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Research Article

Production of Carbon Quantum Dots Based on Oil Palm Fronds for Polyethylene and Polyethylene Terephthalate Microplastics Detection

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Abstract: Abundant plastic waste degradation to microplastics may cause pollution hazards for the environment. The circulation of microplastic pollution supports the initiation of research related to detecting microplastics in the environment. One method for detecting microplastics is the utilization of the fluorescence properties of carbon quantum dots (CQDs). In this study, CQDs will be produced through the hydrothermal method using oil palm fronds as a carbon source because of their lignin levels of up to 26%. To obtain this carbon, oil palm fronds are crushed and then converted into biochar through a pyrolysis process. Biochar is used as a precursor for the manufacture of CQDs produced through the hydrothermal method at temperature variations of 180°C, 190°C, and 200°C. In this study, CQDs were produced with a peak of 291 nm in the UV wavelength range, which indicates the presence of a π-π^* absorption band in the carbon structure of CQDs. The emergence of dominant C=C, O-H and C=O groups in the FTIR test also proves the success of CQDs production through this hydrothermal method. The CQDs produced at the three temperature variations are less than 10 nm in size and have the highest fluorescence intensity at 200°C when excited at 405 nm. Moreover, CQDs exhibit a promising ability for detecting microplastics, as indicated by their decreasing fluorescence intensity trend in response to polyethylene (PE) and polyethylene terephthalate (PET) added solution.

Keywords: Carbon quantum dots; Fluorescence; Microplastics; Oil palm fronds; Quenching

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1. Introduction

Plastic is a product derived from polymeric materials. Plastics are composed of chains of monomers and are lightweight, making them easy to be utilized for various products. According to data from the Ministry of Industry (2019), plastic production in Indonesia reached 5.635 million tons or 87% of the total installed capacity of 6.48 million tons annually. The use of plastics in Indonesia is largely dominated by PE (polyethylene) at 34%, followed by PP (polypropylene) at 31%, and PET (polyethylene terephthalate) at 12% (Ministry of Industry, 2019). These three types of plastics are most commonly found in everyday life as they are used for food packaging, drinking bottles, pipes, and automotive and electronic products.

Plastics in food packaging and other products can degrade into smaller waste particles, usually called microplastics (MP) (Garrido Gamarro and Costanzo, 2022; Uurasjärvi et al., 2020). Microplastics commonly found in the environment originate from the degradation of larger plastics such as pellets, exfoliating agents in cosmetics, and opacifiers. This degradation can occur due to abrasion, UV irradiation, hydrolysis, and biodegradation processes (Sutkar et al., 2023; Mitrano and Wohlleben, 2020). Microplastics pose a more severe threat to ecosystems compared to larger plastics. Due to their small size, microplastics can be ingested by aquatic organisms, thereby introducing microplastics into the food chain and digestive systems, which can cause diseases such as stomach ulcers and digestive tract obstructions (Li et al., 2023; Hu et al., 2022). Moreover, organic microcontaminants and heavy metals can accumulate on the surface of microplastics, leading to further negative impacts on humans and other living organisms (Liu et al., 2021; Alimi et al., 2018).

Various researchers have proposed different methods for detecting microplastic dispersions, such as Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and other instruments. However, these existing methods are economically inefficient, time-consuming, and limited to specific types of plastics. As a result, researchers are exploring alternative microplastic detection methods, one of which is the utilization of carbon quantum dots (CQDs). CQDs are a type of quantum dot that has gained increasing attention due to their excellent photoluminescence (PL) properties, ease of production, cost-effectiveness, low-cost raw materials, water solubility, low toxicity, chemical stability, and high functionality, making them a subject of significant interest in recent years (Saputra et al., 2024; Molaei, 2020). Previous studies have demonstrated the use of carbon quantum dots as metal detectors, fluorescent probes, for degradation, wastewater treatment, and other applications (Kumar et al., 2024; Marpongahtun et al., 2023; Rani et al., 2020).

