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Photoluminescence sensing platform for the detection of 2-Nitrophenol based on Cu-doped carbon dots

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Abstract— **In this study, we presented a facile biogenic strategy to synthesis Cu-doped carbon dots (Cu- CDs) through one-step hydrothermal approach using $CuCl_2·2H_2O$ and dried Ziziphus Mauritiana leaves as reaction precursor. The synthesized CDs were characterized by Photoluminescence, UV-visible spectra, FTIR, EDX, DLS, and FESEM techniques. The synthesized Cu-CDs investigated for detection of 2-nitrophenol through fluorescence. The sensor exhibited high selectivity and sensitivity for the detection of 2-nitrophenol on account of the inner filter effect. Under optimal conditions, the linear detection range is 0-250 µM with the detection limit of 0.025 µM. This method is successfully applied in quantification of 2-nitrophenol in real water samples, which exhibits potential application prospects.**

Index Terms— **Carbon dots, Copper, Ziziphus mauritiana leaves, 2-Nitrophenol**

I. INTRODUCTION

Nitrophenol chemicals, particularly 2-nitrophenol (2-NP) and its isomers, pose a substantial hazard to both ecological and human health due to their toxicity, persistence, and bioaccumulation [1–3]. Conventional analytical methods, like gas chromatography and high-performance liquid chromatography in conjunction with mass spectrometry, provide excellent accuracy but are frequently constrained by difficult sample preparation, high expense, and restricted portability for in situ monitoring [4–6]. These intrinsic constraints have prompted the creation of optical sensing platforms that can detect objects in ambient settings quickly, sensitively, and selectively.

Photoluminescence (PL) based sensing has emerged as a promising optical transduction strategy due to its high sensitivity, ease of signal readout, and downsizing potential [7–9]. The mechanisms that PL sensors usually work on include fluorescence increase or quenching brought on by interactions between the analyte and a fluorescent probe, these mechanisms are frequently linked to charge transfer processes, energy transfer, or inner-filter effects [10–12].

As fluorescent probes, carbon dots (CDs) and carbon quantum dots (CQDs), a subclass of carbon nanomaterials with diameters less than 10 nm, have garnered a great deal of interest [13–17]. Compared to conventional semiconductor quantum dots, CDs, which are made of graphitic carbon cores with an abundance of surface functional groups, have high PL emissions, tunable optical characteristics, good water dispersibility, and low toxicity [18–20].

The simplicity of synthesis from various carbon sources, as well as the flexibility to adjust their photophysical properties by elemental doping or surface functionalization, increase their versatility in sensing applications [21–24]. It has been demonstrated that elemental doping, particularly with heteroatoms like N, B, S, or transition metals (e.g., Fe, Cr, Cu), significantly affects the electronic structure and surface states of CDs, resulting in improved interaction with target analytes, enhanced quantum yields, and customized emission behavior [25–29]. For example, highly selective fluorescent assays for p-nitrophenol detection have successfully used boron, nitrogen copper-doped CDs, where quenching occurs due to inner-filter and surface contact effects [30]. Similarly, through inner-filter effect quenching, chromium (III)-doped CDs have shown sensitive fluorometric detection of p-nitrophenol with low detection limits [31].

Despite improvements in sensing phenolic contaminants with heteroatom-doped CDs, transition metal-doped CDs, particularly those containing copper, are still underexplored for nitrophenol detection. By adding additional energy levels and strengthening certain interactions between the dopant sites and target analytes, transition metal doping into the carbon dot matrix may increase sensitivity and selectivity [32–34]. Building on this idea, Cu-doped carbon dots (Cu-CDs) have recently been created and effectively used as fluorescent probes for the detection of p-nitrophenol through quenching mechanisms caused by electron transfer and inner-filter effects [35]. The potential of Cu-CDs as effective luminous sensors for structurally related nitrophenol analytes is demonstrated by these investigations.

