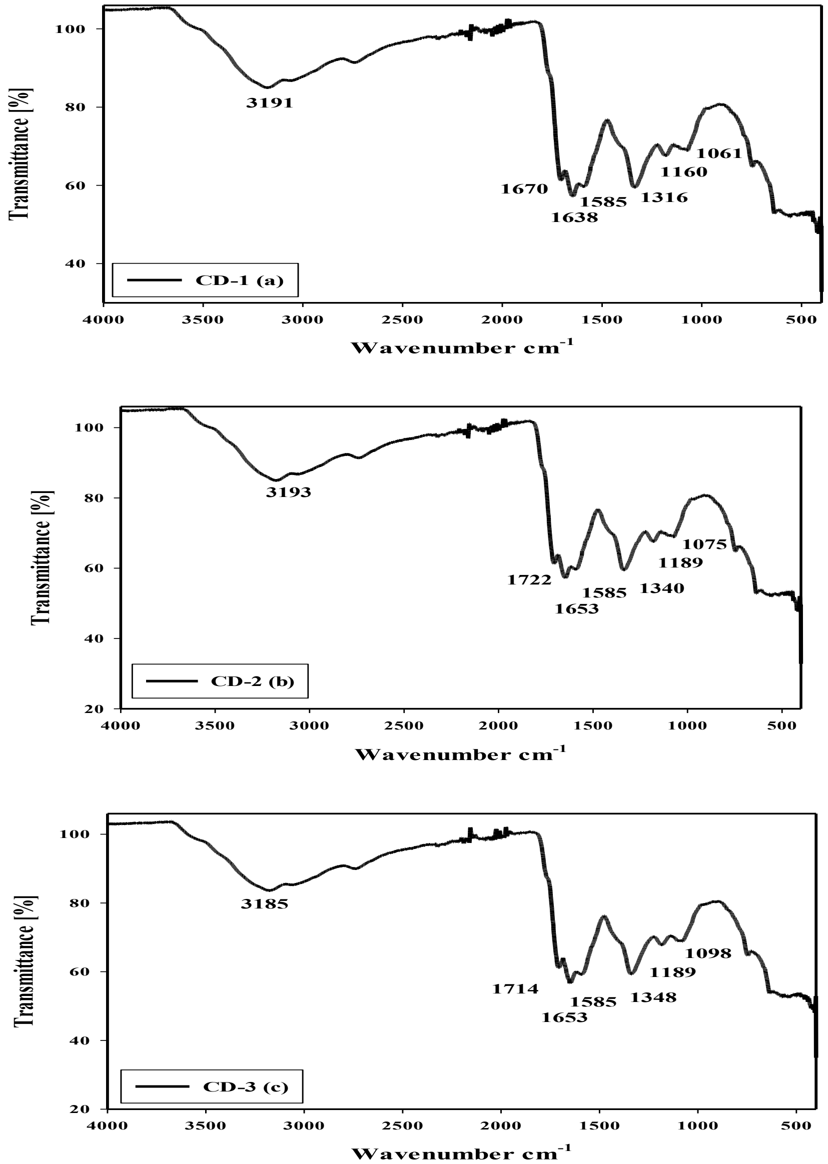

Figure 1. FTIR of synthesized (a)CD-1, (b) CD-2 and (C) CD-3

The particles are still quasi-spherical, but there is a noticeable increase in size variability, with some particles appearing larger than those in CD-1. This increased variability in size could be attributed to the higher oxalic acid content used in synthesizing CD-2, which may have affected the nucleation and growth processes, leading to a broader distribution of particle sizes. Additionally, some degree of aggregation is observed, possibly due to differences in the surface chemistry or residual precursor materials that promote particle clumping. On the other hand, the SEM image of CD-3 shows particles with size distribution and morphology that are more similar to CD-1 but with slightly larger average particle sizes. The increased tartaric acid content in CD-3 likely influenced the carbonization process, resulting in more extensive but relatively uniform nanoparticles. The surface of these particles appears smooth and well-defined, indicating that the synthesis process was well-controlled and that the particles formed with minimal defects. The differences observed in the SEM images of CD-1, CD-2, and CD-3 highlight the impact of the precursor composition on the morphology and size distribution. These morphological characteristics are crucial because they directly influence the optical and electronic properties of the CDs. For instance, smaller and more uniform particles typically exhibit more substantial and consistent photoluminescence, making them more suitable for applications in optoelectronics and bioimaging [36, 37]. Conversely, larger particles or those with a broader size distribution may have more varied properties, which could be advantageous in applications requiring a range of behaviors, such as catalytic processes [38, 39]. Overall, the SEM analysis provides valuable insights into how the synthesis conditions, particularly the precursor ratios, influence the physical characteristics of the CDs. These insights are essential for optimizing the synthesis process to produce CDs with the desired properties for specific applications.

