



Synthesis of C-dots from Table Sugar and Citric Acid for Heavy Metal Detection

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Received 24 October 2022 | Accepted 29 May 2023 | Published 30 November 2023
DOI: <https://doi.org/10.37859/jp.v14i1.4214>

Keywords:

Carbon dots;
Synthesis;
Detection;
Luminescence

Abstract. Heavy metal contamination is one of the problems in pharmaceutical products. Carbon dots are a practical and economical method of heavy metal detection to reduce heavy metal contamination in pharmaceutical products. Carbon dots have been successfully synthesized by microwave irradiation from sugar table doped with urea fertilizer (CD-GU) and citric acid doped with urea fertilizer (CD-AU). The success of the synthesis of carbon dots can be seen from their properties that produce a blue to green luminescence when excited with a 405 nm laser and an orange to red luminescence when excited with a 532 nm laser. When excited with a 405 nm laser, CD-GU produces a strong light blue luminescence, while CD-AU produces a strong green luminescence. CD-GU and CD-AU also exhibit orange luminescence when excited with a 532 nm laser. The intensity of luminescence of carbon dots will decrease when interacting with heavy metals so that it can be applied for heavy metal detection. The results of this study revealed that CD-GU and CD-AU can be used to detect heavy metals.

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1. Introduction

Environmental pollution caused by heavy metals has emerged as a contemporary issue. Due to its potential to contaminate the environment and its long-term toxicity to the human body, heavy metals pose a significant risk (Zuo et al., 2015). The mitigation of heavy metal pollution to people can be achieved by the implementation of feasible, rapid, and cost-effective heavy metal detection techniques to guarantee the absence of heavy metals in items used by humans. The technique of atomic absorption spectroscopy is widely employed for the detection of heavy metals. Nevertheless, this approach is limited by its challenging sample preparation, including the use of acid as a volatile destruction reagent, which increases the risk of losing the test sample and introducing inaccuracies in the analysis. Furthermore, the operational expenses are extremely substantial and the equipment is priced at a costly level (Rodiana et al., 2013).

Carbon dots provide an alternate approach for metal detection (Zuo et al., 2015). Carbon dots refer to carbon-based nanoparticles with dimensions smaller than 10 nm, composed of carbon networks with sp^2 and sp^3 orbitals (Nasir et al., 2019; Gayen et al., 2019). Carbon dots have luminous characteristics, excellent biocompatibility, high solubility, low toxicity, and favourable solubility stability (Sun & Lei, 2017). Carbon dots possess luminescent features and feature electrons that function as both donors and acceptors, rendering them potentially suitable for use in the sensing sector (Wang & Hu, 2014).

Influence of heavy metal ions on donor or acceptor electrons in carbon dots leads to fluorescence quenching, which is the phenomena of decreased intensity or complete disappearance of fluorescence in carbon dots (Zu et al., 2017). The primary focus of this work is on the luminescent characteristics of carbon dots. Carbon dots typically exhibit luminescence within the visible light wavelength range of 400-800 nm when stimulated by ultraviolet (UV) light (Fong et al., 2018). This study aims to investigate the detection of metals using carbon dots under 254/366 nm UV light.

The promising potential of carbon dots as a viable heavy metal sensor strategy motivates researchers to advance their synthesis techniques. Carbon dots can be synthesized conventionally by both top-down and bottom-up approaches. The top-down approach involves the synthesis of nanoparticles via the degradation of larger particles. The techniques encompassed in this compilation are arc discharge, electrochemical oxidation, ultrasonic synthesis, and laser ablation (Wang et al., 2017). The bottom-up approach employs the synthesis of nanoparticles from individual atoms or molecules. According to Wang et al. (2017), bottom-up techniques encompass microwave synthesis, thermal decomposition, hydrothermal treatment, template approaches, and plasma therapy.

Alternative sources for the synthesis of carbon dots include acidic substances like citric acid, organic compounds, and carbohydrate materials such granulated sugar (Seedad et al., 2021; Putro & Akhiruddin, 2019; Ansi et al., 2018). The study conducted by Ansi et al. (2018) shown that carbon dots derived from granulated sugar are capable of detecting lead metal (Pb^{+2}). The study conducted by Song et al. (2014) revealed that carbon dots derived from acidic substances, such as citric acid, have the capability to detect iron metal (Fe^{+3}). The researcher aims to assess the efficacy of carbon dots in detecting different heavy metals derived from distinct precursor materials, specifically carbon dots derived from carbohydrate materials (granulated sugar) and carbon dots derived from acidic materials (citric acid).