However, a great deal of information is still lacking on the precise effects of Cu doping on the PL response to 2-NP, an isomer of p-nitrophenol with unique chemical and electrical characteristics. Therefore, more understanding of the synthesis-structure-response linkages of Cu-CDs is required, especially in regard to sensing performance metrics including interference resistance, selectivity against analogues, limit of detection (LOD), and application in actual environmental matrices. Furthermore, a thorough mechanistic comprehension of the fluorescence amplification or quenching processes caused by 2-NP is yet lacking.

In this study, we produced metal Cu-CDs in a one-step hydrothermal approach and developed a simple yet effective method to detect 2-NP using Cu-CDs. Within two minutes, this analytical system responded to 2-NP with speed. Importantly, the inner filter effect (IFE) induced fluorescence quenching of 2-NP towards Cu-CDs was suggested as the potential cause of the sensing process. Notably, the proposed approach was successfully used to real water samples for the 2-NP assay, indicating that the generated Cu-CDs have a great deal of promise for real-world use in the environmental area. [36-39]

II. EXPERIMENTAL SECTION

2.1. Chemical and materials

Chloride salts of (zinc (Zn^{2+}), magnesium (Mg^{2+}), sodium (Na^{+}), potassium (K^{+}), from Sigma Aldrich (~ 99 % purity). Ascorbic acid, Phenol, 2-nitrophenol, glucose, 4-aminophenol, citric acid, coper nitrate, distilled water, picric acid. The procured chemicals are used as such without doing any other purification process. All other reagents were of analytical grade and no need to be further purification when using. Distilled water was used throughout.

2.2. Instruments

The dynamic light scattering (DLS) was carried out on Horiba Scientific Nano Particle Analyser SZ-100V2. PerkinElmer spectrophotometer was used to record Fourier transform infrared (FTIR) spectra. The absorbance of synthesized carbon dots has been explored by UV-Vi's spectrophotometer (Shimadzu UV-2600). The elemental composition was carried by energy dispersive X-ray spectroscopy (EDX) on a JEOL JSM -7600F system. Horiba FluoroMax-4 spectrometer was employed for examine the fluorescence properties of synthesized material.

2.3. Preparation of Cu-doped CDs

The Cu-CDs were prepared by one step hydrothermal treatment of ber (Ziziphus mauritiana) leaves and $CuCl_2 \cdot 2H_2O$ as reaction precursor as shown in Figure 1. Briefly some dried ber leaves were crushed and 2mg $CuCl_2.2H_2O$ were dissolved in 30 ml distilled water under bath sonication for 5 minutes. Following the mixture solution was transferred into a 50 ml Teflon equipped stainless steel autoclave, the reaction system was sealed and heated at 180 °C for 3h. After the obtained solution was cooled down to room temperature naturally, the products were collected by centrifuging. Finally, the obtained Cu-CDs solution was washed and purified.

2.4. Fluorescence analysis of 2-NP

A 2-NP assay was performed using the protocols outlined below.200 µL of the Cu-CDs solutions were added to 1 mL of Distilled water before various 2-NP doses were added. After that, the solution was diluted with 2 milliliters of ultrapure water and allowed to sit at room temperature for two minutes in order to fully react. Then, the following spectrofluorometery parameters were used to measure the fluorescence intensities: excitation and emission band-passes of 10 nm, photomultiplier tube voltage of 900 V.

2.5 Assay of 2-NP in Real water

The standard addition method was used to examine this probe's performance for the 2-NP test in actual water samples.

