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**Visible LED-based photo-redox properties of sulfur and nitrogen-doped carbon dots designed by solid-state synthesis**

Neha Kaushal, Amit L Sharma and Avishek Saha

Nitrogen-doped and sulfur, nitrogen co-doped carbon dots (CDs) were synthesized by solid-state, solvent-free, microwave synthesis technique at 250 °C. CDs were synthesized using citric acid as carbon precursor and urea or thiourea as nitrogen and sulfur precursor. The solid-state synthesis produced crystalline nitrogen-doped CDs (N-CDs) and nitrogen/sulfur co-doped CDs (S, N-CDs) with a broad absorption band in the visible region. The photoredox catalytic properties of as-prepared CDs were demonstrated by investigating the model photoreduction of methyl viologen using a 410 nm and 455 nm light-emitting diode (LED). The highest concentration of methyl viologen radical cation were monitored in the presence of N-CDs in comparison to S,N-CDs. The results indicate that nitrogen-doped graphitic CDs feature higher charge extraction for photoredox catalysis in comparison to nitrogen, sulfur co-doped CDs.

**Introduction**

Carbon dots (CDs) have attracted significant research interests since their discovery in 2004. Due to their unique properties such as high stability, tunable photoluminescence, chemical inertness, and biocompatibility, CDs have been investigated for a wide range of applications, including photocatalysis, sensing, electrocatalysis, and biomedical applications. In general, CDs consist of organic functional groups covalently attached with core semiconducting graphitic domain featuring photoluminescent properties from UV to near IR region of the solar spectrum. In this context, recent studies have suggested that the conventional bottom-up synthesis of carbon nanodots likely to produce molecular side-products including citrazinic acid and 4-hydroxy-1H-pyrrole[3,4-c]pyridine-1,3,6(2H,5H)-trione (HPPT). Although the presence of a complex variety of photoluminescent molecules in CDs is attractive for sensing applications; these fluorescent molecules are not desirable for photoredox catalytic process.

Thermal solid-phase synthesis could prevent the formation of these complex molecular by-products and facilitate the construction of a graphitic domain of CDs. For example, Reisner et al. have demonstrated that increasing the synthesis temperature will increase graphitized carbon core. They have further reported that graphitized carbon dots have better photocatalytic activity than amorphous carbon dots. In particular, microwave-assisted solvent-free synthesis method has several advantages, including short reaction time, reproducibility, and scalability. Apart from solid-state synthesis, doping of carbon nanodots with heteroatoms like nitrogen, sulfur etc. could be another strategy to improve the photoredox properties of CDs. Nitrogen is the most frequently used element due to its easy doping process and comparative atomic size. Core nitrogen doping can have a consequential impact on photocatalysis by tuning the absorption towards the visible spectrum and by facilitating the light induced charge transfer process. Also, doping of carbon dots with sulphur atoms can result in enhancement of electron donating efficiency of CDs and thus, elevating the efficiency of photocatalytic process. To this end, N and/or S doped CDs have demonstrated enhanced photocatalytic and photoredox properties.

To this end, very few studies are there on photoredox properties of doped and graphitized carbon dots synthesized by solid-phase, bottom-up synthesis. Cailotto et al. has performed comparative studies on photoinduced electron transfer from different amorphous and graphitized carbon nanodots, without introducing any heteroatoms. On the other hand, Prato et al. have investigated photocatalytic properties of nitrogen-doped carbon dots produced by liquid phase microwave synthesis. Still, the crystalline/amorphous nature of the core of carbon dots has not been studied. To enhance the photocatalytic properties of carbon dots, it is crucial to investigate the electron transfer properties of different heteroatoms doped, crystalline carbon dots.

In this work, we have synthesized graphitized N-doped and S, N-doped carbon dots at 250 °C following solid-phase microwave
synthesis route. Further, investigation of the photoinduced electron transfer properties has been done by performing the photoreduction of methyl violagen with 410 and 455 nm LED source. This study could provide an insight into the role of morphology, structure, and optical properties for the potential application of carbon dots for visible light-assisted photocatalytic applications.