2. The Methods

2.1. Tools and materials

The equipment utilized comprises a Microwave, IRSpirit-QATR-S Spectrophotometer, UV-Vis UV-1900i Spectrophotometer, UV lamp operating at wavelengths of 254/366 nm, magnetic stirrer, hotplate, digital scales, 405 nm blue laser (Class II Laser Product), 532 nm green laser (Class III Laser Product), and other glassware frequently employed in laboratory settings. The materials used include granulated sugar (GKP 1), citric acid (Cap Gajah), urea fertilizer (PUSRI), distilled water, lead (II) nitrate, mercury (II) chloride, aluminum (III) chloride, copper (II) sulfate, potassium dichromate, filter paper no. 42 (Whatman), and a 0.22 μ m pore size circular nylon membrane filter.

2.2. Synthesis of carbon dots

To prepare a stock solution, dissolve 4 gr of granulated sugar and 4 gr of urea fertilizer in 50 ml of distilled water. Stir the mixture using a magnetic stirrer for 15 minutes in a beaker. Once the solution has achieved homogeneity, it is transferred into a measuring flask and combined with distilled water up to a volume of 100 ml, followed by homogenization. A volume of 50 ml of the stock solution is transferred into an Erlenmeyer flask which has not been sealed at its opening. Next, the Erlenmeyer flask is subjected to microwave heating at 250oC for 1 hour and 30 minutes. Upon completion of the microwave heating procedure, a crude carbon dots product is produced manifesting as a brown to blackish crust. Allow it to rest for probably 15 minutes to acclimate to ambient temperature. Solubilize the crude carbon dots product in 100 ml of purified water. To obtain a carbon dots solution, the carbon dots solution is filtered using filter paper no 42 (Whatman). Once the filtration process is finished, expose the carbon dots solution to a laser with wavelengths of 405 nm and 532 nm in order to see the fluorescence emitted by the carbon dots solution. Success in the synthesis of carbon dots is shown by the production of fluorescence. Iterate the identical procedures for the production of carbon dots using urea-doped citric acid in a 1:1 weight/weight ratio (Qu et al., 2012).

2.3. Carbon dot characterization

The effectively produced carbon dots are characterized by analyzing the color of their fluorescence using a 254/366 nm UV light. The maximum absorption spectrum of carbon dots is determined by its characterization using a UV-Vis spectrophotometer within the wavelength range of 200-800 nm. Subsequently, the functional groups in the carbon dots are determined by characterisation using Fourier Transform Infra Red (FT-IR).

2.4. Carbon dots for detecting heavy metals

Prepare solutions of metal ions Pb^{+2} , Cu^{+2} , Hg^{+2} , Al^{+3} , and Cr^{+6} , each at a concentration of 0.1 M. The metal detection test was conducted using a paper-based UV lamp with a wavelength of 366 nm. The medium employed was a 25 mm nylon membrane filter characterized by a pore size of 0.22 micrometers. Immerse the membrane filter in the solution containing carbon dots for a duration of 5 minutes. Detach the membrane filter and allow to dry naturally. Detect the fluorescence of the membrane filter when exposed to a 366 nm UV light. Repeat the process by immersing the membrane filter in the metal ion solution containing Pb^{+2} , Cu^{+2} , Hg^{+2} , Al^{+3} , and Cr^{+6} for 5 minutes each sheet individually. Once the paper has thoroughly dried, reexamine the fluorescence using a 366 nm UV lamp to determine if there is a decrease in fluorescence intensity. Carbon dots are capable of detecting heavy metal ions when the fluorescence intensity of the carbon dots decreases or disappears (Gupta et al., 2016).

3. Result and Discussion

Successful synthesis of carbon dots from urea-doped granulated sugar (CD-GU) and urea-doped citric acid (CD-AU) has been achieved using the bottom-up approach utilizing microwave irradiation. The process of carbon dot formation, as described by Zhu et al. (2013) in Yang et al. (2013) and Ansi et al. (2018), starts with the assembly of precursors through hydrogen bonds. These precursors then undergo dehydration and polymerization through the application of energy from microwave irradiation or other sources of energy, such as high temperatures and hydrothermal pressure. Polymerization induces aromatization, resulting in the generation of aromatic groups (C=C) that constitute the primary constituents of the carbon dots core. The subsequent phase is referred to as carbonization and passivation, whereby the carbon core generated will undergo explosion into carbon dots nanoparticles with surface functional groups when its concentration surpasses the supercritical threshold. The carbonization stage induces a transition in color from colorless to blackish brown in the solution. Therefore, it may be inferred that the sample solution, which initially appears brownish to blackish after undergoing the synthesis process, has rather successfully formed carbon dots.