Carbon quantum dots produced from biomass have attracted attention in the research community due to the abundant availability of raw materials, particularly through the utilization of biomass waste (Dong et al., 2024; Abu et al., 2023). This approach of utilizing biomass waste offers a highly cost-effective alternative by leveraging readily available, low-cost waste materials, significantly reducing production expenses while adding value to otherwise discarded resources (Dua et al., 2023). Carbon sources from biomass waste used so far include oil palm empty fruit bunches, seashells, Ginkgo biloba leaves, and orange peels (Abu et al., 2023; Wang et al., 2020). One type of biomass that has not been widely utilized as a raw material for CQDs is oil palm fronds. The lignin content of palm fronds, which reaches 18.53%, indicates that this biomass waste has the potential to serve as a carbon source for CQD production. Compared to oil palm empty fruit bunches (OPEFB), which contain around 16-19% lignin (Said et al., 2021), palm fronds offer an advantage due to their relatively minimal utilization compared to OPEFB. Therefore, oil palm fronds can be readily available for CQDs synthesis without competing with other industrial utilizations such as the production of biofuels, lactic acid and other sustainable biomaterials (Nyakuma et al., 2023; Hidayah and Wusko, 2020; Hermansyah et al., 2019).

The production methods for CQDs vary widely, but they can be generally classified into two main categories: top-down and bottom-up approaches. To harness the carbon source from lignin, a top-down approach known as the hydrothermal process is employed to produce CQDs. Carbon from oil palm fronds was obtained by the pyrolysis method to maximize conversion, as observed in past studies (Marpongahtun et al., 2023; Kusrini et al., 2018). In this method, temperature and heating duration in the oven are critical factors in successfully converting palm frond biochar into carbon quantum dots. Thus, this study aims to investigate the potential of CQDs derived from oil palm fronds, focusing on their application in microplastic detection due to their outstanding photoluminescence properties. It will examine how variations in hydrothermal method temperatures influence the particle size, absorbance, functional groups, and fluorescence intensity of CQD solutions. Moreover, CQDs fluorescence intensity will be analyzed, both before and after interaction with polystyrene and polypropylene microplastics.

2. Methods

Oil palm fronds (OPF) are the carbon source for CQDs production and are obtained from PT Perkebunan Nasional VIII (Bogor, Indonesia). Deionized water and 98% ethanol from Merck (Darmstadt, Germany) were also prepared as solvents for producing CQDs with the hydrothermal method.

2.1. *Biochar Production*

No initial characterization has been conducted on OPF. OPF was let dry for a couple of weeks before milling to ensure a low water content, crushed into smaller pieces with a milling machine, and sieved to 60 mesh with a sieve shaker. Crushed OPF was then measured to 220 g and converted into biochar by pyrolysis method, producing the biochar and two other side products (bio-oil and synthetic gas). The steps for performing the pyrolysis process are based on studies by Bindar et al. (2022), Rahayu et al. (2021), and Handoko et al. (2021) with some modifications.

2.2. *Production of Carbon Quantum Dots (CQDs)*

The biochar from OPF was dissolved using a mixture of ethanol and deionized water with a 75:25 ratio. The resulting solution was subjected to sonication for 20 minutes at a frequency of 20 kHz and then transferred into the hydrothermal autoclave. The hydrothermal process was conducted for 4 hours at varying temperatures of 180°C, 190°C, and 200°C under a pressure of 1 MPa, though it can also be carried out under self-generated pressure (Tekin et al., 2014). After heating, the solution was allowed to cool at room temperature for 24 hours. CQDs were centrifuged for 15 minutes at 6000 rpm and purified through a 0.2 μm membrane filter. The purified CQDs were then put into a dialysis bag for a minimum of 6 hours to separate ethanol from the solution. The production of CQDs is based on previous studies by Kariminia et al. (2024) and Jamaludin et al. (2020). Figure 1 shows the schematic illustration of the preparation process of CQDs from OPF biochar.