Results and discussion

The content of functional groups present in the as-synthesized N-CDs and S,N-CDs determine their optical properties that are evaluated by UV-Vis and photoluminescence spectroscopy. Fig.1(a) shows the UV-Visible absorption spectra of the N-doped and S, N-doped carbon dots with different precursor weight ratios (citric acid: Urea (C/U) and citric acid: thiourea (C/T)) synthesized at 250 °C. N-CD-1 features a prominent peak at ca. 260 nm, which can be assigned to π→π* transition of C=C of sp² domain. Higher urea/thiourea content results in a small peak around 330 nm due to π→π* transition. Also, a broadened shoulder at 410 nm has been observed with its tail extending in the entire visible region might be due to the presence of surface molecular centers. This becomes more evident with the decline in weight ratio of precursors.

To assess the emission induced by π→π* and n→π* absorption transitions, the photoluminescence spectra was analysed by varying the excitation wavelength from 280 to 600 nm. Photoluminescent emission follows the stoke’s type emission, basically the photoluminescent emission wavelength is longer than the excitation wavelength. Also, the photoluminescent emission profile of different CDs in D.I. water shows the typical excitation wavelength-dependent behaviour by shifting the emission wavelength from 430 to 570 nm supporting information-Fig.S1. For S,N-CDs, emission peak maxima occurred at 370 and 430 nm corresponding to excitation wavelength of 340 nm for S,N-CD-1 and S,N-CD-2 respectively which is due to the introduction of sulphur atoms. The excitation dependent emission characteristics are consistent with previously reported S,N-CDs (Fig. 1)24, 25 Interestingly, 3D photoluminescence spectra of the N-CD-1 and N-CD-2 have shown a red shifted emission maxima at 530 nm along with 360 nm, (Fig.1(b) & Fig. S2(b)). This red-shifted emission in case of N-CDs is probably due to the interband transitions (π*→π type) as observed by previous reports based on solid-state synthesis.20, 26 With the increase in the nitrogen content in CDs at higher temperature, a low-lying intermediate state occurs in between the band. This results in narrowing the energy gap and leading to red-shifted interstate transition at 530 nm. Another possible reason for the green emission is likely due to the presence of molecular fluorophores (HPPT) attached to carbon cores.18, 27

Fig. 1 (a) UV-visible absorption spectra of N-CD-1, N-CD-2, S, N-CD-1, and S,N-CD-2 in aqueous dispersion. 3-D photoluminescence spectra of (b) N-CD-2 and (c) S,N-CD-2. (d) HR-TEM images (scale bar = 200 nm) of N-CD-2.

Fig. 2 XRD spectra of N-CD-1, N-CD-2, S, N-CD-1, and S,N-CD-2.
HR-TEM analysis was carried out to identify the morphology, crystallinity, and size distribution. TEM images indicate that the samples prepared by SPMA via microwave method are well dispersed (Fig. 1(d), S3 & S4) and the corresponding size distribution analysis in the histogram is shown in supporting information-Fig. S3 & S4. The value of average nanoparticle size and polydispersity measurement was performed by Gaussian fitting of size distribution. (See Table S1)

The crystalline phase of the prepared CDs samples was characterized by the powder XRD technique. All the samples show a characteristic peak around 27° (2θ), corresponding to d_{002} reflection arises due to interlayer stacking reflection. 4,18 Apart from this, the broad peak at around 13° (2θ) represents the d_{002} reflection indicates the amorphous characteristics which decreases with the increase in the urea/thiourea precursor ratio. Moreover, relatively higher intensity and sharper d_{002} peaks have been observed for higher amount of urea/thiourea precursor enriched carbon dots (N-CD-2, and S, N-CD-2) as compared to N-CD-1 and S, N-CD-1 respectively (Fig. 2).