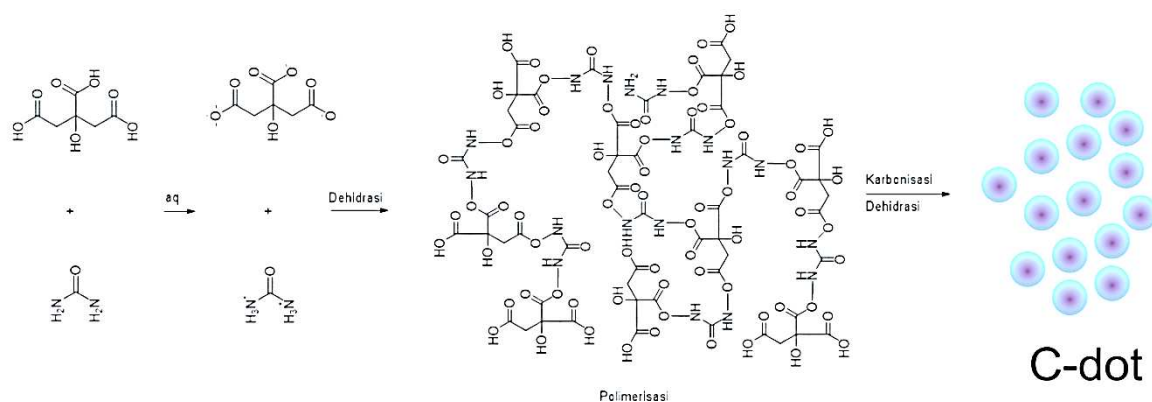


Figure 1. Synthesis pathway for C-dots (Yang et al., 2013).

The analysis revealed that CD-GU exhibited a brownish yellow solution, whereas CD-AU displayed a blackish brown solution (Figure 2). The successful formation of carbon dots by CD-GU and CD-AU can be determined based on the color of the solution. Nevertheless, it is imperative to verify

the actual formation of carbon dots by detecting their hallmark features, specifically the generation of luminescence upon excitation with UV radiation (Fong et al., 2018). In addition, emission measurements from carbon dots were conducted by stimulating them with a 405 nm laser and a 532 nm laser. Based on a study conducted by Pan et al. (2015), it has been observed that carbon dots exhibit a notable change in wavelength as the excitation wavelength increases within the range of 400-570 nm. Consequently, when excited with a 405 nm laser, carbon dots generally emit green luminescence (blue), while when excited with a 532 nm laser, they produce orange lighting (green).

The test findings indicated that CD-GU exhibited intense bright blue luminescence upon excitation with a 405 nm laser, orange luminescence upon excitation with a 532 nm laser, and robust blue luminescence upon excitation with UV lamps of 254 nm and 366 nm. The findings presented here reflect the study conducted by Liu et al. (2017) on the synthesis of carbon dots using urea-doped sugar. Their study revealed that the emission of carbon dots reached its maximum wavelength in the blue area when stimulated by a light source with a wavelength ranging from 340 to 400 nm. The CD-AU material exhibited intense green luminescence when stimulated by a 405 nm laser and 254 nm UV lamp, orange luminescence when stimulated by a 532 nm laser, and intense light blue luminescence when stimulated by a 366 nm UV lamp. In their study on the synthesis of carbon dots from citric acid doped with nitrogen groups, Seedad et al. (2021) observed that the green luminescence color of the carbon dots matched the photoluminescence (PL) spectrum. The spectrum exhibited a peak at 540 nm in the green region when excited by a light source with a wavelength of 408.5 nm, and a peak at 438 nm when excited by a light source with a wavelength of 370 nm. From the acquired results and corroborated data from other researchers, it can be concluded that CD-GU and CD-AU have effectively produced carbon dots.