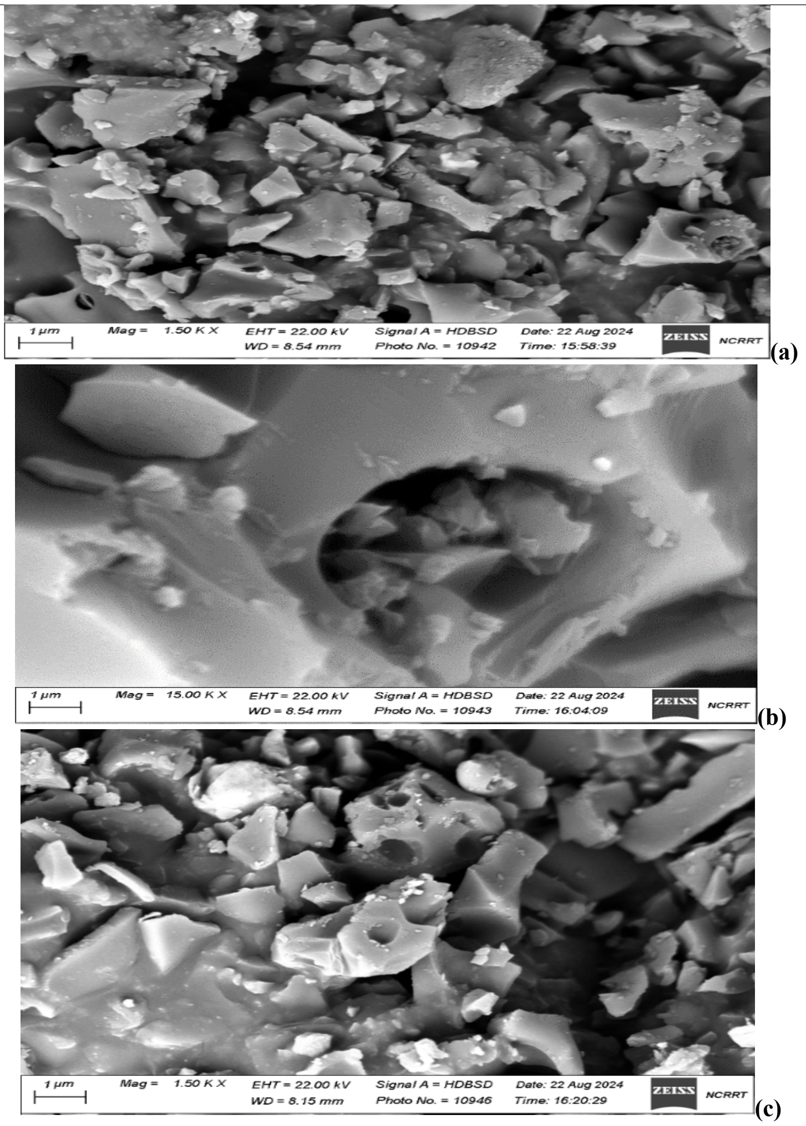

Figure 2. The SEM images of **(a)** CD-1, **(b)** CD-2 and **(c)** CD-3

3.3. TEM analysis of Synthesized Carbon Dots

Figure 3 presents the Transmission Electron Microscopy (TEM) images of the CDs (CD-1, CD-2, and CD-3), offering a closer look at their internal structure, size, and crystallinity. TEM is a high-resolution imaging technique that allows for the visualization of nanoparticles at the atomic level, providing critical insights into their structural and morphological properties. The TEM image of CD-1 shows that the nanoparticles have a well-defined core with a combination of amorphous and crystalline regions. The size of the particles observed in the TEM image is consistent with the SEM results, with most particles being below 10 nm in diameter. The presence of crystalline regions within the particles suggests that during the hydrothermal synthesis, partial graphitization occurred, leading to the formation of ordered carbon structures within the otherwise amorphous matrix [40-42]. This partial crystallinity is crucial for the optical properties of the CDs, particularly their photoluminescence, as the crystalline domains can act as sites for localized electronic states that enhance light emission. For CD-2, the TEM image reveals a more amorphous structure, with fewer and less distinct crystalline regions compared to CD-1. The particles appear slightly larger and show more variability in size, which aligns with the observations from the SEM analysis. The increased amorphous nature of CD-2 could be due to the higher oxalic acid content used in its synthesis, which may have influenced the carbonization process, leading to a less ordered structure. The lack of crystalline domains in CD-2 could negatively affect its photoluminescent properties, as fewer sites for electronic transitions are available, potentially reducing its emission intensity.

