Biocompatible Carbon Quantum Dots Derived from Sugarcane Industrial Wastes for Effective Nonlinear Optical Behavior and Antimicrobial Activity Applications

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ABSTRACT: In this work, the green synthesis of highly fluorescent carbon quantum dots (CQDs) with an efficient quantum yield of 17.98% using sugarcane bagasse pulp as the precursor was conducted by a hydrothermal technique. The high-resolution transmission electron microscopy analysis revealed that the CQDs were competently monodispersed with the particle size ranging between 0.75 and 2.75 nm. The structural properties of CQDs were investigated using X-ray diffraction, Fourier transform infrared, and X-ray photoelectron spectroscopy analyses. The UV–visible spectrum showed two absorption peaks due to the aromatic C=C transitions of π−π* and C==O transitions of n−π*. The fluorescence spectrum of CQDs displayed a strong blue emission. However, the first-ever of its kind, sugarcane industrial solid waste carbon quantum dots caused significant orders to obey the enhancement of the third-order nonlinearity (χ(3)) when compared with other carbon dots (CDs). The calculated nonlinear optical (NLO) parameters such as n2, β, and χ(3) were 1.012 × 10−8 cm/W, 2.513 × 10−14, and 3.939 × 10−7 esu, respectively. The figures of merit were evaluated to be W = 6.6661 and T = 0.0132, which greatly fulfilled the optical switching conditions. Besides, the antibacterial activities of CQDs were screened against aquatic Gram-positive (Benthesicymus cereus and Staphylococcus aureus) and Gram-negative (Pseudomonas aeruginosa, Vibrio cholerae, and Escherichia coli) microbial organisms. Our findings, however, indicate that synergistic sugarcane industrial waste CQDs are promising materials for the functioning of NLO devices, bioimaging, and pharmaceutical applications.

1. INTRODUCTION

Over the past decade, developing countries have consistently encountered the problem of pollution of soil and earth as a major issue, as dumping sugarcane bagasse waste creates a major challenge for yielding numerous million tons of sugarcane molasses every year.1–5 Inexpensive and environmentally friendly green chemistry ideas have now led researchers to synthesize CDs from natural solid waste resources, such as tamarind,6 pomelo peel,7 watermelon peel,8 pineapple peel,9 lemon peel,10 orange peel,11 papaya juice,12 banana juice,13 soy milk,14 potato,15 coffee grounds,16 and cabbage,17 which are used as strong acid precursors. In this perspective, the practice of waste materials to synthesize quantum dots (QDs) to enhance the third-order differential nonlinear properties, which may attract significant attention because of their host applications in energy storage,18 conversion of energy, optical telecommunication, and bioscanning.19 The foremost, prior demonstration of all optical transformation was forced by the performance of materials in the relation between the “prime factors” which describe (i) the nonlinear phase to be shift achievable over a single photon, which is W > 1 or a multiphoton is T < 1; (ii) however, the absorption coefficient is an essential requirement, which is to be satisfied for the primary application of optical switching.20

Recently, infectious diseases triggered by microorganisms, such as viruses, fungi, parasites, or others, have impacted public health concerns, and there is an urgent need to detect, treat, and control these infectious diseases.21 Infections caused by these microorganisms are relatively more challenging as these infections have developed drug-resistant bacteria and viruses, which could be problematic for the current drug treatment.22 Antimicrobial activity applications

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health in many countries and are a leading cause of death globally. Travlou et al.21 stated that nitrogen-containing carbon promotes the creation of active oxygen species, which is correlated with their electron-donating properties. The antimicrobial capacity of CQDs, therefore, has only recently been discovered.22–24 CQDs directed against Gram +ve and Gram −ve microbes have been reported, in which bacterial targeting was established by electrostatic interaction between the anionic microbial membrane and cationic residues on the surface of the C-dots.25,26 CQDs may be used as an important substitute for conventional antibiotic drugs in antibacterial testing.