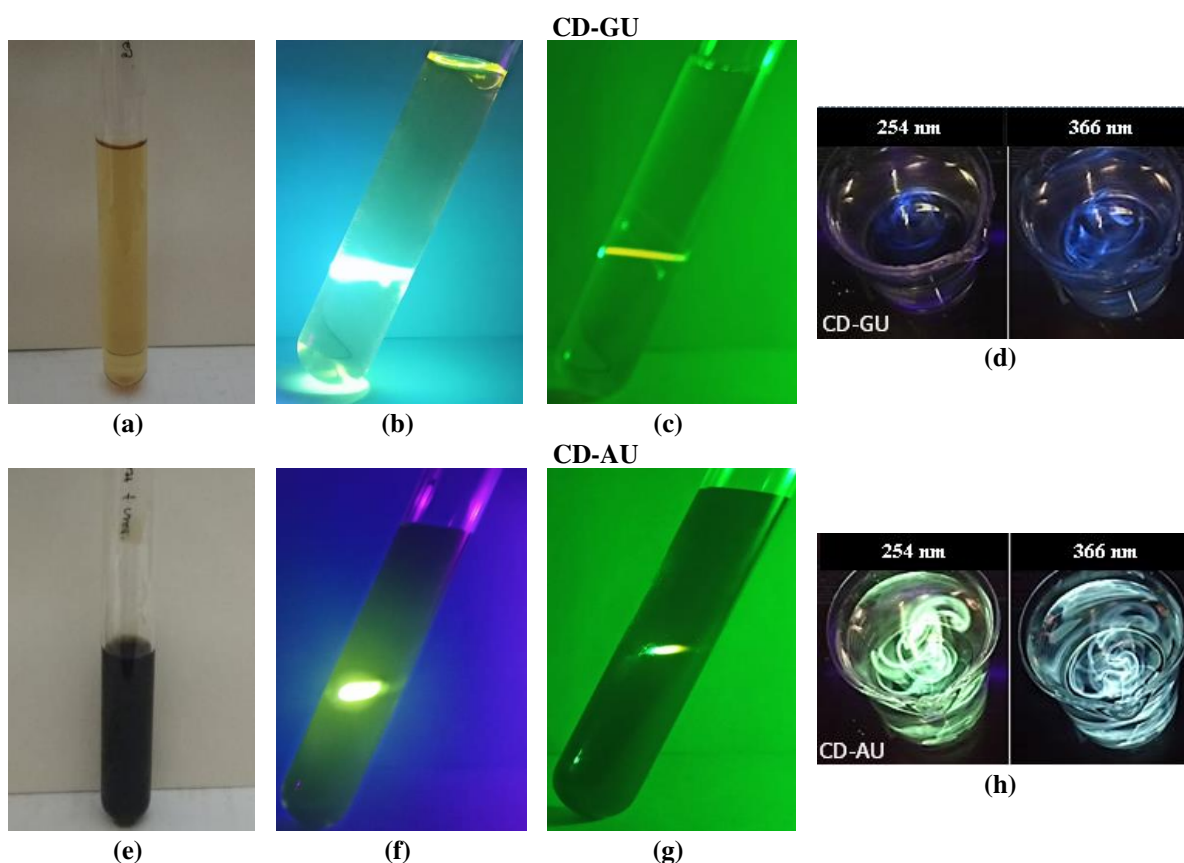


Figure 2. Observational results of luminescence color on carbon dots. a) CD-GU illuminated by room light. (b) CD-GU stimulated by a laser operated at 405 nm. (c) CD-GU stimulated by a 532 nm laser. (d) CD-GU stimulated by a UV light radiating at 254/366 nm. e) CD-AU illuminated by ambient light. (f) CD-AU stimulated by a 405 nm laser. (g) CD-AU stimulated by a 532 nm laser. (h) CD-AU stimulated by a UV light radiating at 254/366 nm.