In contrast, the TEM image of CD-3 shows a structure that is more similar to CD-1, with a mix of amorphous and crystalline regions. However, the particles in CD-3 are slightly larger, consistent with the increased tartaric acid content used in its synthesis. The presence of crystalline regions within the particles suggests that the synthesis conditions were favorable for partial graphitization, resulting in CDs with potentially enhanced optical properties. The balance between amorphous and crystalline domains in CD-3 is essential for achieving a combination of strong photoluminescence and stability. The TEM analysis of the three CD samples highlights the significant influence of precursor composition on the internal structure and crystallinity of the CDs. These structural properties are vital for their performance in various applications, particularly in optoelectronic devices, where the degree of crystallinity can directly impact the efficiency of light emission and the stability of the material. Understanding the relationship between the synthesis conditions and the resulting structure of the CDs is crucial for designing materials with tailored properties for specific technological applications.

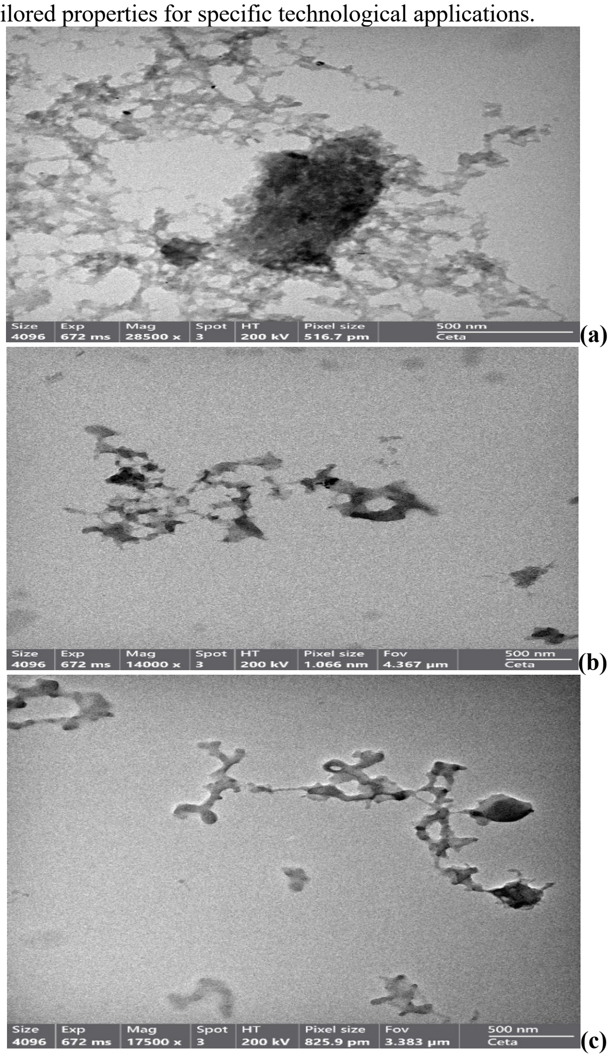

Figure 3. the TEM image of **(a)** CD-1, **(b)** CD-2 and **(c)** CD-3

3.4. FT-Raman analysis of Synthesized Carbon Dots

Figure 4 presents the FT-Raman spectra of the CDs (CD-1, CD-2, and CD-3), providing detailed insights into their vibrational modes and the carbon structure. Raman spectroscopy is an essential tool for characterizing carbon-based materials, as it provides information on the crystallinity, disorder, and the presence of defects within the carbon lattice. The Raman spectrum of CD-1 shows prominent peaks corresponding to the D and G bands, located at approximately 1365 cm^{-1} and 1576 cm^{-1}, respectively. The G band is associated with the in-plane stretching of sp^2 hybridized carbon atoms in graphitic materials, indicating the presence of ordered, graphitic structures within the CDs. The D band, conversely, is related to the presence of defects and disorder in the carbon lattice, typically arising from the presence of sp^3 hybridized carbon atoms, vacancies, or other structural imperfections. The intensity ratio of the D and G bands (ID/IG) is a critical parameter in evaluating the degree of disorder in carbon materials. For CD-1, the ID/IG ratio suggests a moderate level of disorder, indicating that the CDs contain a mixture of ordered and disordered carbon structures. This combination is beneficial for applications where high conductivity (associated with ordered graphitic domains) and enhanced surface reactivity (due to defects and disorder) are required. In the case of CD-2, the Raman spectrum shows a higher ID/IG ratio than CD-1, indicating a greater degree of disorder in the carbon lattice. This increased disorder is likely due to the higher oxalic acid content used in the synthesis, which may have formed more defects during the carbonization process. The higher degree of disorder in CD-2 could impact its optical properties, potentially leading to lower photoluminescence efficiency but higher reactivity, which might be advantageous in catalytic applications.

