Microwave-Assisted Synthesis of Carbon Quantum Dots (CQDs) from Biomass-derived Oil Palm Trunks Biochar for Lead Ion (Pb^{2+}) Detection

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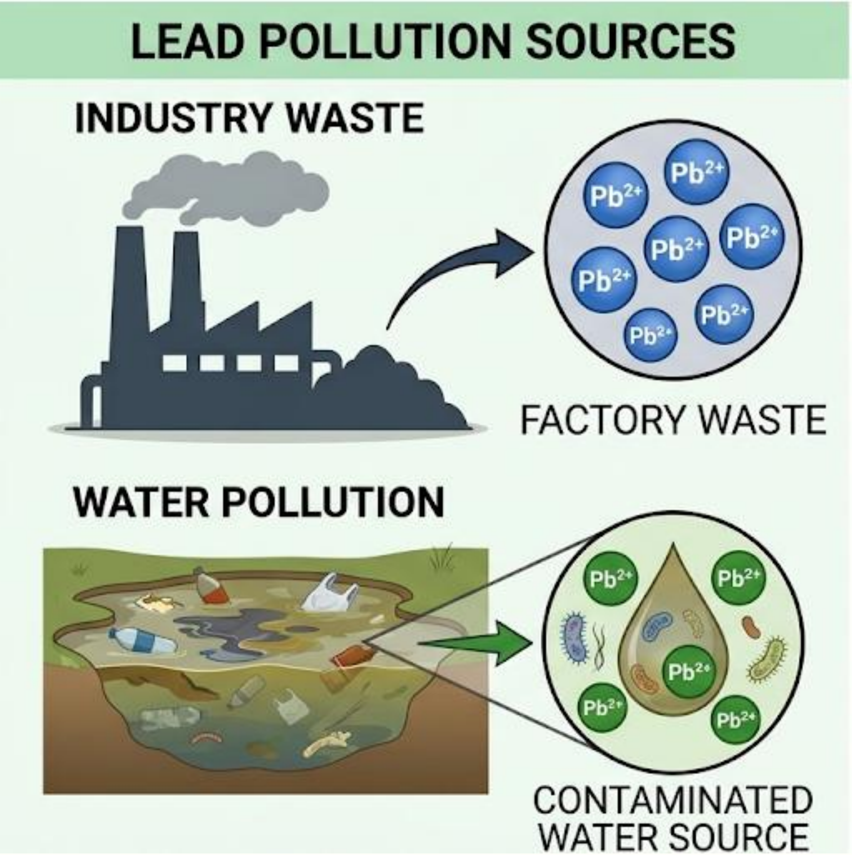
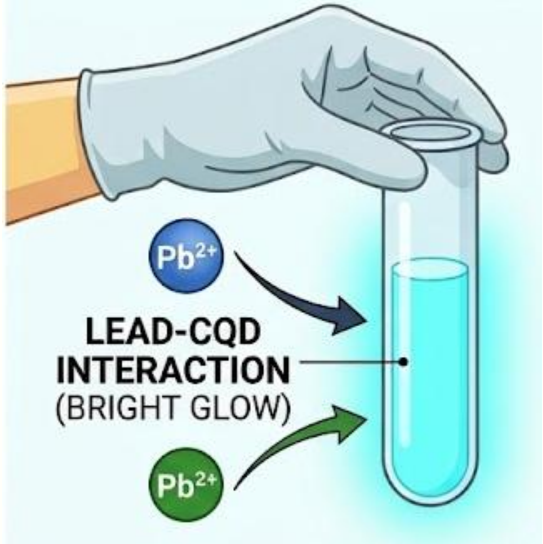
- **Lead (Pb^{2+}) is highly toxic** heavy metal often found in industrial wastewater, posing severe health risks even at low concentrations (*Mandal et al., 2022*).
- Conventional methods are **costly, time-consuming, and require bulky laboratory equipment.**
- Therefore, a fast, simple, and affordable Pb^{2+} detection method is needed.