However, in the present report, we have explored the nonlinear optical properties of CQDs using industrial waste (sugarcane bagasse pulp) as a carbon source synthesized by a hydrothermal method. The synthesized carbon quantum dots were investigated using different techniques, such as X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), high-resolution transmission electron microscopy (HRTEM), UV–visible absorption, and fluorescence quantum yield measurements. The NLO behavior was characterized by the Z-scan technique using a continuous-wave laser (532 nm, 100 mW). Also, antimicrobial activities were tested against selected microorganisms.

2. EXPERIMENTAL PROCEDURES

2.1. Materials and Methods. Newly harvested sugarcane bagasse pulp was collected from sugarcane industrial waste and cleansed with deionized water. The pulp was dehydrated in sunlight for 3 days before being ignited at 70 °C in the air atmosphere to form a carbon matrix. Citric acid (CA) [C6H8O7] and aqueous ammonia were bought from E-Merck (99.99%), and all of the chemicals were of systematic grade.

Industrial waste (sugarcane bagasse pulp) CQDs were prepared using a hydrothermal approach. Briefly, 2 g of yielded carbon (sugarcane bagasse pulp) and 2 g of CA were homogeneously mixed with 25 mL of double-distilled water, and aqueous ammonia was added to the precursor to set the pH to 7. The isolated precursor was completely shifted to the autoclave at a stable temperature of 200 °C for 6 h. The reactive mixture solution was ultrasonicated for 1 h and centrifuged for 60 min at 5000 rpm to remove superior undissolved particles. Eventually, the black solid precipitate was removed and the supernant liquid was stored for further characterization and use.

2.2. Characterization of CQDs. Powder XRD measurements of the CQDs using Cu Kα radiation (1.5404 Å) were conducted on a Bruker AXS D8 Advance diffractometer at a scanning speed of 0.1 min−1 with 20 ranging from 10 to 80°. HRTEM images were collected using a JEOL/JEM2100 microscope (operated at 200 kV). The FTIR spectrum was recorded with an FTIR spectrometer (PerkinElmer spectrometer) in the spectral range of 4000–400 cm−1 at ambient temperature. A linear optical absorption spectrum of CQDs was recorded using a Shimadzu spectrophotometer (UV-1800), and the sample was immersed in water. Fluorescence studies were carried out with a single-beam PerkinElmer fluorescence spectrometer (model LS45) at ambient temperature (RT). Third-order nonlinearity of CQDs was scrutinized using the Z-scan method (Holmarc Z-scan, model HO-ED-LOE-03).

2.3. Z-scan Analysis. The higher-order NLO parameters were examined by the Z-scan method. In this technique, the sample was focused using the focal length of a convex lens of 103 mm, the optical path length of 675 mm, the aperture radius (r0) of 1.25 mm, and the beam radius (ω0) of 3.5 mm. Initially, the prepared material was dispersed in deionized water. The scattered particles were separated in a 1 mm cuvette and placed on a conversion point, which was moved from the positive to the negative direction in the Z-axis along the propagation route of the laser beam. For the accuracy of each movement, the translation of the sample holder can be monitored by a computer. The associated transmitted intensity of the sample was recorded by a detector.

2.4. Antimicrobial Activity. The antibacterial activities of the synthesized fluorescence CQDs against Staphylococcus aureus, Bacillus cereus, Escherichia coli, Vibrio cholera, and Pseudomonas aeruginosa were evaluated using the well diffusion process. A The petri dish and the sample were sterilized at 120°C for 30 minutes prior to the antibacterial testing. The newly prepared bacterial inoculums were swabbed throughout the surface of the nutrient agar medium (growth medium) using a sterilized cotton swab to maintain uniform distribution of the bacteria across the plate surface. First, the stock solution of CQDs was mixed with sterile distilled water. Then, 0.01 mg/mL carbon quantum dots were loaded into the well and incubated for 24 h at 37 °C. Successfully, the inhibition zone (mm) formed in the Petri dish was observed.