III. RESULTS AND DISCUSSION

3.1. Characterization of the Cu-CDs

The FT-IR spectra of clean CDs and Cu-CDs show distinct bands representing surface functional groups. O-H/N-H stretching is represented by a large peak at 3200–3500 cm⁻¹, whereas aliphatic C–H stretching is responsible for the bands at 2920–2850 cm⁻¹. C=O stretching is attributed to the peak at 1650–1700 cm⁻¹, while aromatic C=C vibrations are attributed to the band at 1550–1600 cm⁻¹. C–N and C–O groups are indicated by signals in the 1200–1400 cm⁻¹ range. The carbon core structure of Cu-CDs is preserved while the coordination of Cu ions with surface functional groups is confirmed by minor changes in the hydroxyl and carbonyl regions as well as new bands at 500–700 cm⁻¹ (Cu–O/Cu–N). UV-Vis. spectra indicate a high absorption at 250-280 nm due to π-π* transitions of aromatic C=C bonds, and a shoulder at 300-350 nm due to n-π* transitions of surface groups. Cu-CDs show slight broadening and intensity variations, suggesting electronic interactions between surface states and Cu species. The lack of a discernible plasmonic band indicates that copper is coordinated as opposed to metallic. For both samples, fluorescence emission (λex = 330 nm) displays a blue emission peak at 423 nm. Cu-CDs exhibit somewhat lower intensity, suggesting that metal-ligand interactions and increased non-radiative routes have partially quenched fluorescence. EDX mapping verifies the regular distribution of C, O, and Cu elements, and FESEM pictures show a thin, wrinkled nanosheet-like shape (Figure 4).

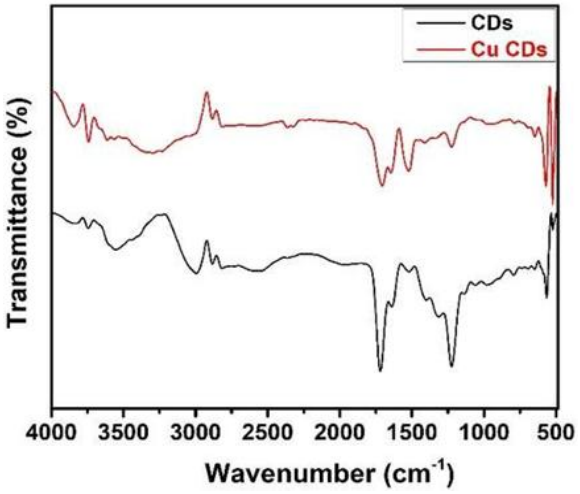

Figure 1. FT-IR spectra of CDs and Cu-doped CDs.

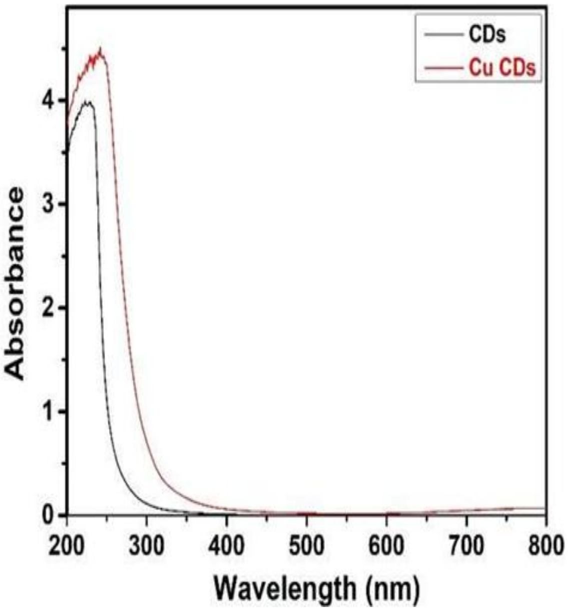

Figure 2. UV-Vi's spectra of CDs and Cu-doped CDs

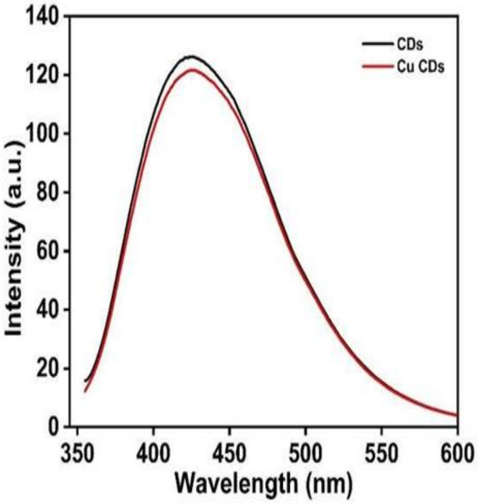

Figure 3. PL spectra of CDs and Cu-doped CDs.