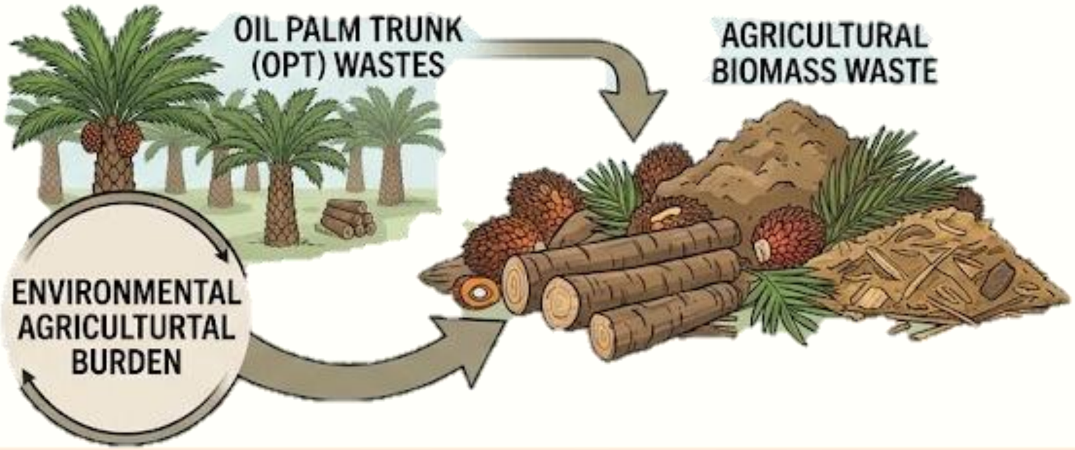
Why CQDs for Pb^{2+} Detection?

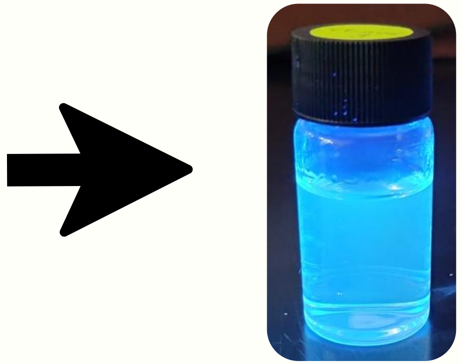
- ✓ **Abundant Functional Groups:** Carboxyl and hydroxyl groups on the CQDs surface selectively bind to Pb^{2+}.
- ✓ **Fluorescence Quenching:** Pb^{2+} binding immediately dims the CQDs' light output, creating an easy-to-read signal.
- ✓ **High Surface Area:** Nanoscale size maximizes contact points with Pb^{2+}.
- ✓ **Water Soluble:** Disperses easily in natural water samples without needing toxic chemical solvents.
- ✓ **Instant Results:** Rapid, real-time detection without complex, time-consuming lab equipment.



Where can sources of CQDs be obtained?

- ✓ **Origin:** Derived sustainably from agricultural biomass waste, specifically **Oil Palm Trunk (OPT)**.
- ✓ **Abundance:** Highly available from major agricultural waste.
- ✓ **High Carbon Content:** Naturally rich in carbon matrix, serving as an ideal precursor for CQDs synthesis.
- ✓ **Waste-to-Value:** Converts environmental agricultural burden into high-value functional nanomaterials.





How can CQDs be synthesised?

- **Microwave-assisted** method was selected because it is **rapid, energy-efficient, simple**, and suitable for producing CQDs from biomass with good control over their properties.
- In microwave-assisted methods the precursors are heated due to absorption of the direct microwave radiation.
- This process **is more energy efficient** because energy is absorbed directly by the molecules instead of the reaction vessel.

Methodology: Microwave-assisted method

OPT pyrolysis

- Temperature: 700˚C.
- Heating rate: 5 °C/min (under a constant N_2 purge)
- Soaking time: 2 hours.

