Facile synthesized carbon dots for simple and selective detection of cobalt ions in aqueous media

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CHEMICAL ENGINEERING | RESEARCH ARTICLE

Facile synthesized carbon dots for simple and selective detection of cobalt ions in aqueous media

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Abstract: A simple method was demonstrated for detecting cobalt ions based on the fluorescence quenching of carbon dots (CDots). The best quantum yield for CDots synthesized by microwaving lime and methanolamine is 49.42%. The detection range of this research is 0–200 µM with a detection limit of 1.63 µM, respectively. Because of the interaction between functional groups and metal ions, CDots made from lime are more sensitive to cobalt ions than CDots made from other oranges. This study presents a platform for demonstrating the high sensitivity of cobalt ion sensing using CDots. Overall, these findings suggest that functional groups influence the quantum yield and detection limit of CDots, which will be beneficial for the further development of CDots for sensing purposes.

Subjects: Environment & Health; Chemistry; Physical Chemistry; Materials Science; Applied Physics

Keywords: CDots; functional groups; quantum yield; oranges; quantum yield; detection limit

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Heri Sutanto is a Full Professor in Department of Physics, DiponegoroUniversity. He also leads the Smart Materials Research Center (SMARC), Diponegoro University. His research focuses on the development of nanomaterials, thin film, photocatalyst, radioprotector materials and radiotherapy. He has more than 100 publications in scientific journals. He is also the recipient of numerous awards. Hadiyanto is a Full Professor in Department of Chemical Engineering, Diponegoro University. He also leads the Center of Biomass and Renewable Energy (C-Biore), Diponegoro University. His research focuses on the development of bioprocess, food engineering, microalgae, and green-energy. Ilham Alkian is a doctoral student atthe Department of Environmental Sciences, Diponegoro University. This reported research is part of a roadmap for the development of environmentally friendly advanced materials for heavy metal detection by the SMARC laboratory.

PUBLIC INTEREST STATEMENT

Heavy metal pollution is a major global concern. One of the heavy metals, cobalt, is widely used in various applications such as batteries, catalysts, and paint. Humans may be exposed to it by breathing air, drinking water, or eating cobalt-containing foods because it is distributed in the environment as cobalt ions. The excess presence of cobalt ions in the human body leads to various negative effects. Nowadays, carbon dots have demonstrated unique properties as sensing platforms for heavy metal detection. The detection of cobalt by carbon dots is simple and highly sensitive. In this study, we show the carbon dots with various functional groups synthesized from various oranges by using the microwave method. We also demonstrate how the carbon dots are used as a sensing platform for detecting the presence of cobalt in water. Therefore, this study will be beneficial for simple and highly sensitive detection of the presence of the cobalt in the environment.

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1. Introduction

Considering its favorable physiochemistry, cobalt is widely used in various applications, including batteries, catalysts, and paint (Manohar et al., 2006; Rafighi et al., 2010). In biology, cobalt ion (Co^{2+}) is one of the metal ions essential for the formation of the vitamin B complex in the human body. As Co^{2+} ions are dispersed in the environment, humans may be exposed to it by breathing air, drinking water, and eating food that contains cobalt. A small amount of Co^{2+} ions can increase red blood cell production (Awual et al., 2014). Furthermore, it regulates the catalytic activity of an enzyme or a cofactor, altering the metabolism of several elements (Liao et al., 2018). Nonetheless, an excess of Co^{2+} ion can cause poisoning and a variety of adverse effects on the human body, including vomiting, paralysis, diarrhea, hypotension, dermatitis, and even death due to myocardial infarction or heart attack (Ahmadpour et al., 2009; C.-L. Li et al., 2015; Liao et al., 2018; Wang et al., 2017).

As a result, the Co^{2+} ion should be detected to reduce the adverse effects on human health. Many methods for detecting Co^{2+} ions have been developed, including capillary electrophoresis, spectroscopy, atom absorption, and photocolorimetry (Du et al., 2019). On the other hand, these proposed methods require a complex procedure and a longer time for detection. Nowadays, a method based on fluorescence material carbon dots (CDots) has been developed by many researchers for detecting heavy metals (Y. Huang et al., 2019; W. Tian et al., 2019). This method has advantages such as easiness, sensitivity, and fast for in-situ detection (Devi et al., 2019; Long et al., 2021; Luo et al., 2020). Basically, CDots are carbon particles with a size of less than 10 nm that have been utilized as intelligent materials for various applications (Gogoi & Karak, 2017; H. Li et al., 2019; T. Liu & Li, 2020; Sun et al., 2022; Yan et al., 2020). It shows potential for detecting heavy metals due to their unique properties such as solubility in water, high photostability, low toxicity, high biocompatibility, multicolor emission, and high quantum yield fluorescence (L. Li & Dong, 2018; H. Liu et al., 2018; Sagbas & Sahiner, 2018; P. Zhao & Zhu, 2018).