Further characterization of CD-GU and CD-AU was conducted using FT-IR to identify the functional groups present in their structures. In Figure 3a, the FT-IR test of CD-GU revealed a wide absorption band at 3197.84 cm^{-1} , which was identified as O-H stretching (Sari, 2019). The absorption bands observed at 1653.96 cm^{-1} and 1589.63 cm^{-1} correspond to the stretching vibrations of C=O and C=C bonds, respectively (Sari, 2019; Dachriyanus, 2004). Furthermore, the CD-GU structure exhibits the presence of a COO- group, as evidenced by the absorption bands seen at 1413.80 cm^{-1} , 1362.33 cm^{-1} , and 1316.59 cm^{-1} (Ansi & Renuka, 2020; Sari, 2019). Two absorption bands at 1044.98 cm^{-1} and 1013.53 cm^{-1} correspond to the stretching vibrations of C-O-H bonds (Ansi & Renuka, 2020). The FT-IR spectra of CD-GU does not exhibit any discernible vibration of the N-H bond. This phenomenon may arise due to the obstruction of the absorption band associated with the N-H bond vibration by the wide absorption band of the O-H bond.

As shown in Figure 3b, the FT-IR test of CD-AU reveals a wide absorption band at 3160.67 cm^{-1} , which is attributed to O-H / N-H stretching (Eliana et al., 2016). These absorption bands at 3042.02 cm^{-1} and 3036.30 cm^{-1} correspond to the stretching vibrations of aromatic C-H bonds (Seedad et al., 2021). The optical spectra at 2819.02 cm^{-1} and 2781.85 cm^{-1} are believed to represent the vibrational modes of O-H aldehyde, as reported by Eliana et al. in 2016. The detected absorption band at 1693.98 cm^{-1} is believed to arise from the vibrations of C=O bonds (Seedad et al., 2021). It is probable that the absorption band at 1646.81 cm^{-1} arises from C=C vibrations (Dachriyanus, 2004; Seedad et al., 2021). Based on the studies by Seedad et al. (2021) and Zhu et al. (2013), it is believed that the absorption band at 1571.04 cm^{-1} arises from N-H bending. Furthermore, it is probable that aromatic C-N vibrations may generate absorption bands at measurements of 1429.52 cm^{-1} , 1412.37 cm^{-1} , 1355.19 cm^{-1} , and 1275.13 cm^{-1} (Eliana et al., 2016; Seedad et al., 2021). According to Seedad et al. (2021), the absorption bands observed at 1183.64 cm^{-1} and 1147.91 cm^{-1} are believed to arise from C-N stretching effects. The carbon-oxygen bond vibration is detected in the absorption band at 1053.56 cm^{-1} (Seedad et al., 2021).

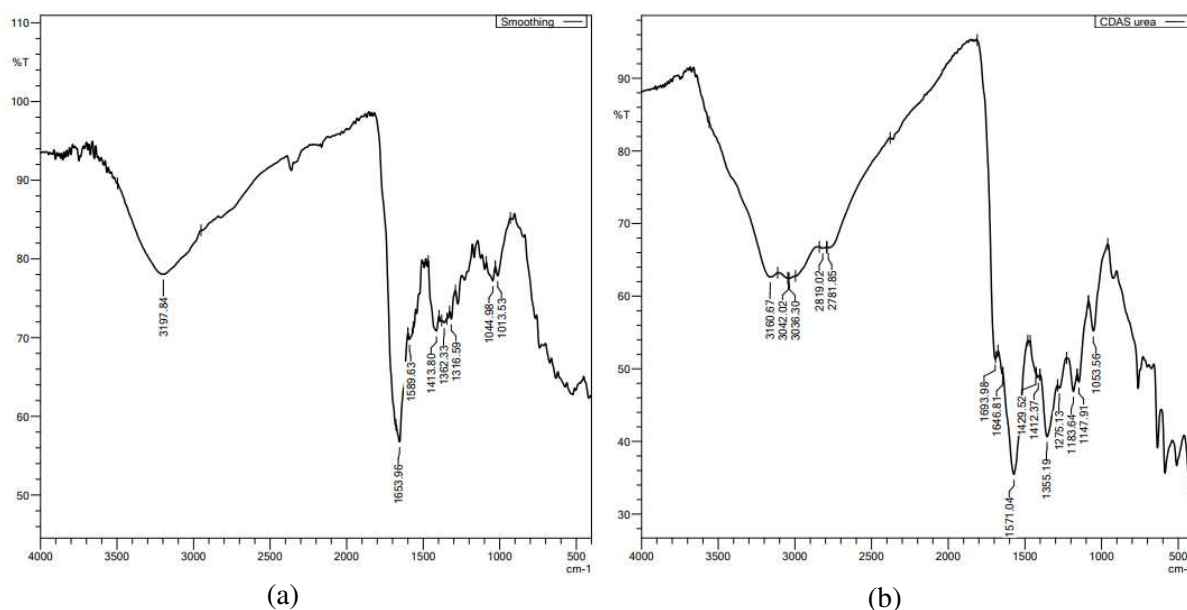


Figure 3. Carbon dot FT-IR spectrum. (a) FT-IR spectrum of CD-GU. (b) CD- AU FT-IR spectrum.