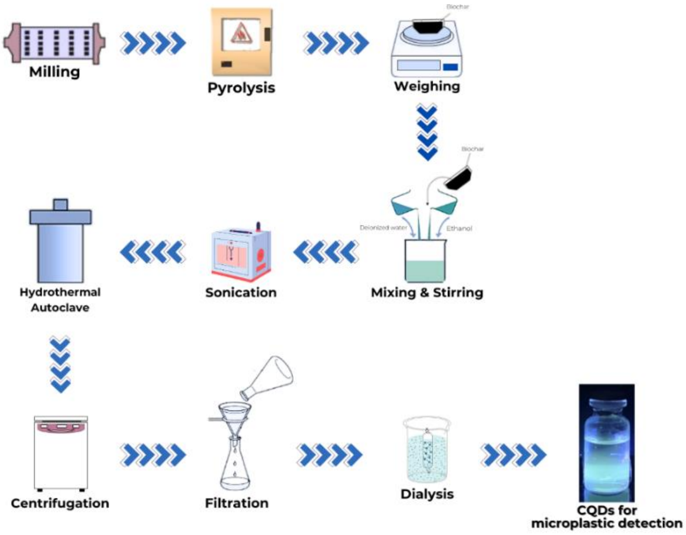


Figure 1 Schematic of the synthetic process for the CQDs from oil palm fronds.

2.3. Microplastics Detection

Polyethylene and polyethylene terephthalate with variations of 0.2 g, 0.4 g, and 0.6 g was added to a 10 mL CQDs solution, then was exposed to 365 nm UV light to study the emission of CQDs produced (Zhao et al., 2023).

3. Results and Discussion

3.1. Biochar Production

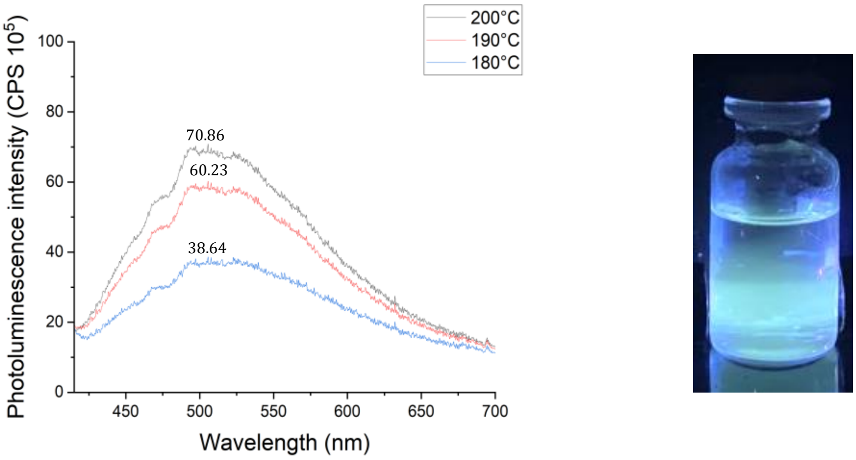
OPF was successfully converted into biochar by the pyrolysis method with operating conditions at 400°C with a heating rate of 10°C per minute, 10 minutes holding time and with the presence of N_2 to create an inert environment inside the reactor (as shown in Figure 2). The biochar yield was approximately 39.45%, which is notably lower than that reported in earlier studies(Bindar et al., 2022). This difference in yield may be attributed to variations in the water content of the OPF before pyrolysis and the lignocellulose ratio in the OPF.

Figure 2 Produced biochar from pyrolysis process.

3.2. CQDs Characterization

Carbon quantum dots were successfully produced with hydrothermal method at 180°C, 190°C, and 200°C. The variations in heating temperature significantly affected their characteristics, such as photoluminescence (PL) intensity and morphology. The temperature variation for hydrothermal was limited to a maximum of 200°C due to the hydrothermal reactor's maximum heating condition. PL intensity was observed with a hybrid fluorescence spectroscopy at an excitation wavelength of 405 nm. This excitation wavelength was selected based on the emission colour displayed by the CQDs under UV light, which is bluish-green. Several previous studies have also reported that excitation at 405 nm exhibits stronger fluorescence intensity than excitation at 360 nm (Pan et al., 2008).