Nowadays, precursors of CDots based on natural materials have become popular as carbon sources since they are environment-friendly, abundant in quantity, easy to be found, and low-cost (Das et al., 2018; L. S. Li et al., 2018; Thambiraj & Shankaran, 2016). Many studies reported CDots with carbon sources from fruits such as Carica papaya (Kasibabu et al., 2015), tomato (Lai et al., 2020), pear, avocado, kiwi (Dias et al., 2019), and from perennial grass such as sugar cane (G. Huang et al., 2017).

Therefore, herein we study the CDots with carbon sources from orange families such as santang honey (Citrus sinensis), lime (Citrus aurantifolia), lemon (Citrus limon), mandarin (Citrus reticulata), and sweet orange (Citrus sp). We choose oranges since they are easy to be planted in Indonesia and can be found in many countries. Furthermore, monoethanolamine (MEA) is added to the orange extract as a passivating agent. The microwave method was used to synthesize CDots in this study (Edison et al., 2016; H. Yang et al., 2018; He et al., 2018; Zuo et al., 2016). According to our findings reported previously, this method is simple, reproducible, and easy to control temperature and microwave power (Alkian et al., 2019; J. Liu et al., 2017; Sutanto et al., 2020). The optical properties of the CDots are also characterized and analyzed. The CDots are used to determine the range and detection limit by probing the fluorescence of Co^{2+} at various concentrations.

2. Experimental details

Synthesis of CDots. In this study, various oranges are used as carbon sources. Monoethanolamine (Merck GmbH) is used as passivating agent. Cobalt (II) nitrate hexahydrate (Merch GmbH) is used as a precursor for cobalt ions. To synthesize CDots, 30 mL of extract oranges and 5 mL of MEA were first dissolved in DI water and stirred at 500 rpm for 5 min at room temperature. Then, the solution was put under an ultrasonic wave for 30 min, followed by a microwave for 60 min (Sutanto et al., 2020). The CDots solution was centrifuged at 1500 rpm for 5 min, followed by filtration using filter paper. The absorbance of CDots was measured by using a UV-vis spectrophotometer (Pharo 300 Spectroquant). The functional groups in CDots were analyzed using a Fourier Transform Infra-Red Spectrophotometer

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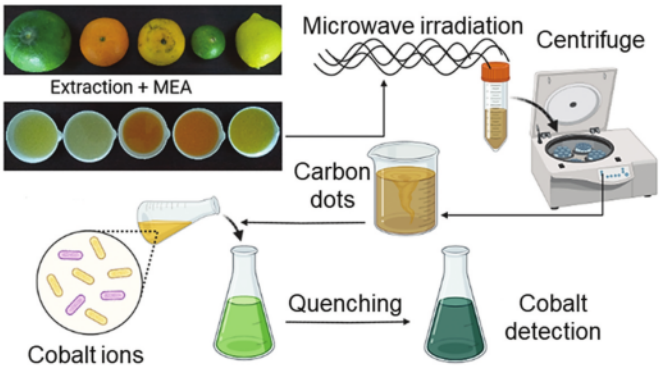

Figure 1. Synthesis schematic of CDots and cobalt ion detection.

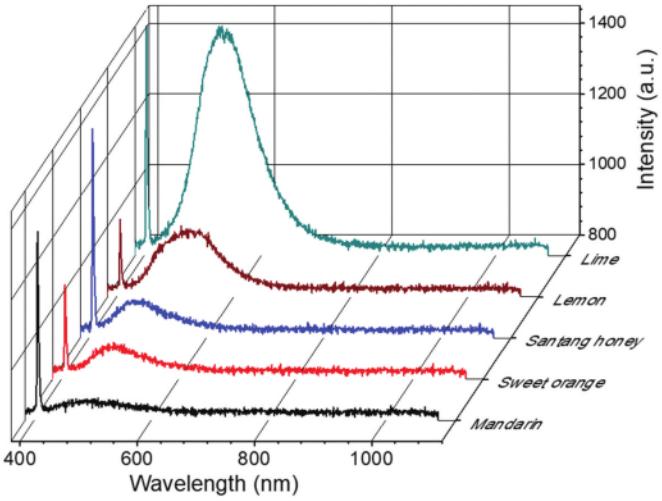

Figure 2. Photoluminescence (PL) spectra of CDots synthesized from various oranges.