Precursor preparation

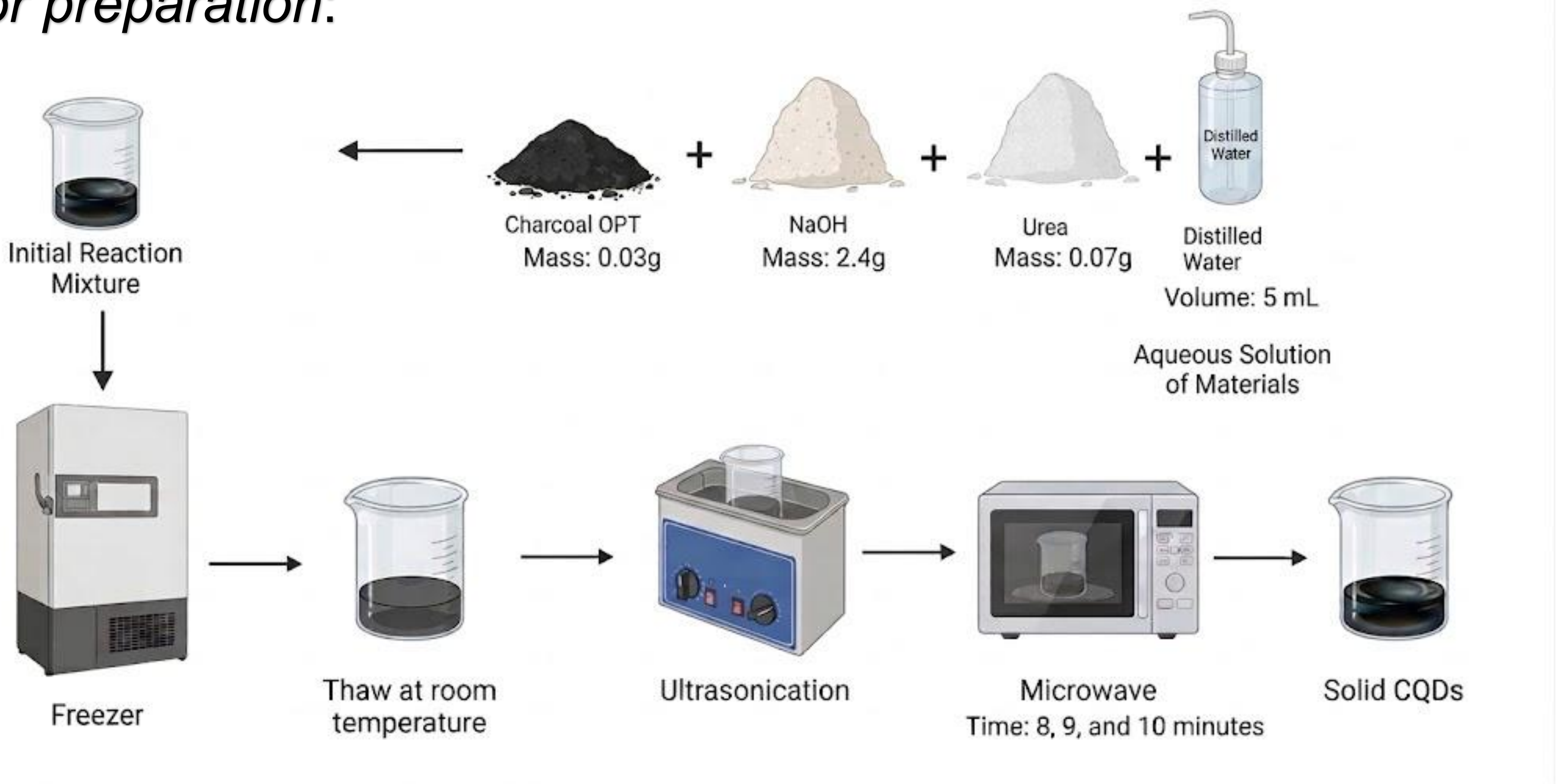
- Mass: 0.03g mixed with 5 mL distilled water.
- Activator: NaOH (0.07g) mixed with Urea (2.4g)
- After being microwaved, the samples were transferred to the freezer and allowed to remain there overnight.

Sample characteristics and Pb^{2+} testing

- CQDs were tested for UV-Vis, FTIR, and PL analysis.
- Using 50 ppm of Pb^{2+} and testing using PL analysis to check the photoluminance and quenching effect to the CQDs.

Methodology: Microwave-assisted method

Precursor preparation:

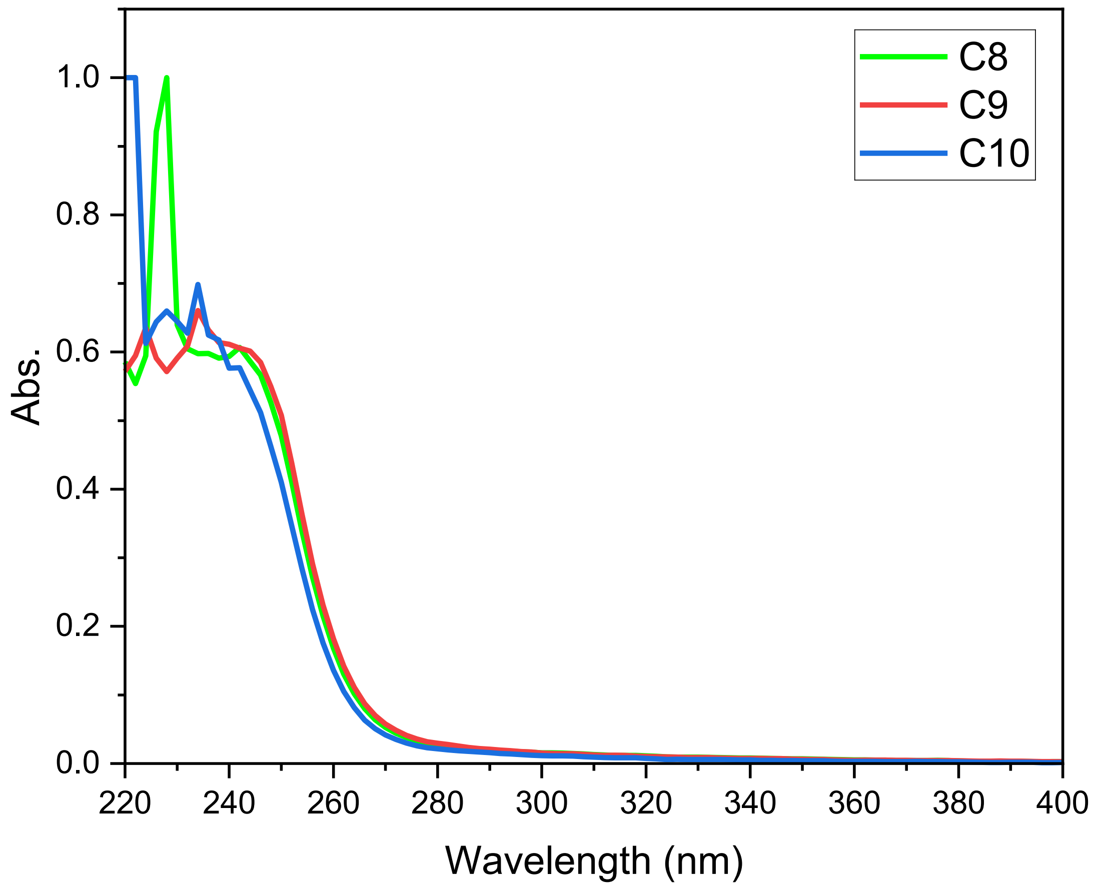


- Sample characteristics: UV-Vis, FTIR and PL analysis
- Detection of Pb^{2+} using PL analysis – to evaluate the detection capability for Pb^{2+} (quenching effect)

Results & Discussion:

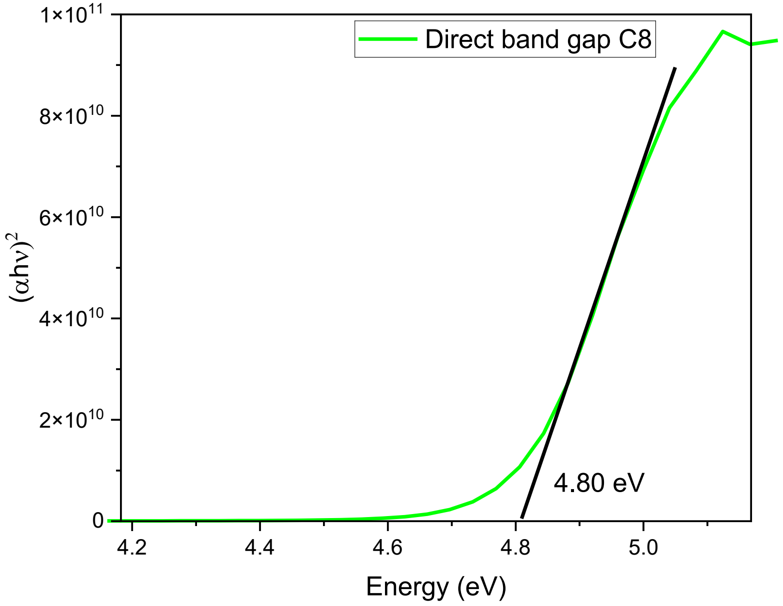
UV-Vis Analysis:

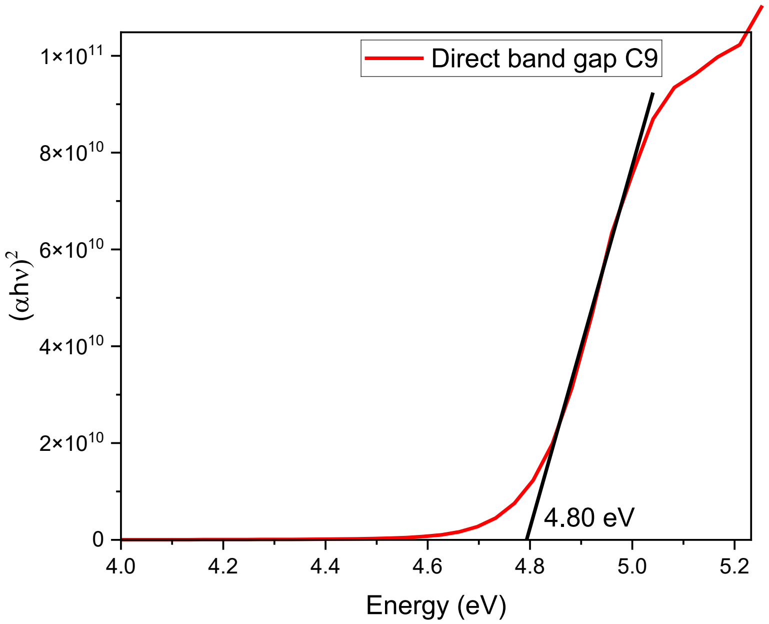
- **Primary Absorption Band (220–260 nm):**
  - Broad peak features at centered around 230–250 nm, typical for aromatic rings, carbonyl transitions ($\pi \rightarrow \pi^*$ or $n \rightarrow \pi^*$) in sp^2 carbon domains.
- Peak Differences Below 240 nm:
  - **C8 (Green):** Exhibits high-intensity sharp peak at approximately 230 nm, due to π – π* transition of aromatic C=C bonds in sp^2 hybridisation of aromatic domains.
  - **C9 (Red):** Displays a minor peak near ~234 nm indicates π–π* transition with possible contribution from surface states.
  - **C10 (Blue):** Shows a sharp spike at 220 nm and a moderate local peak at ~234 nm, also refer to π–π* transition