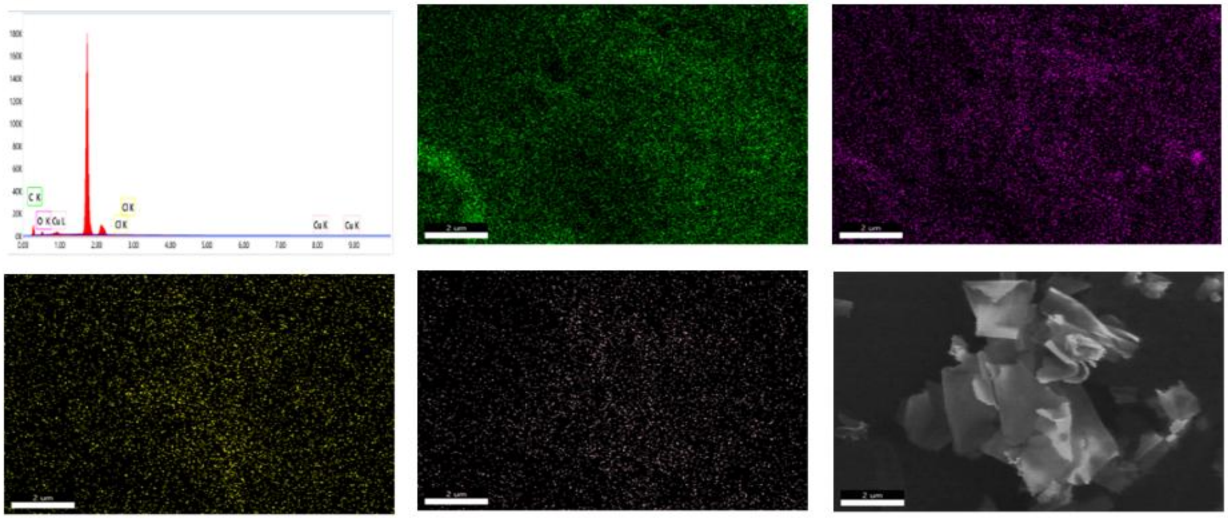

Figure 4. EDX mapping and FESEM image of Cu-doped CD

3.2. Optimization and optical properties of synthesized Cu-CDs

The photoluminescence spectra of Cu-CDs recorded at different excitation wavelengths (300-350 nm) exhibit excitation-dependent emission. Notably, the highest PL intensity is observed at 330 nm (Figure 5a), indicating the most efficient excitation of emissive states. Therefore, 330 nm was selected as the optimal excitation wavelength for subsequent studies.

The pH-dependent PL response of Cu-CDs was evaluated over a wide pH range. The emission intensity varies significantly with pH, indicating the sensitivity of surface functional groups to protonation-deprotonation processes. Maximum fluorescence is observed at neutral conditions (Figure 5b), while quenching occurs under highly acidic and basic environments, therefore all experiments were performed at pH 7.