(Perkin Elmer Spectrum 2). The fluorescence intensity of CDots was analyzed using Optical Multi Analyser Spectrograph (SA100W-HPCB1024/C) with a diode laser of 405 nm as the excitation source. The morphology and the size of CDots were characterized by transmission electron microscopy (TEM, HT7700, 120 kV). The schematic for the synthesis of CDots and the cobalt ion detection is shown in Figure 1.

Quantum yield calculation. QY of quinine sulfate is determined as a reference of fluorescence (Issa et al., 2020; W. Liu et al., 2017). Quinine sulfate was dissolved in 0.1 M H_2SO_4 (QY = 0.54, η = 1.33), and CDots were dissolved in DI water. QY was calculated by using the equation of $QYc = QYs \times (Ic/Is) \times (As/Ac) \times (\eta c/\eta s)^2$, where QY is quantum yield, I is integral of fluorescence, A is the intensity of absorbance with an excitation wavelength of 405 nm, η represents index bias of solvent, and the subscript of "c" and "s" represent CDots and quinine sulfate, respectively.

Detection of cobalt ion. Heavy metal detection was done by dissolving cobalt ions with a concentration of 0–200 μM in CDots solution that possesses the best QY. The detection of cobalt was performed by observing the change in fluorescence intensity of the CDots solution. The fluorescence spectrum was characterized by using an OMA spectrograph at room temperature

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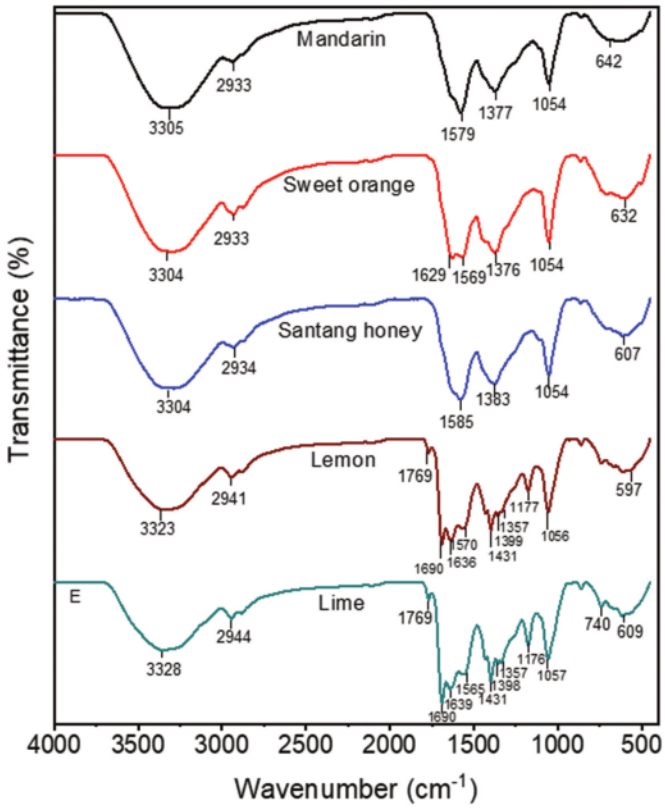

Figure 3. Fourier transform infrared spectroscopy (FTIR) spectra of CDots synthesized from various oranges.

with an excitation wavelength of 405 nm. The detection limit of cobalt ions is determined according to the following equation

$$LoD = \frac{3\sigma}{K_{sv}}$$

where LoD is the detection limit, α is the standard deviation, and K_{sv} is the Stern-Volmer quenching constant.