The Raman spectrum of CD-3, similar to CD-1, shows both D and G bands, with an ID/IG ratio that suggests a lower degree of disorder than CD-2 but higher than CD-1. This intermediate level of disorder reflects the influence of the tartaric acid content in the synthesis, which may have promoted the formation of fewer defects compared to CD-2, while still allowing for a degree of disorder that can enhance surface reactivity.

The FT-Raman analysis provides valuable insights into the structural properties of the CDs, particularly regarding the balance between order and disorder within the carbon lattice. This balance is crucial for determining the optical and electronic properties of the CDs, as well as their suitability for various applications [43-45]. For instance, CDs with a higher degree of graphitization may be more suitable for applications requiring high electrical conductivity, such as electronic devices. At the same time, those with more disorder may be better suited for applications requiring high surface reactivity, such as in catalysis or sensing.

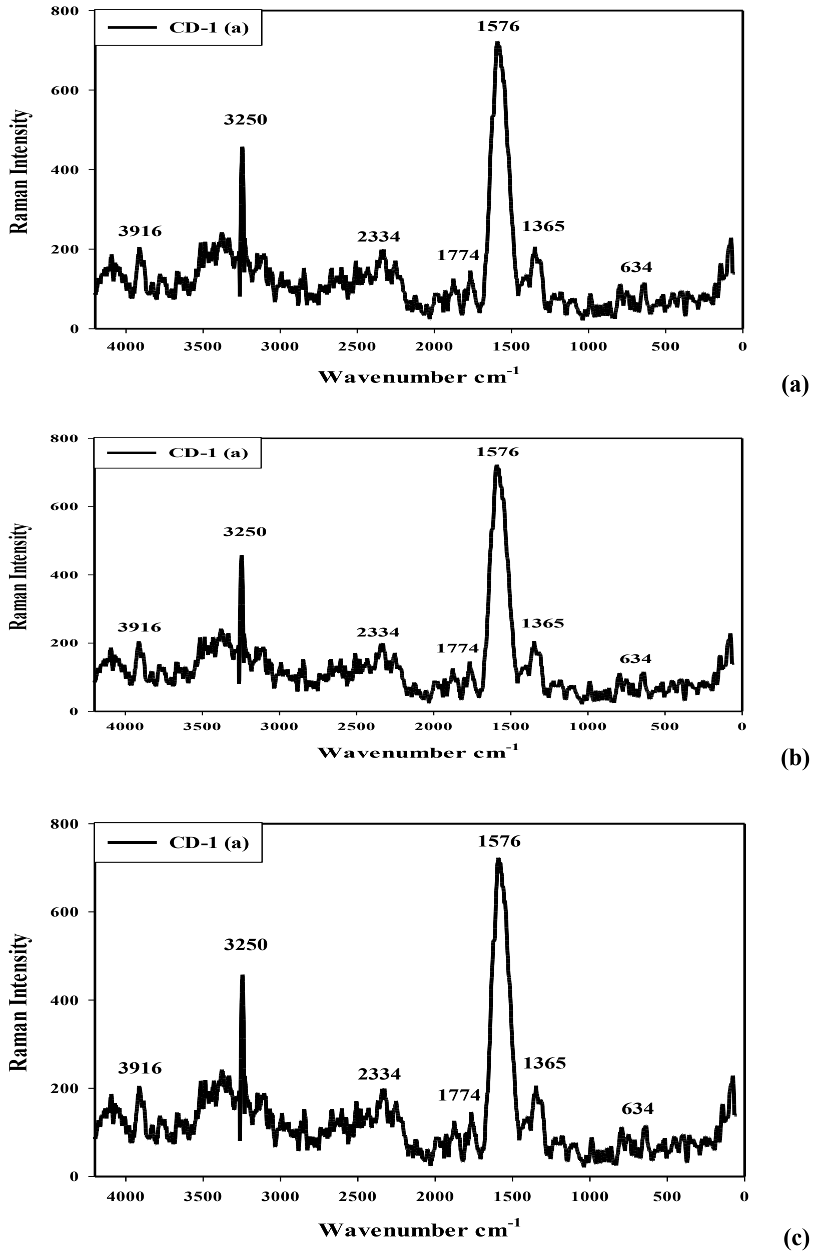

Figure 4. The FT-Raman spectroscopy of **(a)** CD-1 , **(b)** CD-2 and **(c)** CD-3