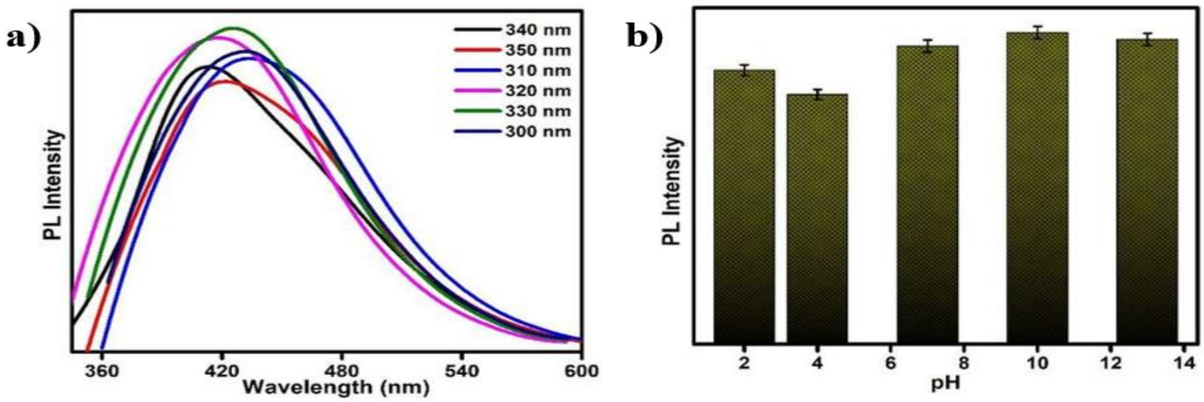

Figure 5. PL spectra at (a) different wavelengths and (b) different pH values.

3.4. Fluorescence assay for 2-NP

Cu-CDs are considered to be an excellent candidate for detecting 2-NP under optimal conditions. The response of Cu-CDs fluorescence to varying concentrations of 2-NP in the Cu-CDs solution was seen in Fig. 6(a), 6(b). It is evident that as concentration of 2-NP increases, the fluorescence intensity of Cu-CDs steadily drops. The fluorescence response (F_0/F) of Cu-CDs shows a concentration-dependent increase with analyte addition. A gradual enhancement is observed at lower concentrations, followed by a sharp rise at higher concentrations, indicating strong interaction between the analyte and Cu-CDs (Figure 7). The limit of detection (LOD) is as low as 0.025 μM (R2 = 0.998,) and the linear detection range is 0.5–250 μM.

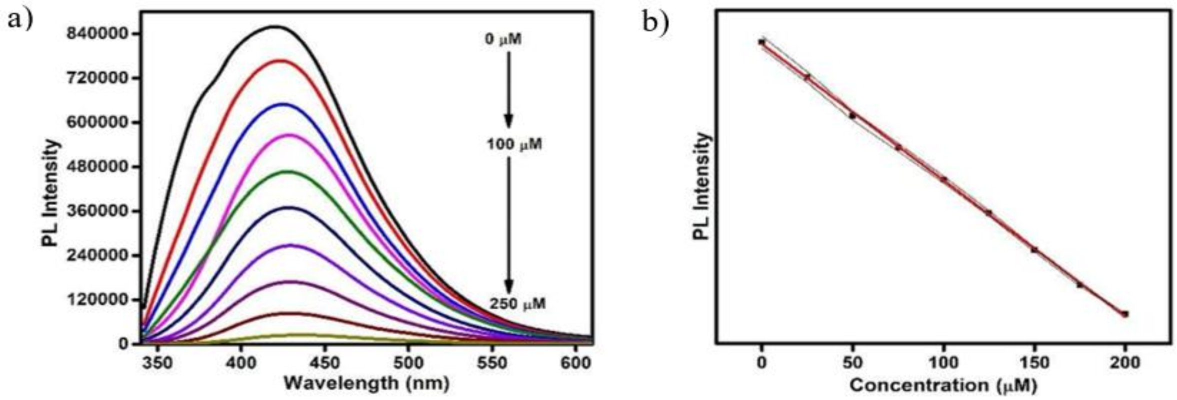

Figure 6. a) PL spectra of Cu-CDs in the presence of 2-NP and b) decrease in PL intensity with 2-NP concentration.