3. Result and Discussion

Figure 2 depicts the PL spectra of CDots synthesized from various oranges. At the emission wavelength of 490 nm, the fluorescence intensity of mandarin, sweet orange, and santang honey is relatively low, whereas lemon has a slightly higher intensity at 510 nm. On the other hand, Lime has the highest fluorescence intensity of 1450 cps at 532 nm. As a result, the emission wavelength of lemon and lime is practically higher than those of the others. This bright green fluorescence color is consistent with previous studies reporting the optical characteristics of citrus fruit-based CDots (Gudimella et al., 2021; Hoan et al., 2019). The emission range is proportional to the integral area of the fluorescence spectrum that affects the QY. The difference in intensity and emission range of these CDots is expected to be attributed to differences in their molecule density and chemical properties, e.g., functional groups (S. Miao et al., 2020; X. Miao et al., 2018).

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The FTIR spectra of CDots synthesized from various oranges are presented in Figure 3. The C = C vibration is responsible for the strong band at 1579 and 1629 cm^{-1}. The presence of a C = C peak indicates that the Cdots have a graphitic structure. The wavenumber of 3305 cm^{-1} is attributed to O-H bonding which represents the hydroxyl group on the surface of CDots. Its hydroxyl group can improve the hydrophilicity of dots (Y. Liu et al., 2017; Tu et al., 2019; F. Yuan et al., 2016; C. Zhao et al., 2019). Additionally, the absorbance values at 2933 and 1377 cm^{-1} correspond to C-H bending and C-H stretching, respectively (Ensafi et al., 2017; Gong et al., 2017; Sinha et al., 2020). The C = O bonding can be found at 1690 cm^{-1}. This finding indicates that the CDots contain a lot of oxygen on its surface and are very soluble in water. Thus, the CDots can be easily applied as detectors for analytes, whether in the laboratory or actual wet environment. In addition, the C = N and C = C bonding can be found at 642 and 1629 cm^{-1}, respectively (M. Tian et al., 2019).

We also used TEM characterization to examine the size of the CDots synthesized from lime. Figure 4a,**b** shows the TEM image of the CDots and their size distribution. The size of the CDots synthesized from lime ranged from 2 to 20 nm, with an average size of 8.82 nm. This result demonstrates that nanometer-sized CDots can be successfully synthesized from lime using a simple method.

The absorbance spectrum of CDots was analyzed using UV-Vis to elucidate the optical properties of CDots (Figure 5a). In the UV region, a peak was found at 230 nm, which represents π − π* transition of C = C and C = N bonding in the aromatic ring. This absorption usually does not have fluorescence. In the visible region, the absorption peaks at 300, 308, 310, 361, and 346 nm are associated with n-π* transition related to the C-N/C = N and C-O/C = O on the surface of the CDots. The peak shifting of CDots synthesized from lemon and lime might be associated with the higher number of C = O, C = N, C = C/C-H, and C-N functional groups.

The emission of CDots is mainly caused by conjugated carbon and bonded oxygen. The functional groups on the surface of CDots have different energy levels, resulting in an emission trap. The COC and COH groups on sp2-hybridized carbons can cause significant local distortion, resulting in various energy gaps. Such new energy gaps could be found in the π − π*gap's band tail. The n-π* transitions become dominant due to the formation of C = O in CDs. As the number of COH and COC groups in CDs increases, a large number of epoxide (or hydroxy)-related localized electronic states are expected to form below the π* state, resulting in the formation of numerous new energy levels between the n-π* gaps (schematized in Figure 5b). As a result, many different types of radiative recombination could occur, resulting in a wide range of excitation energies. As a result, the O-containing groups in CDs may play an important role in the formation of energy gaps and quantum yield. According to our result, the CDots synthesized from lemon and lime have a higher number of O-containing groups than other CDots, leading to higher quantum yield.

Furthermore, N-containing groups could act as surface passivator, allowing for more effective radiative recombination of surface-confined electrons and holes, although some believed that CN bonds could alter electronic structures. The nitrogen on the CDots surface will give additional energy levels as emission traps. These energy levels have narrower HOMO (highest occupied molecular orbital) and LUMO (lowest unoccupied molecular orbital). Once the CDots are excited with the same energy, the excited electron number, and the emission intensity will be higher. However, the energy required for non-radiative relaxation will be higher, resulting in a longer emission wavelength than the excitation wavelength (Yan et al., 2019). The PL intensity will be higher because CDots synthesized from lemon and lime have more N-containing groups than those synthesized from other oranges, especially the appearance of the functional groups C = N (ν = 1690 cm^{-1}) and C-N (ν = 1176 cm^{-1}). This phenomenon confirms that nitrogen from MEA as a passivator can only coat the surface of CDots made from lime and lemon, not found in other oranges. The abundance of C = N and C-N functional groups proves that CDots made from lime has more surface defects and emission traps to produce high PL. This finding complements the previous research phenomenon where the -COOH functional group affects PL production