Maximum wavelength measurements on CD-GU revealed absorption at a wavelength of 279 nm. The observed peak can be attributed to the electron transition $n \rightarrow \pi^*$. It is believed that the $n \rightarrow \pi^*$ electron transition arises via the C=O bond on the surface of the carbon dots. Based on the analysis of the infrared (IR) spectrum of CD-GU, which includes the C=O group, and the study conducted by Liu et al. (2017) on carbon dots synthesised from urea-doped sucrose, it was observed that the carbon dots exhibited absorption at a wavelength of 280 nm, which can be attributed to the C=O group. The CD-AU peak wavelength measurements revealed absorption at wavelengths of 406 nm, 272 nm, and 249

nm. The absorption at 406 nm, which signifies the $n \rightarrow \pi^*$ electron transition, is almost certainly caused by the presence of either the C=O group or the C=N group conjugated on the surface of the carbon dots. Consequently, the maximum wavelength of the $n \rightarrow \pi^*$ transition in CD-AU is expected to be higher than that of CD-GU. The conjugation system facilitates the electron transition to π^* by reducing the energy divergence between the ground state and the excited energy level (Suhartati, 2017). Spectral absorption at 272 nm and 249 nm indicates the electron transfer from π to π^* in the center of the carbon dots. It is believed that the electron transition $\pi \rightarrow \pi^*$ at a wavelength of 272 nm arises from the aromatic C-N orbital. It is probable that the electron transition $\pi \rightarrow \pi^*$ at a wavelength of 249 nm arises from the C=C bonding on carbon dots.

Heavy metal detection testing was conducted using CD-GU and CD-AU methodologies. The fundamental concept behind heavy metal detection testing using carbon dots is to monitor the decrease in intensity or suppression of the luminescence generated by carbon dots when exposed to heavy metal ions, using a 366 nm UV lamp (Gupta et al., 2016). Lead (Pb^{+2}), copper (Cu^{+2}), mercury (Hg^{+2}), aluminum (Al^{+3}), and chromium (Cr^{+6}) were among the heavy metals that were examined. The heavy metal detection test utilizing CD-GU demonstrated that CD-GU had the capability to detect all the metals examined, albeit with varying degrees of sensitivity towards each unique metal. Compared to the reduction in luminescence intensity of CD-GU interacting with other metals, Figure 5a demonstrates that the reduction in luminescence intensity of CD-GU interacting with Al^{+3} ions is smaller. Concurrently, the luminescence of CD-GU when it interacts with Cr^{+6} ions is entirely eliminated. Accordingly, it may be inferred that CD-GU exhibits higher efficiency in the detection of chromium metal. Similarly to CD-GU, the heavy metal detection test utilizing CD-AU (Figure 5b) yielded data where the luminescence of CD-AU interacting with Cr^{+6} ions is extinguished. The distinction lies in the fact that the decrease in the luminescence intensity of CD-AU which interacts with Hg^{+2} ions is of lesser magnitude. Should CD-GU exhibit inefficiency in the detection of aluminum metal, it follows that CD-AU would also lack efficiency in the detection of mercury metal.

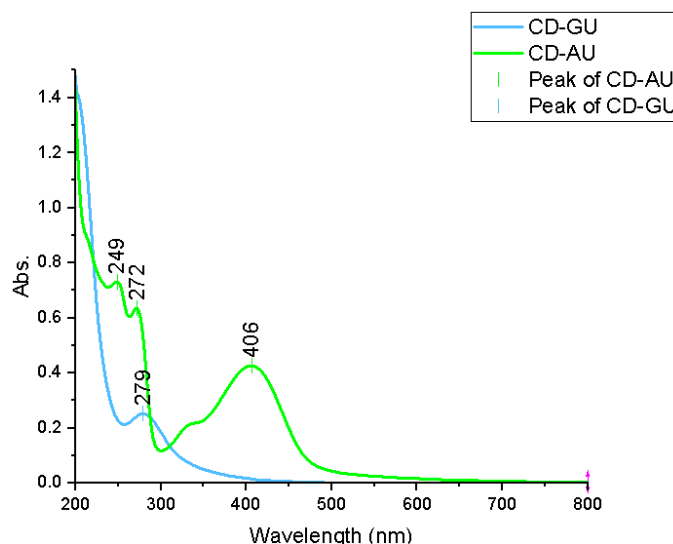


Figure 4. UV-Vis spectrum of carbon dots.