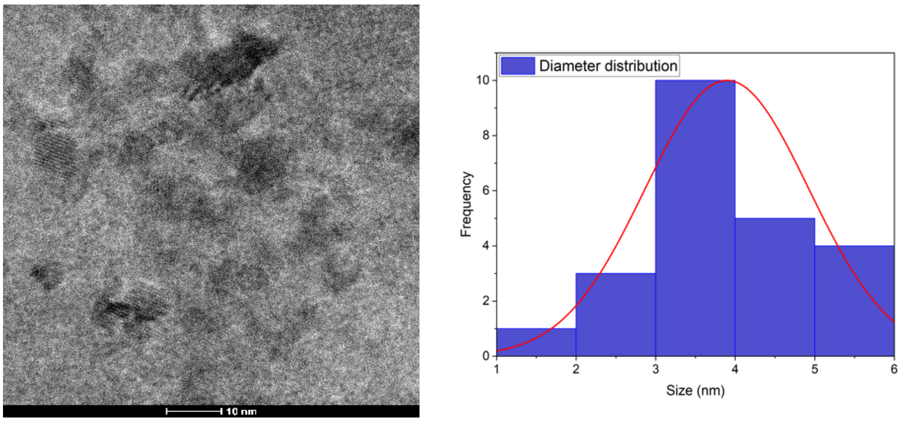
With CQDs producing at 200°C being the highest intensity, as shown in Figure 3, this variation shows the best potential of CQDs as a detector for microplastic detection. Figure 3(a) shows that the intensity data for CQDs prepared at 180°C, 190°C, and 200°C are 38.64×10^5 CPS, 60.23×10^5 CPS, and 70.86×10^5 CPS, respectively. These results confirm that CQDs produced at 200°C exhibit the highest intensity, supporting their use as simple biosensors for microplastic detection through both visual and quantitative methods (Hallaji et al., 2023). The solution also emits a bright blue-green light after exposure to UV light, showing that the CQDs produced have a rough diameter size of 2-5 nm (Ding et al., 2021; Das et al., 2019), which confirmed a successful CQDs production using hydrothermal method. The size and morphology of CQDs were later analyzed using a High-resolution transmission electron microscopy (HR-TEM) instrument.



(a) (b)

Figure 3(a) Photoluminescence intensity of CQDs at 180°C, 190°C, and 200°C; (b) Emission of CQDs under UV light

The size distribution of CQDs shows an average of 4.045 ± 1.245 nm, in accordance with the definition of CQDs being quantum dots with less than 10 nm size. Controlling the size of carbon quantum dots (CQDs) using top-down methods presents significant challenges compared to bottom-up approaches (Nallayagari et al., 2022). The top-down synthesis often results in a broader size distribution and less precise control over the final dimensions of the CQDs. Visual-wise, the CQDs produced were more aggregated than dispersed, as shown in Figure 4(a). This phenomenon may occur due to concentration, solvent characteristics and functional groups on the surface of CQDs (Ru et al., 2022). Aggregation can result in aggregation-induced emission (AIE), occurring when CQDs are synthesized at higher temperatures because of temperature’s effect on particle size. AIE is a phenomenon in which CQDs exhibit stronger fluorescent emission in aggregated form compared to dispersed CQDs (Praveena et al., 2024; Adsetts et al., 2020). Enhanced emission signals can improve detection sensitivity, making CQDs highly effective for microplastic detection. Other than morphology, CQDs were also analyzed with UV-visible (UV-Vis) spectroscopy and FTIR (Fourier-transform infrared) spectroscopy to study their chemical composition.



(a) (b)

Figure 4 (a) Morphology of CQDs at 200°C, observed under HR-TEM at 590,000x magnification. (b) Distribution of CQDs diameter size.

The UV-Vis spectrum for 200°C from Figure 5 shows that the highest peak was found at a wavelength of 291.2 nm. CQDs with a wavelength of 200-300 nm have strong absorption due to the π-π^* transition of the aromatic C=C bond in the carbon structure (Yadav et al., 2023). The FTIR spectrum (Figure 6) was dominated by the presence of O-H, C=O, and C=C functional groups, which are associated with carbon and water. These results further supported the CQDs production with the hydrothermal method at 200°C.