3.5. Removal of Lead ions from aqueous solution

Table 2 presents the data on the removal efficiency of lead ions (Pb^{2+}) from an aqueous solution using the synthesized CDs (CD-1, CD-2, and CD-3). The removal efficiency is a critical parameter for evaluating the potential of CDs as adsorbents in environmental remediation applications, particularly for removing heavy metals from contaminated water sources [46, 47]. The data in **Table 2** shows that CD-1 exhibits the highest removal efficiency, with 77.2% of lead ions removed from the solution. This high efficiency can be attributed to the surface functional groups on CD-1, which likely include carboxyl and hydroxyl groups that can chelate with lead ions, facilitating their adsorption onto the CD surface [48-50]. The relatively uniform size and distribution of CD-1 nanoparticles, as observed in the SEM and TEM images, also contribute to their high surface area, providing more active sites for lead ion binding. This combination of favourable surface chemistry and high surface area makes CD-1 a highly effective adsorbent for lead ion removal, demonstrating its potential for use in water purification systems. CD-3 also shows a relatively high removal efficiency, with 71.2% of lead ions removed. The slightly lower efficiency compared to CD-1 may be due to differences in the surface functional groups or fewer active sites for lead ion binding. However, the close similarity in removal efficiency suggests that CD-3 still possesses many beneficial properties of CD-1, making it a suitable candidate for environmental remediation applications [22, 51]. The intermediate size and surface characteristics of CD-3, as revealed by the SEM and TEM analyses, likely contribute to its effectiveness in lead ion removal, providing a balance between surface area and functional group availability. In contrast, CD-2 shows a significantly lower removal efficiency, with only 27.5% of lead ions removed. This low efficiency is likely due to the higher degree of disorder and the different surface chemistry observed in CD-2. This may result in fewer or less effective binding sites for lead ions [52, 53]. As indicated by the Raman spectra, the increased disorder could lead to a more heterogeneous surface with fewer carboxyl or hydroxyl groups available for chelation, reducing the adsorption capacity. Additionally, the larger and more variable particle sizes observed in the SEM and TEM images could result in a lower surface area-to-volume ratio, limiting the number of active sites available for lead ion adsorption. The data in **Table 2** underscores the importance of surface chemistry and particle morphology in determining the adsorption efficiency of CDs for heavy metal ions. The differences in removal efficiency among the three samples highlight the impact of precursor composition and synthesis conditions on the functional properties of the CDs. For environmental applications, optimizing the synthesis process to produce CDs with high surface area, uniform particle size, and an abundance of functional groups is crucial for maximizing their adsorption capacity and effectiveness in removing contaminants from water.

Table 2. shows the removal of lead ions.

Sample	**Pb^+ removal**
CD-1	77.2 %
CD-2	27.5%
CD-3	71.2%

4. Conclusion

This research provides a comprehensive analysis of the impact of precursor ratios on the synthesis and properties of carbon dots (CDs) using tartaric and oxalic acids in a hydrothermal method. The study successfully demonstrates the potential of these CDs in environmental remediation, particularly in lead ion removal from aqueous solutions. Among the three synthesized samples, CD-1, with a 1:1 ratio of tartaric to oxalic acid, exhibited the highest lead ion removal efficiency of 77.2%, significantly outperforming CD-2 and CD-3. The superior performance of CD-1 can be attributed to its optimized surface functionalization and structural order, which were achieved through careful control of the precursor ratio. FTIR, TEM, and Raman analyses provided detailed insights into the CDs' surface chemistry, morphology, and graphitization, revealing that the 1:1 precursor ratio led to a well-balanced combination of these properties. The findings underscore the importance of precursor ratio optimization in tailoring CD properties for specific applications. This study contributes to the broader understanding of carbon dot synthesis and its implications for environmental applications. This research offers a rational approach to designing CDs with enhanced performance by establishing clear correlations between synthesis parameters and CD properties. The integration of optical property analysis with environmental remediation performance further highlights the multifaceted potential of CDs in addressing real-world challenges. The novel approach presented in this study provides a valuable framework for optimizing carbon dot synthesis for environmental applications. The successful demonstration of lead ion removal efficiency underscores the practical relevance of this research and its potential to inform the development of more effective and sustainable water treatment technologies. As the field of carbon-based nanomaterials continues to evolve, the insights gained from this study will undoubtedly contribute to the advancement of environmental nanotechnology and the broader application of CDs in various domains.

Conflicts of interest

There are no potential conflicts of interest.

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