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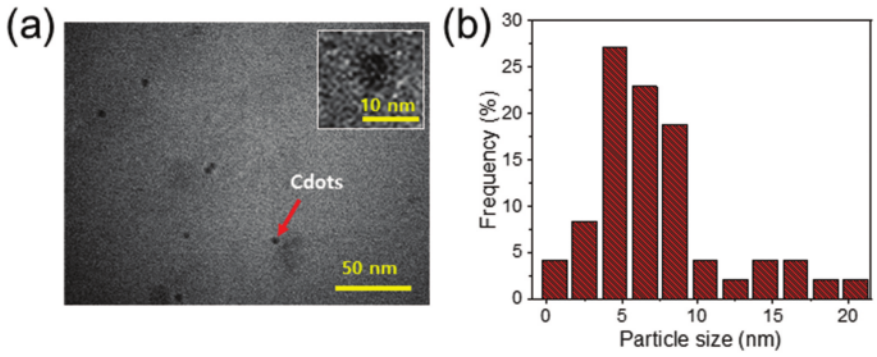

Figure 4. (a) The transmission electron microscopy (TEM) image of CDots synthesized from lime; (b) The size distribution of TEM image for CDots synthesized from lime.

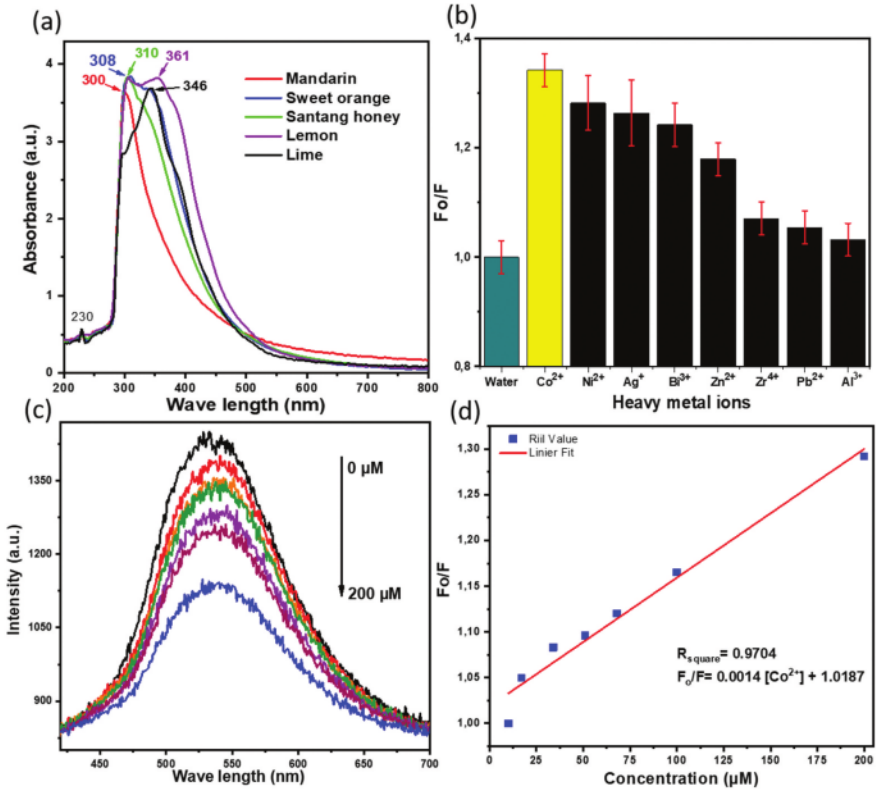

Figure 5. (a) Absorbance spectra of CDots from various oranges; (b) Selectivity of CDots for heavy metal ions; (c) Fluorescent quenching by cobalt ion; (d) The linear relationship between F/F_o and concentration of cobalt ion.

(X. Miao et al., 2018). In other words, we found that the number and type of functional groups in CDot affected the fluorescence intensity and absorbance used in calculating the quantum yield.