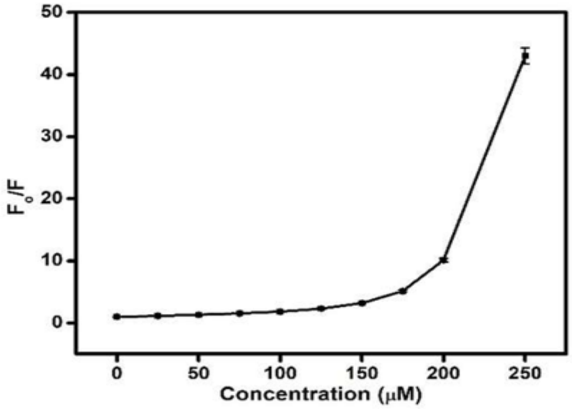

Figure 7. F_0/F versus analyte concentration for Cu-CDs.

3.5. Sensing mechanism for detection of 2-NP

The synthesized Cu-CDs have been used for the detection of 2-NP through photoluminescence spectroscopy to understand the sensing mechanism, the absorption, excitation and emission spectra of the synthesized nanomaterials was examined properly with UV-Vis. And photoluminescence spectroscopy. Further, UV-Vi's spectra of 2-NP also monitored to investigate the interaction or sensing mechanism. It has been observed that inner filter effect (IFE) may occur between synthesized material and 2-NP. As shown in Figure 8, the emission peak for Cu-CDs obtained at~ 423nm and absorbance peak for 2-NP observed at ~416 nm. The close proximity in emission wavelength of sensor and absorbance of analyte clearly indicates the IFE mechanism. These outcomes suggest that strong overlap between the emission of Cu-CDs and absorbance of 2-NP which is mainly due to the IFE.

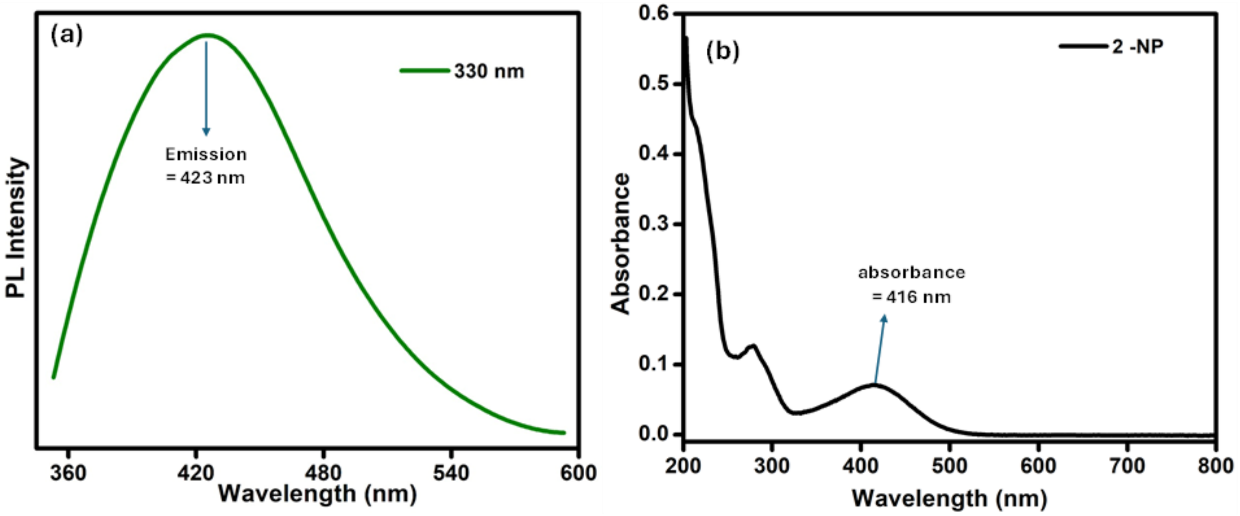

Figure 8. a) PL emissions of Cu-CDs and b) UV-vis. Spectra of 2-NP.