3. RESULTS AND DISCUSSION

3.1. HRTEM Analyses. Figure 1a demonstrates that the ultrafine particles are more uniform, which are spherical quantum dots with a mean particle size of 1.7 ± 0.2 nm. The success rate of the green synthesis hydrothermal approach in producing carbon dots was confirmed by the HRTEM images, which are displayed in Figure 1b. The lattice fringes of CQDs with an interplanar distance d of ~0.323 nm are associated with graphitic carbon.27 In addition, the fragment size distributions of CQDs were analyzed to obtain the stabilized particle size and are depicted in Figure 1c. It was found from the CQD distribution curve that particles are distributed randomly with an average particle size varying from 0.75 to 2.27 nm and that the mean CQDs were of 1.7 ± 0.2 nm. The XRD pattern of synthesized CQDs is displayed in Figure 1d, which shows a broad peak
position at $2\theta = 21-28^\circ$. This broad peak is associated with the (0 0 2) plane and suggests the disordered pattern of carbon dots, due to the addition of N- and O-containing groups.28,29 These observations are in good agreement with those previously reported for CQDs.30–33

3.2. FTIR Analysis. Figure 2 indicates the FTIR spectrum of the as-prepared CQDs. The absorption peak at 3407 cm$^{-1}$ is related to O$\equiv$H/N$\equiv$H and a sharp peak at 2925 cm$^{-1}$ corresponds to the methyl or methylene (C$\equiv$H) groups.34 The peaks at 1593 and 1403 cm$^{-1}$ correspond to the distinctive absorption peaks of C$\equiv$O and COO$^{-}$ functional groups of CQDs, respectively.35 The peaks in the region 1260–1240 cm$^{-1}$ are attributed to the C$\equiv$N stretching, and the peak at 1033 cm$^{-1}$ is allocated to the C$\equiv$O/S$\equiv$O stretching vibration.36 The presence of the hydroxyl group (O$\equiv$H) plays a vital role in strengthening the antibacterial effect of the as-prepared CQDs.37–39

3.3. X-ray Photoelectron Spectroscopic (XPS) Measurement. XPS is used to examine the components of surface groups and the structure of as-prepared CQDs. Moreover, the three major points at 284.63, 398.85, and 530.07 eV, as shown in Figure 3a, can be ascribed to C 1s, N 1s, and O 1s, respectively, suggesting the efficient formation of CQDs. The C 1s spectra in Figure 3b show three peaks at 284.80, 283.8, and 287.10 eV, which are assigned to C$\equiv$C/C$\equiv$C, C$\equiv$OH/C$\equiv$O$\equiv$C, and C$\equiv$O/C$\equiv$N, respectively. As shown in Figure 3c, XPS spectra of N 1s exhibit two peaks at 399 and 397 eV corresponding to C$\equiv$N$\equiv$C and C$\equiv$N groups, respectively. The distribution of O 1s in Figure 3d indicating two peaks at 532.18 and 530.44 eV attributed to the presence of C$\equiv$OH/C$\equiv$O$\equiv$C and C$\equiv$O bonds, respectively, and the graphite structure of the prepared CQDs corresponding to the peak at 284.63 eV referring obviously to C 1s are consistent with those from FTIR analysis.40 XPS demonstrated that the surface of nitrogen-containing functionalized CQDs is properly connected with hydroxyl and carbonyl functional groups.41,42

3.4. Optical Studies. The UV$\rightarrow$visible spectrum of as-prepared CQDs in aqueous solution is given in Figure 4. The spectrum displays two corresponding peaks at 233 and 332 nm in a supernatant solution of carbon dots. The absorption peak centered at 233 nm can be ascribed to the $\pi$$\rightarrow$$\pi^*$ transitions of the aromatic C$\equiv$C and the peak at 332 nm is involved in the n$\rightarrow$$\pi^*$ transition of C$\equiv$O or the C$\equiv$OH bond of the CQDs.43–45 The diluted CQDs show an intense sky blue color upon illumination by a UV-light source (365 nm), which is
shown in the inset of Figure 4. The following equation is used to determine the linear optical absorption coefficient ($\alpha$)

$$\alpha = \frac{2.303 \times A}{t}$$  \hspace{1cm} (1)

where $A$ is the absorption and $t$ is the sample thickness. The transmittance ($T$) is given by

$$T = \frac{(1 - R)^2 \exp(-\alpha t)}{(1 - R)^2 \exp(-2\alpha t)}$$  \hspace{1cm} (2)