QY is the most critical indicator for indicating the successful synthesis of CDots. It represents the ratio of the number of emitted and absorbed photons. The QY obtained for CDots synthesized from mandarin, sweet orange, santang honey, lemon, and lime are 1.62, 2.96, 10.90, 28.07, and 49.42%, respectively. These results reveal that CDots from lime has the highest QY compared to other samples,

almost equal to the QY of quinine sulfate (54%) as reference material fluorescence. The CDots lime in this study resulted in a relatively high QY that could compete with various other CDots synthesized using the microwave method and the same standard fluorescence material (Dias et al., 2019; Lai et al., 2020; Z. Yang et al., 2013). The photographic images of these CDots are shown in Figure 6 and 7. Thus, the CDots from lime is chosen as the material for detecting heavy metal cobalt (Co^{2+}) in this study. For this experiment, the various concentration of Co^{2+} is prepared by dissolving them in DI water.

The sensitivity indicates the capability of CDots for detecting heavy metals. Various heavy metals Zn^{2+}, Zr^{4+}, Al^{3+}, Ag^{+}, Pb^{2+}, Ni^{2+}, Bi^{3+}, and Co^{2+} with a concentration of 200 μM have been prepared to test the selectivity of CDots from lime. Figure 5b shows that the addition of water does not affect the fluorescence. The addition of Al^{3+}, Pb^{2+}, and Zr^{4+} slightly changes fluorescence intensity. Meanwhile, the addition of Co^{2+} significantly affects the intensity of fluorescence (F_i/F_0) which is more significant than other heavy metals. It implies that the CDots have high selectivity on cobalt ions. As shown in Figure 5c, the PL emission of CDots at around 532 nm decreases as the concentration of Co^{2+} increases from 0 to 200 μM. The result demonstrates that the CDots from lime are highly sensitive to the presence of Co^{2+}. Furthermore, the F/F_0 linear regression is linearly correlated with the concentration of Co^{2+} as presented in Figure 5c. This correlation is defined by $F/F_0 = 0.0014\ [C] + 1.0187$ with R^2 of 0.9704. The slope of 0.0014 μM^{-1} is the constant of quenching Stern-Volmer. The standard deviation, σ is $7.7x10^{-4}$, and the detection limit of Co^{2+} is 1.63 μM. The detection limit of CDots is in line with the studies reported previously, as summarized in Table 1. This result implies that CDots with natural sources can compete with other CDots based on synthetic materials for detecting cobalt. Besides, the CDots from lime shows a high capability for detecting Co^{2+} even at extremely low concentration, which is lower than the threshold set by WHO of 1.7 mM (Bano et al., 2019; Kim et al., 2006).

The detection of cobalt ions by CDots lime only uses an optical turn-off mechanism, which is much simpler when compared to other nanomaterial mechanisms that require I–V gates or other additional devices for heavy metal detection (Han et al., 2021). The absorbance spectrum of Co^{2+} and the fluorescence spectrum of CDots are shown in Figure 6a. The absorbance spectrum of Co^{2+} has a peak at 521 nm, while the fluorescence spectrum of CDots shows a peak at 532 nm. Both spectra are caused by UV-light emission used as the excitation source for luminescence (405 nm). An extinction of fluorescence of CDots by Co^{2+} might be associated with the presence of Co^{2+} on the surface of CDots, forming a complex formation for non-fluorescence. It happens since both UV absorbance spectra and emission spectra of CDots overlap. Therefore, a strategy that can be proposed to improve the detection ability of cobalt in the lowest possible concentration is to modify the fluorescence spectrum of CDots to match the absorbance spectrum of cobalt at a wavelength of 521 nm. If there is a similarity of peaks between the fluorescence of CDots and the absorbance of heavy metals, it will widen the overlap area, which impacts the selectivity and sensitivity.

These results reveal that the mechanism of fluorescence quenching of CDots synthesized from lime by Co^{2+} is an electron transfer (ET) mechanism. Non-emissive electron transfer occurs between the CDots and the metal ion quencher in the ET mechanism. In this case, both CDots and the metal ion quencher act as electron donors or acceptors, resulting in an anion or cation radical at the end. To distinguish the donor and acceptor in the scheme, ET can be labeled as reductive ET (CDots-electron acceptor) or oxidative ET (CDots-electron donor). Fluorescence quenching can occur in ET when an electron from the donor is photoexcited and then transferred to the acceptor, resulting in fewer electrons returning to the ground state. The energy gap between the CDots and the metal ion quencher was the driving force for such electron transition. The energy gap in reductive ET is between the LUMO (quencher) and the VB (CDots), while in oxidative ET, it is between the LUMO (CDots) and the CB (quencher).