Raman characterization is helpful to detect the intrinsic properties of sp2 carbon materials. Raman spectra of all the carbon dots, as shown in Fig. S5, reveal that two usual peaks at around 1359 (D peak) and 1580 cm⁻¹ (G peak). The peaks were observed with varying intensity values. Typically, the G band at 1580 cm⁻¹ is a characteristic of crystallised graphitic carbon which illustrates the E₂g mode for graphite that signifies the vibration of sp² bonded C-atoms present in the lattice. 28, 29 While the presence of D band at around 1360 cm⁻¹ is an evidence of amorphous characteristics which corresponds to the vibration of C-atom associated with dangling bonds in the termination plane of graphite with disordered nature. 30

FTIR Spectra of carbon dot samples were measured to obtain the structural information (Fig. 3). The bands in the range of (3400 to 2900 cm⁻¹) signify the presence of stretching vibrational modes of (O-H) and (N-H) functional groups. Peaks at ~1710 and 1638 cm⁻¹ peaks are associated with the carbonyl and amide functional groups. 31 These peaks are stronger in N-CDs as compared to S, N-CDs signifying the presence of more amino functional groups. The signal at 1384 cm⁻¹ is assigned to the stretching mode of C-N. 18, 31 In S,N-CDs, additional absorption features at 1080 and 735 cm⁻¹ are ascribed to the stretching vibration of C-S and C-S respectively. 21

Next, we turned to X-ray photoelectron spectroscopy (XPS) to investigate the elemental composition of carbon dots. In the full scan XPS spectrum of N-CD-2, as shown in Fig. 4(a), three characteristic peaks were found at 286.4, 400.6, and 532.4 eV, which corresponds to C (1s), N (1s), and O (1s), respectively. On the other hand, two additional peaks S (2s) = 229.7 eV and S (2p) = 164.7 eV, were observed for S, N-CD sample, confirming the presence of sulfur atoms. 21 For N-CDs, the deconvoluted high-resolution spectrum of C (1s), Fig. 4 (b), exhibits three characteristic peaks at 284.6 eV (sp² carbon, C=C), 285.7 eV (C=S or C-N), and 287.8 eV (C=O). 20, 32 The relatively high intensity of 284.6 eV peak indicates the presence of sp² enriched carbon atoms. 18 The peaks from high-resolution spectra of O (1s) were observed at 531.1 eV, 532.2 eV confirming the formation of C=O and C-OH/C-O-C bonds, respectively. This might be due to the presence of atmospheric oxygen and from the solvent as well. 33, 34 as shown in Fig. 4(c). The two peaks for the deconvoluted N 1s high resolution spectra at 398.5 and 399.9 eV, as shown in Fig. 4(d) can be assigned to pyridinic and pyrrolic nitrogen respectively. 25 From this, for S,N-CD sample, the deconvoluted spectra of S (2p) showed five peaks at 161.6 eV for (S-H) bonds, 163.6 eV for (C=S-C, where, n=1 or 2)/represents thiophene like structure, 164.9 eV for (C=S- bonds), 168.0 eV for (C=O) bonds, and 169.2 eV represents (C=SO₃) bonds formation in prepared samples (See Fig. S6). 36 The elemental composition analysis from XPS spectra reveals that N-CD contains (33.8, 36.8, and 29.4)% carbon, oxygen, and nitrogen, respectively, while for S,N-CD sample, the carbon, oxygen, and nitrogen contents are (29.4, 36.8, and 29.4)% respectively along with 30.9% sulfur.

Time-resolved photoluminescence of N-CDs and S,N-CDs were monitored using time-correlated single photon counting (TCSPC) to understand excited state properties. The
photoluminescence decay was monitored with pulsed excitation at 377 nm and emission at 450 nm. (See Fig. S7) Photoluminescence decays of all the CDs demonstrate biexponential decay (time constants are reported in supporting Table S2). To this end, nitrogen doped carbon dots (N-CD-2) reveals the most extended lifetime with time constants of 2.94 and 10.06 ns. Interestingly, sulphur doped carbon dots (S,N-CDs) features slightly shorter lifetime.