The reflectance ($R$) and linear refractive index ($n_0$) in terms of the absorption coefficient ($\alpha$) can be determined using the following equation:

$$R = \frac{\exp(-\alpha t) \pm \sqrt{\exp(-\alpha t) T - \exp(-3\alpha t) T + \exp(-2\alpha t) T^2}}{\exp(-\alpha t) + \exp(-2\alpha t) T}$$  \hspace{1cm} (3)

The values of transmittance and $R$ can be used to measure the $n_0$ of prepared carbon quantum dots from the following equation:

$$n_0 = -(R + 1) \pm \frac{\sqrt{2}}{(R - 1)}$$  \hspace{1cm} (4)

From the recorded absorption spectrum, $n_0$ was calculated, and a graph is drawn between $n_0$ and $\lambda_i$ as presented in Figure 5. The calculated linear refractive index ($n_0$) of the prepared fluorescent CQDs was found to be 1.234 at a wavelength of 532 nm, and it is used to evaluate the higher-order NLO susceptibility ($\chi^{(3)}$) of the carbon quantum dots (CQDs).

**3.5. Fluorescence Analysis.** The fluorescence spectra are reported for the diluted sample at the wavelength of excitation ($\lambda_{ex} = 330$ nm). Peng et al.\textsuperscript{18} suggested that a higher quantum yield was obtained by surface states of carbon quantum dots.

The citric acid solvent plays a significant role when it is added to the CDs, and it improves their fluorescence nature. The findings are more similar to the results obtained with the polystyrene foam leftover soot CQDs.\textsuperscript{14} In this article, fluorescence spectra of CQDs for different concentrations (0.02–1 mL) have been investigated and are exhibited in Figure 6. This fluorescence emission intensity gradually enhances as the concentration of the solution increases, which is further evidence for the enhancement of emission properties. The CQDs exhibited a sky blue color using a long-wave UV-light source at 365 nm, as displayed in Figure 4. We found this diverse range of fluorescence emissions to be immensely beneficial and efficient compared with green fluorescence CQDs.\textsuperscript{52} The result shows that the carbon quantum dots could be a better replacement for conventional coloring applications for fluorescent labeling.\textsuperscript{53}

**3.6. Quantum Yield (QY) Measurement.** The QY of the as-prepared CQDs was measured by diluting the sample in deionized water. The solution was taken from a 10 mm quartz cuvette to measure UV-Vis and fluorescence spectra. Quinine sulfate of 0.1 M [H$_2$SO$_4$] was used as a standard reference, for which the QY is 0.54.\textsuperscript{54} The following equation was used to evaluate the QY

$$QY = \frac{\eta^2}{\eta_{ref}} \frac{\lambda}{\lambda_{ref}} \frac{A}{A_{ref}}$$  \hspace{1cm} (5)

where $QY_{ref}$ is the QY of the reference material (0.54 for quinine sulfate), $\eta$ is the refractive index of the solvent, $\eta_{ref}$ is the refractive index of quinine sulfate, $A$ is the absorption at the given wavelength, and $I$ is the integrated fluorescence emission intensity. The fluorescence QY of the carbon quantum dots at $\lambda_{ex} = 330$ nm was calculated to be 17.98%, and the integrated luminescence intensity of carbon quantum dots was compared to that of standard quinine sulfate.\textsuperscript{55}