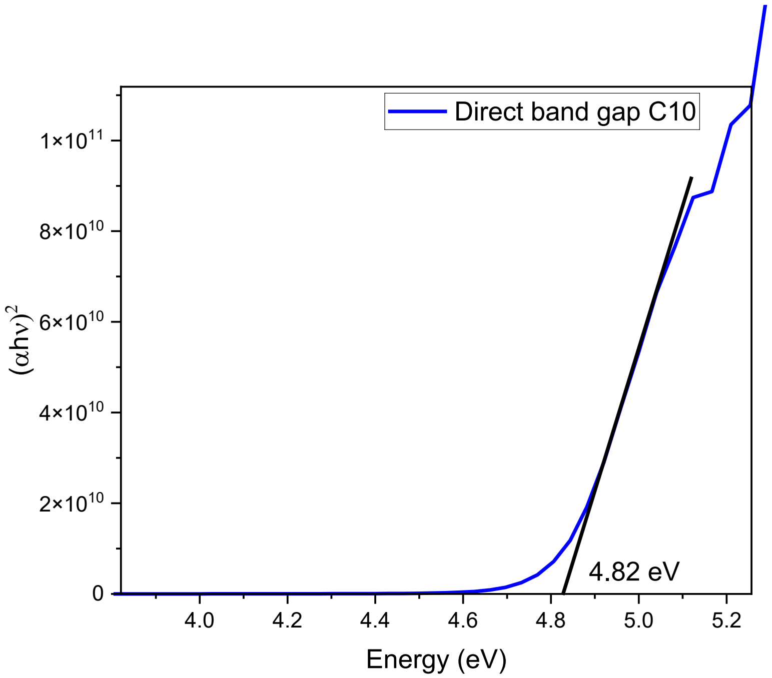


Results & Discussion:

UV-Vis Analysis:



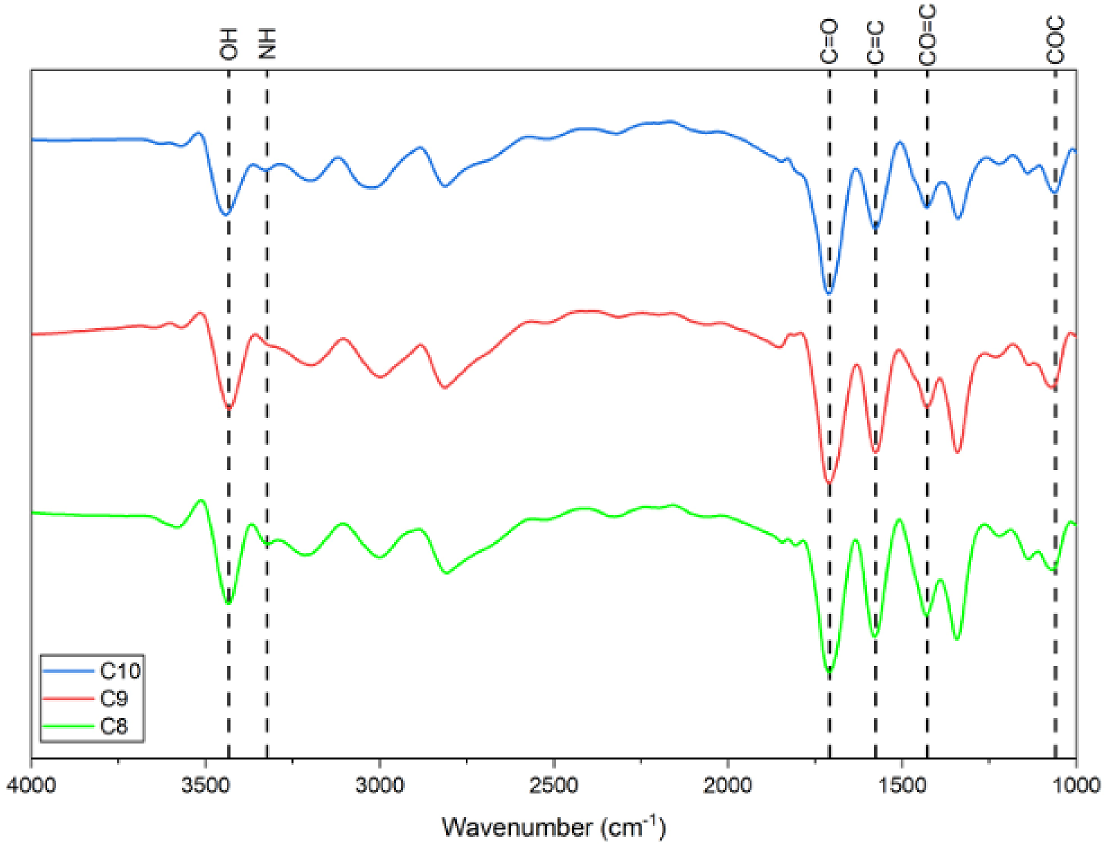




- The band gap energy was calculated using the **Tauc plot**.
- The **smaller the carbon domains**, the **stronger the quantum confinement** in biochar OPT to happen.

Results & Discussion:

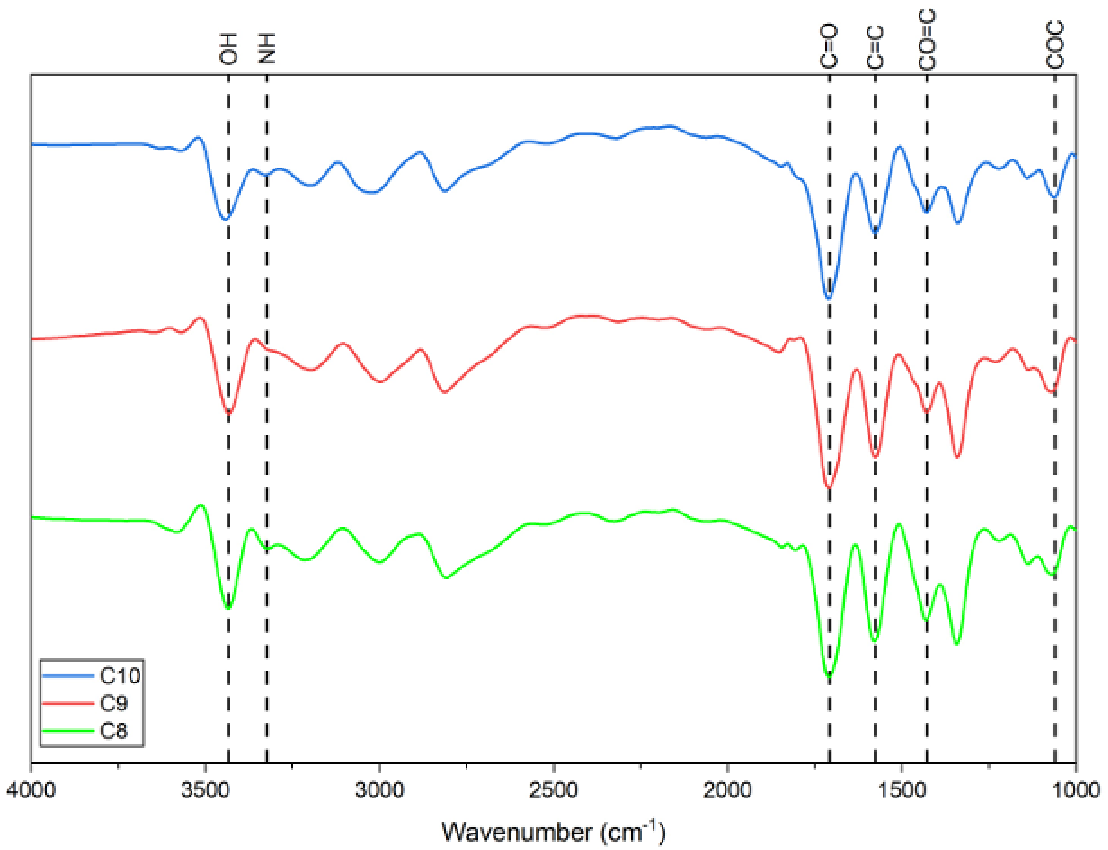
FTIR Analysis:



- **High-frequency region (3200–3400 cm⁻¹):**
  - Broad peak at ~3400 cm⁻¹ corresponds to O–H stretching (hydroxyl groups).
  - Shoulder at ~3200 cm⁻¹ corresponds to N–H stretching (amine groups).
  - These groups transform charcoal from hydrophobic to hydrophilic, enabling stable aqueous dispersions.
  - Essential for aqueous metal ion sensing applications.