To understand the photoredox catalytic properties of N-CDs, and S,N-CDs, 410 and 455 nm wavelength LED was employed to study the conversion efficiency for photoreduction of methyl viologen cation (MV$^{2+}$) -0.45 V vs NHE in the presence of EDTA as sacrificial hole scavenger (See Fig. S8 & S9). Photo-redox of methyl viologen is a blue color radical cation has been extensively studied to understand the electron transfer properties of different nanomaterials, including carbon dots.$^{37-39}$ Positively charged MV$^{2+}$ likely to be attached on the surface of negatively charged carbon dots through electrostatic interaction, which facilitates electron transfer from CDs to MV$^{2+}$ under photo-excitation.$^{40}$ Conversion of MV$^{2+}$ to MV$^+$ is a reversible process indicated by colour change from colourless to blue, that is monitored by presence of 605 nm peak in UV-Vis absorption spectra. In this case, the Zeta potential values of all the samples are in the range of -34.3 to -17.96 mV (See Table S3 and Fig. S10). Among all the CDs, N-CD-2 demonstrates highest conversion (~3.5%) and (~7.0%) efficiency on illumination with 455nm and 410nm LED respectively, as shown in Fig. 5(a) & Fig. 5(b) respectively. It should be noted that, although previous reports on carbon dots-based photoreduction of methyl viologen have obtained higher efficiency, they have employed high-intensity UV-LED (365 nm) or solar simulators as light sources.$^{22,23}$ The overall trends for the conversion efficiency was observed as $(N$-CD-2)>S, N-CD-2> S,N-CD-1> N-CD-1 for 410 nm LED and (N-CD-2)>S, N-CD-2> N-CD-1> S, N-CD-1 for 455 nm LED. This illustrates that the presence of nitrogen plays a more important role than sulfur for the photoinduced electron properties of carbon dots. Also, higher conversion efficiency was observed in case of 410 nm LED as compared to illumination with 455nm LED because of higher absorption of all the prepared CDs in the near visible region as observed from UV-Vis absorption spectroscopy -Fig.1(a). The higher conversion efficiency can be explained by the long photoluminescence lifetime of N-CD-2. Furthermore, recent ultrafast spectroscopic studies have demonstrated that back electron transfer between the photoexcited holes in carbon dots and methyl viologen radical cation is slower in N-doped carbon dots in comparison with other heteroatom doped carbon dots.$^{41}$

Fig. 4 (a) Survey-scan XPS spectra of N-CD and S,N-CD samples. High-resolution XPS spectra of N-CD-2 sample: (b) C 1s, (c) O 1s, and (d) N 1s.
Materials and methods

Chemicals

Citric acid (Sigma-Aldrich, ≥99.5%), urea (TCI Chemicals, >99%), Thiourea (Sisco Research Laboratories Pvt. Ltd. AR, 99%), EDTA (Loba Chemie Laboratory Reagents and Fine Chemicals, 99%) and methyl viologen dichloride hydrate (Aldrich, 98%) were used as purchased and without further modification.