3.6. Selectivity detection for 2-NP

The method's selectivity is an important factor. Therefore, a variety of common chemicals (glucose, ascorbic acid,4-amino phenol, phenol, zinc, potassium, sodium, magnesium, picric acid, catechol) were examined, including various ions, amino acids, and substances with structures comparable to 2-NP. It showed maximum quenching effect in presence of 2-NP as compared to other analytes (Figure 9a). As stated in, when compared to other interfering species, the fluorescence of the Cu-CDs was considerably quenched upon the addition of 2-NP (Figure 9b). This means that other substances had minimal effect on the fluorescence of the Cu-CDs at the same concentration, suggesting that Cu-CDs have excellent selectivity for 2-NP assay.

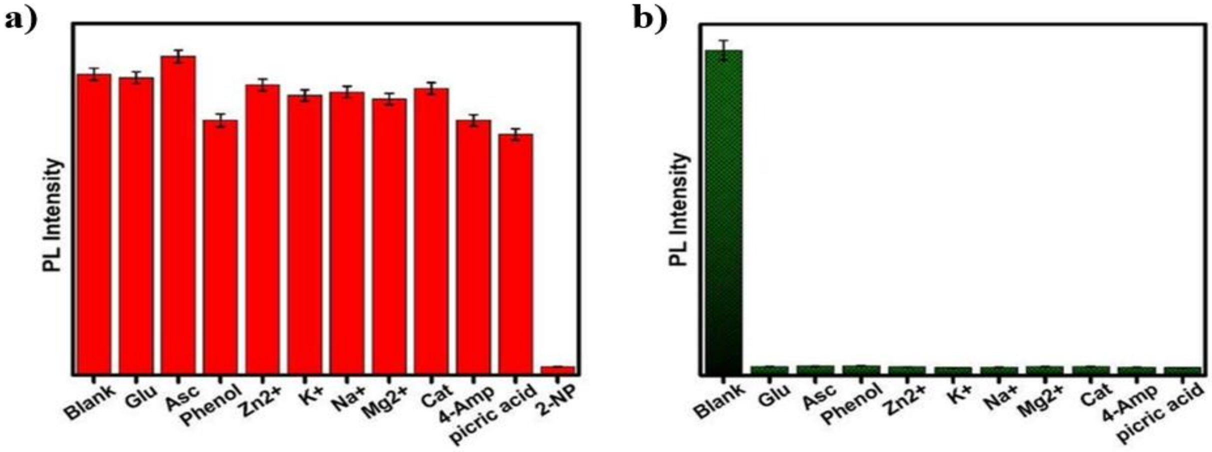

Figure 9. (a) 2-NP showed maximum quenching as compared to other analytes. (b) Effect of interfering ions on fluorescence quenching.

3.7. Application of the 2-NP detection method in water sample

The practical applicability of the Cu-CDs sensor was evaluated in real water samples using spike–recovery experiments. As shown in Table 1, satisfactory recoveries ranging from 97.5% to 101% were obtained for lake, rain, and tap water at different concentration levels (25–100 µM). These results demonstrate the reliability, accuracy, and potential of the developed system for real-sample analysis.

Table 1: Recovery (%) of analyte in real water samples at 25–100 µM using Cu-CDs.

Samples	25 µM	50 µM	100 µM
Lake water	99%	98%	97.5%
Rain Water	98%	99%	101%
Tap Water	99.5%	100%	99.7%

IV. CONCLUSIONS

In summary, we synthesized copper doped carbon dots using a simple and environment friendly one pot hydrothermal method. The successful doping of copper was confirmed through FTIR, EDX and fluorescence analysis. A high level of selectivity and a sensitivity sensing platform based on a Cu-CDs probe was built to detect 2-NP. The inner filter effect of 2-NP toward Cu-CDs was identified as a potential mechanism for fluorescence sensing. Significantly, the suggested sensing approach is effectively used to identify 2-NP in real water samples, indicating possible use in environmental monitoring.

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