ET sensing of metal ions is simple because the surface of CDots is generally rich in many chemical groups, i.e., electron-donating groups; on the other hand, the surface of quenchers is usually rich in electron-withdrawing or accepting groups. In this study, CDots can be quenched by Co^{2+} ions using the ET quenching mechanism. The ET mechanism of the lime-based CDots is

Table 1. Our result and reported studies of Cobalt (II) ions detection using carbon dots

Carbon source	Nitrogen source	Method	Emission	QY	Range	Detection limit	Ref
p-phenylenediamine	Asparagine	Hydrothermal	Green	15.5%	0.3–65 μM	22 nM	(M. Tian et al., 2021)
glycine	polyethyleneimine	Hydrothermal	Blue	57%	0.5–3 μM	0.12 μM.	(Bano et al., 2019)
Carbopol 934	diethylenetriamine	Hydrothermal	Blue	38.7%	0–40 μM	0.45 μM	(Kong et al., 2016)
pyridoxal 5-phosphate	Ethanediamine	Hydrothermal	Blue	15.4%	0–60 μM	0.053 μM	(Liao et al., 2018)
L-cysteine	x	Hydrothermal	Blue	13.2%	10 nM –100 μM	5 nM	(C.-L. Li et al., 2015)
Citric acid	l-cysteine	Hydrothermal	Blue	x	0.08–100 μM	80 nM	(Chen et al., 2017)
polyetherimide	Lysine	Microwave	Blue	x	10 nM –5 μM	2 nM	(Wang et al., 2017)
kelp	Ethylenediamine	Microwave	Blue	23.5%	1–200 μM	0.39 μM	(C. Zhao et al., 2019)
Lime	mono ethanolamine	Microwave	Green	49.4%	0–200 μM	1.63 μM	*This work

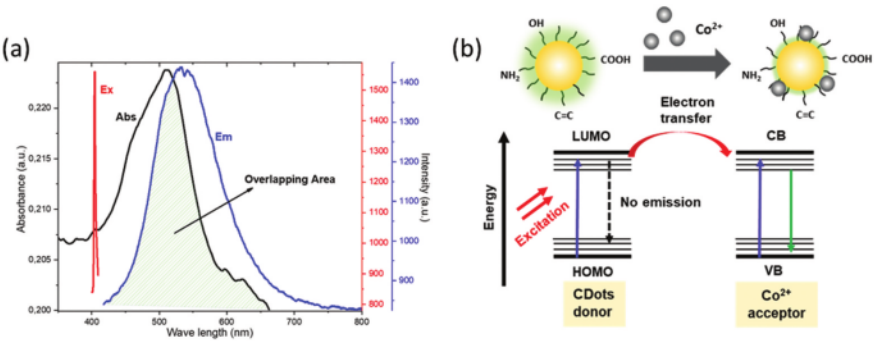

Figure 6. (a) Overlap scheme of CDots spectrum and (b) Schematic of cobalt ion detection via electron transfer (ET) mechanism.

Figure 7. (a) The photograph image of CDots solution synthesized from various oranges without UV irradiation (top) and under UV irradiation (bottom); (a,f) Mandarin, (b,g) sweet Orange, (c,h) santang honey, (d,i) lemon, and (e,j) lime.

depicted in Figure 6b. We expect that chemical groups on CDots (for example, N) acting as an electron donor would prevent tuning the local electronic density of the prepared CDots, promoting coordination between Co^{2+} ions and chemical groups on the CDots surface. It facilitates non-radiative e/h+ recombination, which results in electron transfer from the excited states of CDots to the half-filled d-orbitals of Co^{2+} ions. This leads to significant fluorescence quenching via the ET mechanism, which is also observed in the aqueous solution of CDots containing Co^{2+} ions.

4. Conclusion

We successfully demonstrated the synthesis of CDots from various oranges such as santang honey (Citrus sinensis), lime (Citrus aurantifolia), lemon (Citrus limon), mandarin (Citrus reticulata), and sweet orange (Citrus sp). The CDots are synthesized by using the microwave method. The QY obtained for CDots synthesized from mandarin, sweet orange, santang honey, lemon, and lime are 1.62, 2.96, 10.90, 28.07, and 49.42%, respectively. We found that the emission wavelength and absorbance are altered by the number and types of functional groups in CDots. Furthermore, the CDots from lime can detect cobalt ions even at very low concentrations.

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

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