**3.7. Z-scan Analysis.** The third-order nonlinear optical parameters of carbon quantum dots were examined using the Z-scan method.\textsuperscript{56} This technique has indeed been established for a diverse number of uses, such as optical switching, optical limiting, etc. The material is caused by the laser pulse when it either focuses or defocuses, which depends on the nonlinearity of the materials. Nonlinear absorption occurs in the ground state ($S_0$) and then in the first and the next larger singlet state ($S_1$, S$_2$). The T$_1$ and T$_2$ energy states describe the lowest and the highest triplet transformation based on the pulse size, wavelength, and pump intensity. The system (S$_1$-S$_2$and T$_1$-T$_2$) are classified as excited-state absorption (ESA), and is related to as reverse saturable absorption (RSA) because its cross-sections are greater than for the ground state.\textsuperscript{57–59} The measurement begins from −Z where the transmittance is relatively constant ($T = 1$). The normalized condition ($T = 1$) of Z-scan is exhibited in Figure 7a. The sample is shifted in the direction of emphasis ($Z = 0$) and then reaches +Z. If the sample has a positive nonlinearity ($n_2 > 0$), then the transmittance graph has a valley first and then a peak, as seen in Figure 7b. If the sample has $n_2 < 0$, the graph is precisely the opposite (a peak followed by a valley), as seen in
near the focus ($Z = 0$). If the intensity of the transmission peak is high, it indicates saturable absorption (SA), and, on the other hand, if the intensity of the transmission is less (valley), it is called reverse saturation absorption (RSA). To obtain the NLR index of the carbon quantum dots, the disparity between the standardized transmission intensity peak and valley ($\Delta T_p - v$) in the curve of ratio of closed and open aperture standardized $Z$-scan patterns is calculated, as displayed in Figure 8b. The actual and imaginary parts of the NLO susceptibility ($\chi^{(3)}$) values of the CQDs are calculated using the following equations \(^{63,64}\)

$$
\text{Re} \chi^{(3)}(\text{esu}) = \frac{10^{-4} \epsilon_0 \gamma^2 n_0^2 n_2}{\pi} \left( \text{cm}^2/\text{W} \right) \quad (6)
$$

$$
\text{Im} \chi^{(3)}(\text{esu}) = \frac{10^{-2} \epsilon_0 \gamma^2 n_0^2 \beta}{4\pi^2} \left( \text{cm}/\text{W} \right) \quad (7)
$$

Here, $\epsilon_0$ is the permittivity of free space ($8.854 \times 10^{-12} \text{ F/m}$), $c$ is the velocity of light in vacuum, and $n_0$ is the linear refractive index of the carbon quantum dots. The third-order nonlinear susceptibility ($\chi^{(3)}$) of the carbon quantum dots could be evaluated by the equation

$$
\chi^{(3)} = \sqrt{\text{Re} \chi^{(3)} + (\text{Im} \chi^{(3)})^2} \quad (8)
$$

The values calculated for the NLO parameters $n_2$, $\beta$, and $\chi^{(3)}$ are summarized in Table 1. The NLO susceptibility is found to be higher than those of several other nonlinear optical materials, as seen in Table 2.\(^{65-69}\) Therefore, synthetic carbon dots are a good fit for optical switches if the conditions $W > 1$ and $T < 1$ are fulfilled.\(^{70}\)

$$
W = \frac{n_2 I}{a \lambda} \quad (9)
$$

### Table 1. Third-Order NLO Measurement Values of Prepared CQDs

| third-order NLO parameters | values |
|-----------------------------|--------|
| laser beam wavelength ($\lambda$) | 532 nm |
| linear absorption coefficient ($\alpha$) | 9.902 |
| linear refractive index ($n_0$) | 1.2348 |
| nonlinear absorption coefficient ($\beta$) | $2.513 \times 10^{-4} \text{ cm/W}$ |
| nonlinear refractive index ($n_2$) | $2.012 \times 10^{-8} \text{ cm}^2/\text{W}$ |
| real part of the third-order susceptibility ($\text{Re} \chi^{(3)}$) | $3.917 \times 10^{-7} \text{ esu}$ |
| imaginary part of the third-order susceptibility ($\text{Im} \chi^{(3)}$) | $4.120 \times 10^{-8} \text{ esu}$ |
| third-order nonlinear optical susceptibility ($\chi^{(3)}$) | $3.939 \times 10^{-7} \text{ esu}$ |