Synthesis of CDs

Carbon dots were synthesized using the solid-state microwave bottom-up synthesis approach. Briefly, 0.5 g of citric acid and 0.5 g of urea was mixed uniformly with mortar and pestle. The weight ratio of citric acid and urea was varied for N-CD-1 and N-CD-2. 0.5 gm of the resultant mixture was taken in a glass microwave vial with an inner volume of 30 ml and sealed with a PTFE-lined cap. The mixture was heated at 250 °C for 10 min with a microwave reactor (Anton-Paar, Monowave 200). During the process of microwave heating, stirring was done with a magnetic stir bar present in the reaction mixture at 600 rpm. After 10 minutes of reaction, the system was cooled down to 55 °C. The inner temperature and pressure conditions were managed and maintained through an internal IR thermometer and pressure sensor, respectively. Then, the product was extracted from a vial and dissolved in DI water to form a homogeneous aqueous dispersion through ultra-sonication process and purification was done via dialysis using Spectra/MWCO:500-1000D for three consecutive days. Followed by dialysis, the final product was obtained by the freeze drying process. The same procedure was employed for the synthesis of S, N-CDs samples by replacing urea with thiourea as a source of nitrogen and sulfur. All the samples are denoted by their doped atoms as, nitrogen doped carbon dots and nitrogen and sulphur co-doped carbon dots with weight ratio (1:1 and 1:3) represented as N-CD-1, N-CD-2, S,N-CD-1 and S,N-CD-2 respectively.

Characterization

UV/Vis spectra of the aqueous dispersion of CDs were obtained by Cary 4000 UV-Vis Double Beam Spectrophotometer. The crystalline nature of the CDs was characterized with Model D8 Advance, Bruker AXS X-ray diffractometer using Cu Kα radiation. Fourier transform infrared spectra were obtained using Nicolet iS10 FTIR spectrophotometer using IR grade KBr powder. The photoluminescence spectra of the aqueous dispersion of CDs were measured with Cary Eclipse Fluorescence spectrophotometer. High-resolution transmission electron microscopy images were recorded on 200 kV JEM-2100 transmission electron microscope. The samples were prepared by drop-casting onto carbon-coated copper grid, followed by drying at room temperature. X-ray Photoelectron Spectroscopy (XPS) analysis was performed with Thermo Scientific XPS, Model MULTILAB 2000 base system, having Twin anode Mg/Kα (300-400 W), X-Ray Source electron gun having spot size of < 50 mm diameter. PL lifetime decays of an aqueous dispersion of carbon dots were monitored using 377 nm picosecond pulsed diode lasers (5 mW) excitation and time-resolved spectrophotometer.

Conclusions

In this work, solid-state microwave synthesis of heteroatom-doped carbon dots is demonstrated. The photoredox properties of doped carbon dots were studied by investigating model photoreduction of methyl viologen. The graphic nature of the carbon dots was more prominent in urea/thiourea precursor enriched carbon dots. To this end, nitrogen-doped carbon dots with higher urea precursor content demonstrates the highest photoconversion efficiency of methyl viologen in the presence of 410 and 455 nm wavelength LEDs. The results herein reported show that nitrogen-doped carbon dots are better electron transfer agents than sulfur, nitrogen-co-doped carbon dots under visible light irradiation.
Photo-redox catalytic studies

The photo-redox properties of carbon dots were studied by performing Photo-reduction experiments of Methyl viologen to MV⁺ Radical, using 410 nm and 455 nm LEDs (Holmarc Opto-Mechatronics Pvt. Ltd., Model-HO-HBL-3M). Briefly, 50 μg/mL of an aqueous solution of CDs were prepared in ethylene diamine tetracetic acid (EDTA, 0.1 M, pH 6) in addition of MV⁺ (1 mM). Then, ~3 mL of the solutions was irradiated with a visible Light-emitting diode lamp (LED, wavelength = 455 nm, irradiance of 33 mW/cm² and wavelength = 410 nm, irradiance of 26 mW/cm²), after purging with Argon gas for 10 minutes. Furthermore, the generation of MV⁺ radical cation was monitored (for 60 min) by the emergence of a new peak around 605 nm (ε = 13700 M⁻¹ cm⁻¹) using a UV-visible spectrophotometer. The monochromatic nature of LED light sources was supported with absorption spectra taken via absorption spectrometer (Avantes Starline, Avaspec-3648). (Fig. S13 & S14)

Author Contributions

The manuscript was written through the contributions of all authors. All authors have approved the final version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

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