According to Zu et al. (2017), the quenching phenomena can be attributed to five distinct mechanisms. The first mechanism is static quenching, which arises from the formation of a complex in the ground state between carbon dots and metal ions. This complex alters the absorption pattern of carbon dots before and after interaction with metal ions. The second mechanism is dynamic quenching, which occurs when metal ions collide and cause carbon dots in an excited state to return to the ground state, so impacting the fluorescent lifetime. Förster resonance energy transfer (FRET) is the phenomenon where carbon dots and metal ions interact dipole-dipole, resulting in an overlap between the emission spectra of carbon dots and the absorption spectrum of metal ions. This overlap leads to a

decrease in the fluorescence lifespan. Photoinduced electron transfer (PET) is the process of electron transfer between carbon dots and metal ions to create a complex that can revert to its ground state without emitting photons. This interaction reduces the fluorescent lifetime and creates an energy gap between the carbon dots and metal ions. The inner filter effect (IFE) occurs when the absorption spectrum of metal ions coincides with the emission spectrum of carbon dots. As a result, the excitation light of the carbon dots is attenuated by the metal ions, but the absorption spectrum and fluorescent lifetime of the carbon dots remain unchanged before and after interaction with the metal ions.

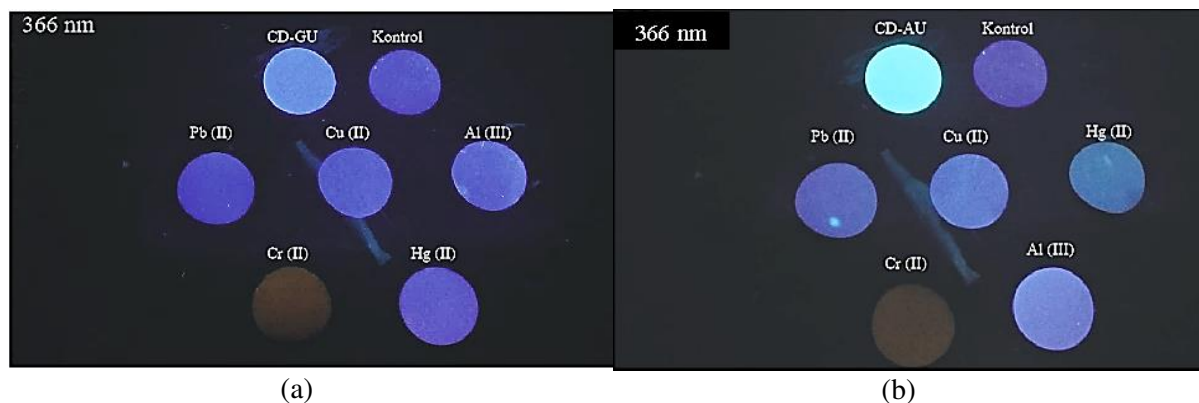


Figure 5. Results of heavy metal detection tests employing carbon dots under 366 nm UV light. (a) CD-GU's metal detection test (b) CD-AU metal detector test.

Conclusion

The synthesis of carbon dots from urea-doped granulated sugar (CD-GU) and urea-doped citric acid (CD-AU) has been achieved by the bottom-up approach utilizing microwave irradiation. The successful synthesis of carbon dots is demonstrated by the existence of intense light blue luminescence generated by CD-GU when stimulated by a 405 nm laser, and intense green luminescence generated by CD-AU when stimulated by a 405 nm laser. Furthermore, the existence of vibrations from the C=C bond, which is the fundamental component of carbon dots, observed in the FT-IR spectrum, provides further evidence for the successful synthesis of CD-GU and CD-AU. The results of heavy metal detection experiments using CD-GU and CD-AU indicate that both CD-GU and CD-AU exhibit higher efficiency in detecting chromium metal. This is evident from the data, which demonstrates a significant decrease in luminescence intensity in CD-GU or CD-AU when bound to Cr^{+6} ions, as compared to interactions with other metal ions.

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