### Table 2. Comparison of Third-Order NLO Susceptibility ($\chi^{(3)}$) Values for Other Nonlinear Optical Materials and CQDs

| materials | method                | ($\chi^{(3)}$) (esu) | ref |
|-----------|-----------------------|-----------------------|-----|
| sugarcane waste CQDs | hydrothermal          | $3.939 \times 10^{-7}$ | present work |
| orange waste CQDs | hydrothermal          | $2.774 \times 10^{-7}$ | 65  |
| N-CDs      | one-step wet chemical | $12.5 \times 10^{-12}$ | 66  |
| boron-doped C-dots | microwave heating     | $5.0 \times 10^{-15}$  | 67  |
| carbon dots (CDs) | pyrolysis             | $11.3 \times 10^{-13}$ | 68  |
| CDs        | ultrasonication       | $4.6 \times 10^{-13}$  | 69  |
where $I$ is the irradiance of the laser beam. The figures of merit were evaluated to be $W = 6.6661$ and $T = 0.0132$, which significantly fulfilled the condition. Hence, the synthesized sugarcane industrial waste CQDs are suitable for all optical switching and power conversion device applications.

3.8. Antibacterial Activity. The antibacterial assays of CQDs against Gram $+$ve and Gram $-$ve bacteria were evaluated. CQDs have been used to suppress the growth of bacteria (Figure 10). The ZOIs obtained for the microorganisms are presented in Table 3. The antibacterial function of CQDs is demonstrated in Figure 11. The antibacterial behavior may be attributed to various functional groups present in CQDs that can interfere with cellular enzyme functions and inhibit cellular proliferation. The large $\pi$-conjugated carbon quantum dot system easily attached through electron transfer to the bacterial cell wall. $^{71,72}$ The antibacterial mechanism of the CQDs has been widely speculated, as per the literature, to be based on electrostatic interactions, ROS, or light irradiation. ROS generation has essentially important antibacterial activity. $^{73-77}$ The hydroxyl radicals and nitrogen groups are confirmed from FTIR and XPS studies. CQDs include nitrogen elements that possess positive charges that link them with negatively charged microbes, and CQDs penetrate into the cell membrane and ultimately result in the death of microorganisms. Several studies have reported nitrogen-containing CQDs with assured antimicrobial activity against Gram $+$ve and Gram $-$ve microorganisms. Yadav et al. $^{78}$ have reported CNQDs that could effectively produce superoxide and hydroxyl radicals and interact with Staphylococcus aureus and E. coli pathogens. Travlou et al. $^{21}$ have developed N-doped CQDs with specific antimicrobial activity against E. coli and B. subtilis. Interestingly, in the present study, the CQDs show more inhibition toward Bacillus cereus, Staphylococcus aureus, Pseudomonas aeruginosa, Vibrio cholera, and Escherichia coli, and their antibacterial activity is compared with those of other CQDs (Table 4). $^{79-86}$ Therefore, the synthesized CQDs can be used for pharmaceutical applications.

4. CONCLUSIONS

Industrial waste (sugarcane bagasse pulp) CQDs were synthesized by the hydrothermal method with a 17.98% quantum yield. The average particle size of the CQDs was 1.7 $\pm$ 0.2 nm with a spherical shape, which was determined by
for the traditional dyes that are used for biosensing applications. A high nonlinear absorption (third-order NLO susceptibility) and biomedical applications. The CQDs exhibited optical switching condition, proving that CQDs can be a good material for optical switching applications. The CQDs exhibited good antibacterial activities against tested bacterial strains. The above results suggest that biocompatible and quickly prepared CQDs are a suitable material for photonic devices, bioimaging, and biomedical applications.

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**Notes**

The authors declare no competing financial interest.

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