Results & Discussion:

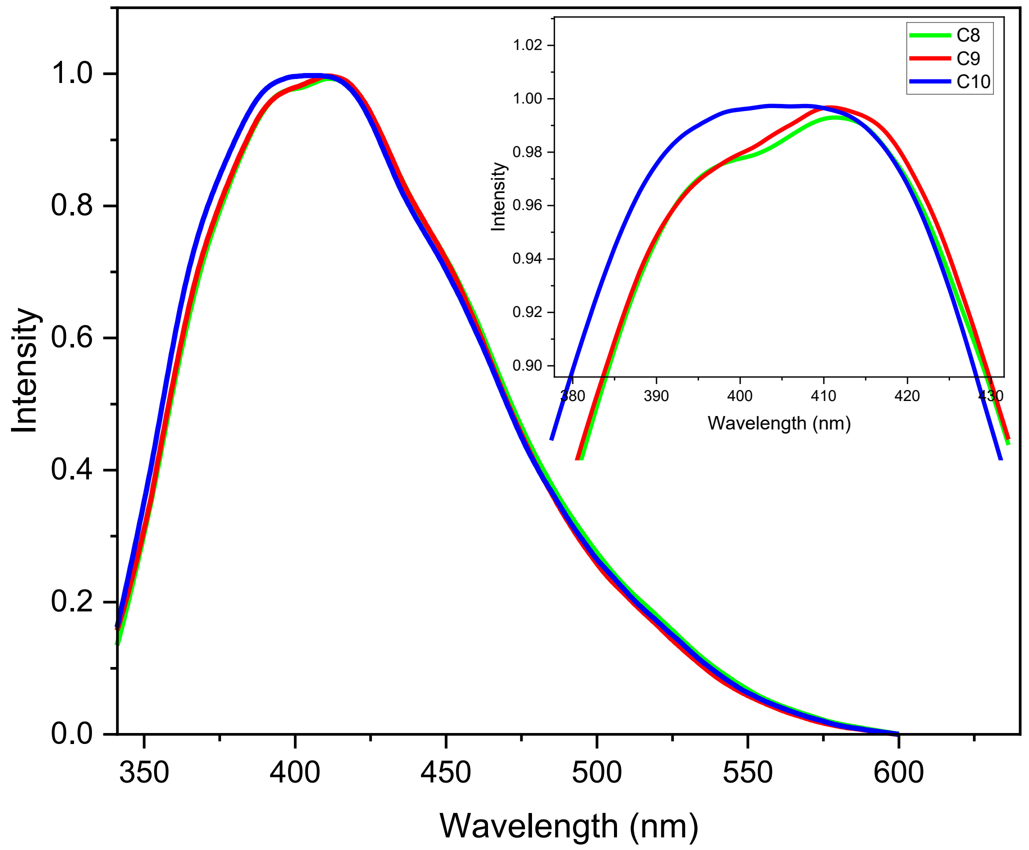
FTIR Analysis:



- **Fingerprint region (1600–1800 cm^{-1}):**
  - Sharp peak at ~1720 cm^{-1} indicates C=O stretching (carbonyl/carboxyl/amide groups).
  - Peak at ~1640 cm^{-1} corresponds to C=C stretching of the graphitic core.
  - The well-defined C=C peak suggests a highly ordered sp^2 carbon structure, inherited from the charcoal precursor.
- **Lower-frequency region (1000–1450 cm^{-1}):**
  - Peaks near 1450 cm^{-1} indicate C–N stretching, confirming nitrogen doping from urea.
  - Peak at ~1050 cm^{-1} corresponds to C–O–C or C–O stretching.
  - These oxygen- and nitrogen-containing groups enhance fluorescence and provide active sites for Pb^{2+} binding.

Results & Discussion:

PL Analysis:

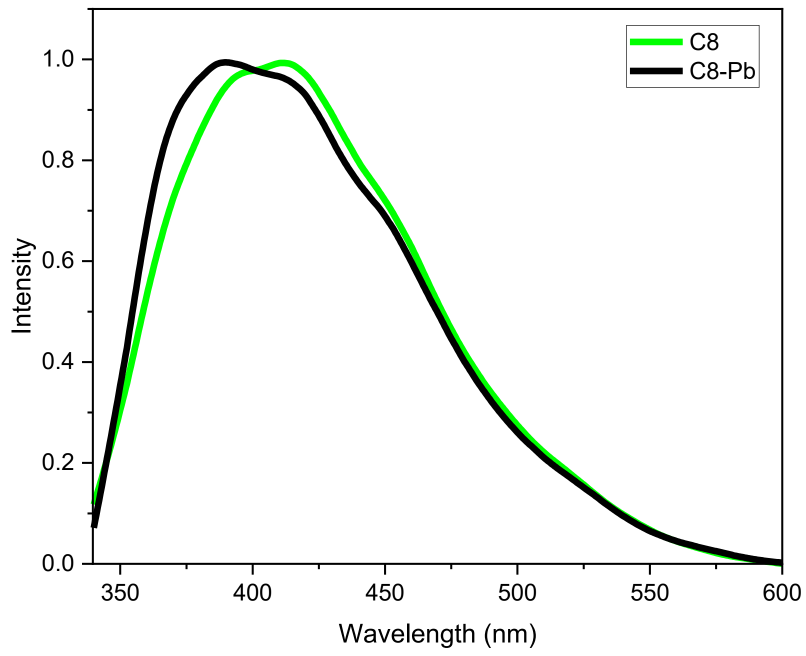


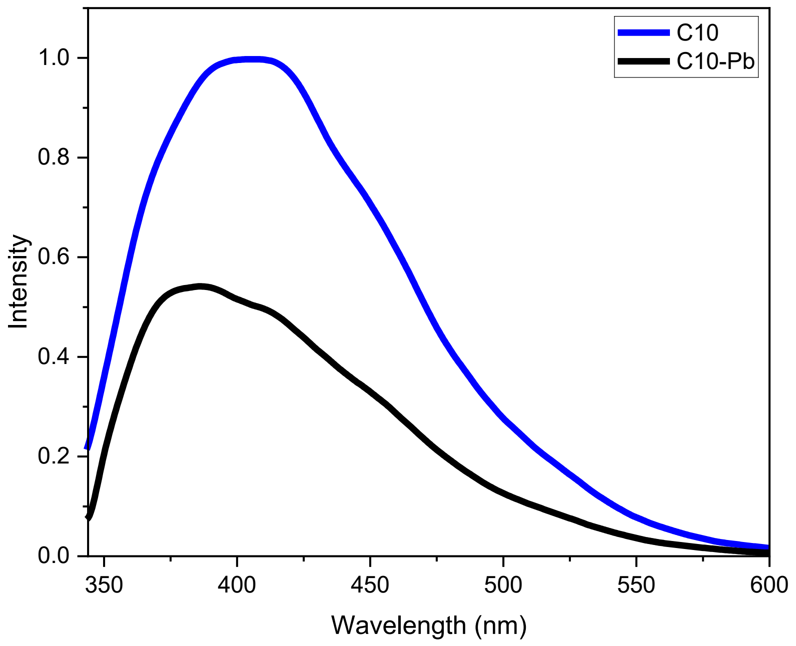
- Biochar CQDs show emission peak changes with time.
- Emission peaks for C8 and C9 at 411–411.5 nm, respectively.
- Emission peak for C10 shows **blue-shifts** to 405.5 nm.
- The size reduction is attributed to prolonged microwave irradiation, which causes **surface etching or fragmentation of graphitic domains**.
- The size estimation was calculate using **effective mass approximation (EMA) model.**

Sample	Emission peak (nm)	Calculated CQDs radius, r (nm)	Calculated CQDs diameter, d (nm)
C8	411	1.781	3.562
C9	411.5	1.783	3.566
C10	405.5	1.759	3.519

Results & Discussion:

PL Analysis:





- A **blue-shift trend** was observed for the Biochar OPT samples.
- Emission peak for **C8** is shifted from **411 nm to 391.5 nm.**
- Emission peak for **C10** is shifted from **405.5 nm to 383.5 nm.**
- Fluorescence intensity dropped significantly.

Samples	Emission peak (nm)	Calculated CQDs radius, r (nm)	CQDs diameter, d (nm)
C8-Pb	391.5	1.706	3.413
C10-Pb	383.5	1.676	3.352

Results & Discussion:

PL Analysis:

Samples	Emission peak (nm)	CQDs diameter, d (nm)
C8	411	3.562
C8-Pb	391.5	3.413
C10	405.5	3.519
C10-Pb	383.5	3.352

- Results show a measurable reduction in effective particle size for all samples after Pb^{2+} exposure.
  - **C8**: ~3.562 nm → ~3.413 nm (≈0.149 nm reduction)
  - **C10**: ~3.519 nm → ~3.352 nm (≈0.167 nm reduction)
- Effect of synthesis duration:
  - Samples synthesized for **10 minutes show the higher diameter reduction.**
  - Longer synthesis time produces:
    - **Higher density of active surface** functional groups
    - **Stronger Pb^{2+} binding**
    - Greater modification of the CQDs electronic structure.

Conclusion:

- **Sustainable & Low-Cost Precursor**: CQDs were successfully synthesized from agricultural biomass waste (OPT Biomass) and converting an environmental burden into high-value nanomaterials.
- **Rapid & Energy-Efficient Synthesis**: The **microwave-assisted method** was effective because energy is absorbed directly by the precursor molecules, providing a rapid, energy-efficient reaction with good control over the CQD properties.
- **Clear Sensing Signal via Photoluminescence Quenching**: Exposure to 50 ppm Pb^{2+} produced significant fluorescence quenching, blue-shifting of photoluminescence emission peaks, and a measurable reduction in calculated effective particle diameter.

Acknowledgment:

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TERIMA KASIH / *THANK YOU*

Any Questions?