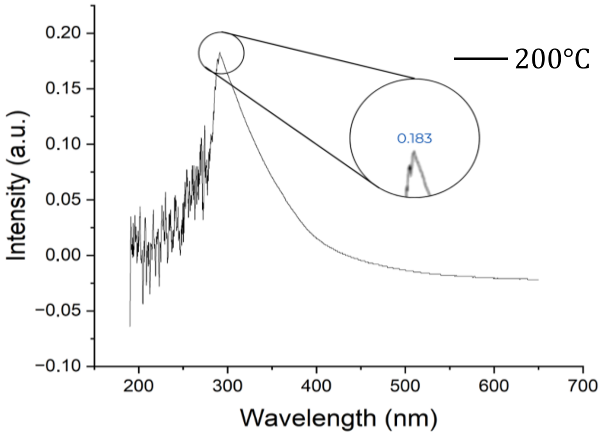


Figure 5 UV-visible spectrum of biochar-based CQDs from OPF at 200°C.

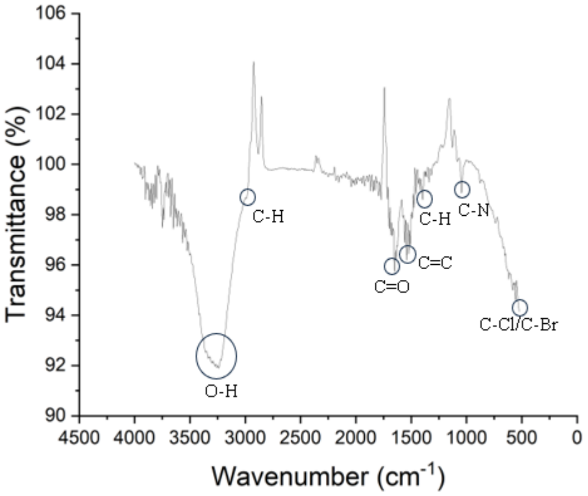


Figure 6 FTIR spectrum of CQDs synthesized at 200°C

3.3. Microplastics Detection

The addition of microplastics to CQDs solution was expected to decrease the photoluminescence intensity of the solution due to the quenching mechanism (Zu et al., 2017). Quenching in this study refers to the interaction between hydroxyl groups and the surface of microplastics through hydrogen bond formation. Several quenching mechanisms, such as static quenching and dynamic quenching, can occur in the process of microplastic detection by CQDs. Functional groups on the surface of microplastics (e.g., ester, amide, or hydroxyl) can interact with functional groups on the surface of CQDs (e.g., carboxyl, amino, or hydroxyl), forming a stable complex which can be seen in Figure 7. These stable complexes prevent the generation of non-radiative defect states on the CQDs, which may help protect against degradation under various conditions (Dua et al., 2023).

Functional groups can also increase the conjugation of CQDs, enhancing fluorescence intensity for better microplastic detection (Zhao et al., 2023).

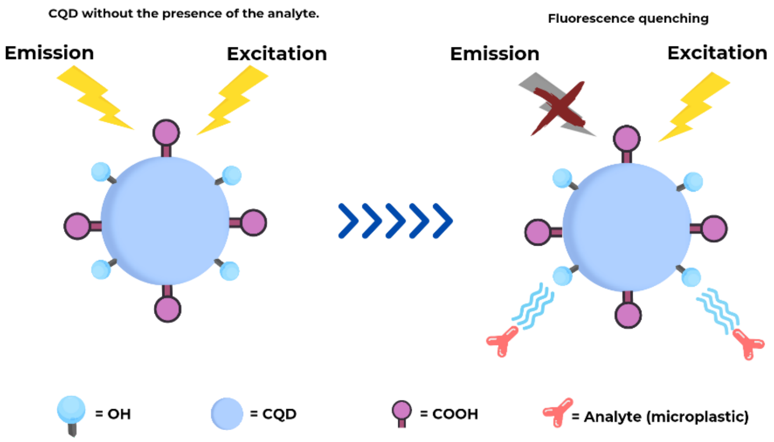
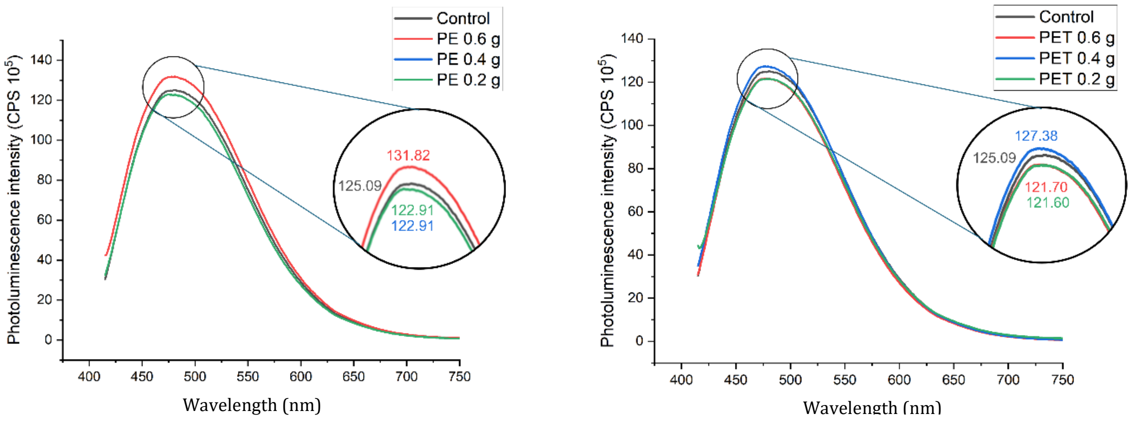


Figure 7 Schematic of quenching mechanism in CQD sensors.



(a) (b)

Figure 8 (a) CQDs with polyethylene microplastics addition. (b) CQDs with polyethylene terephthalate microplastics addition.

For variations 0.2 g and 0.4 g of polyethylene (PE) microplastics, Figure 8 showed that photoluminescence intensity for both variations resulted in the same value which is 122.91 × 10^5 CPS. In contrast, the variation of 0.6 g showed an increase in intensity reaching up to 131.82 × 10^5 CPS, which is even higher than the control (125.09 × 10^5 CPS). This phenomenon may occur because of the CQD limit of detection and the amount of solution used as a detector. However, the result for polyethylene terephthalate (PET) microplastics was different from PE. While the variations 0.2 g and 0.6 g of PET showed a decrease in intensity with values peaked at 121.60 × 10^5 CPS and 121.70 × 10^5 CPS, respectively, the 0.4 g PET variation exhibited an increase in intensity, reaching 127.38 × 10^5 CPS, which was higher than the control. Storage and exposure to light can also influence the stability of CQDs (Dua et al., 2023). Overall, the data suggests that the quenching mechanism has occurred, as indicated by the reduced fluorescence intensity (Zu et al., 2017). However, further studies are required to investigate the effects of varying CQD concentrations.

4. Conclusions

CQDs produced at 200°C with the hydrothermal method possess great potential as microplastic detectors. The characteristics of CQDs, such as <5 nm size, chemical composition dominated by the presence of O-H, C=O and C=C groups and proven to have π-π^* transitions from the aromatic C=C bonds in the carbon structure. CQDs also display the good ability to detect microplastics as they show a decreasing trend in addition to the solution. While this study demonstrates a promising application of CQDs in detecting PE and PET microplastics, the challenges related to the limit of detection, stability and interactions with various microplastic types must be addressed to fully realize the potential of CQDs. Despite these challenges, the versatility and broad applicability of CQDs in environmental monitoring and other fields highlight their immense potential, suggesting a promising future for their development and utilization.

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Author Contributions

NSS and ACK: Conceived, designed the experiments, supervised, writing and finalized the draft. MGM: Methodology, formal analysis, data curation, and writing original draft. IS: Methodology. RR, FA, KL: review and editing. DDP and RS: Editing and Finalized the draft.

Conflict of Interest

The authors declare no conflicts of interest.

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