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Sustainable Green Synthesis Of GO/C-Dots Composites From Tobacco Stem Waste Via Liquid Sonication Exfoliation And Oven Methods

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Abstract: This study investigated the utilization of tobacco stem waste as a green precursor for the synthesis of GO, C-Dots, and GO/C-Dots composites with 1:1 and 1:3 ratios through oven drying methods and liquid sonication exfoliation (LSE). Characterization techniques, including UV-Vis-NIR spectroscopy, XRD, FTIR spectroscopy, SEM, PL, and TRPL, were used to evaluate the synthesized materials. The GO/C-Dots composites exhibited an energy band that classifies them as organic semiconductors. XRD analysis showed a change in crystallinity and indicated an increase in C-Dots content. FTIR analysis showed a shift in functional groups at 1:1 to 1:3 ratios that showed an inverse trend. SEM analysis showed that the addition of C-Dots significantly affected the surface morphology and elemental composition of GO. TRPL test showed consistent results with the trend of PL intensity where C-Dots had the highest value and GO the lowest. This finding highlights the potential of GO/C-Dots composites as sustainable materials, with further exploration needed to assess their applications in various fields such as optoelectronics, catalysis, and environmental science.

Keywords: Carbon nanodots; Graphene oxide; Liquid sonication exfoliation; Oven heating; Waste tobacco sticks.

Introduction

Tobacco plants are widely cultivated in specific regions with suitable land conditions. In Indonesia, tobacco is predominantly grown in areas like Wonosobo Regency, Central Java (Ditjenbun, 2016), of which almost 35% of land is planted with tobacco and in 2018, produced 1.980.15 tons of tobacco with one notable production area being Candiyasan Village, Kertek District (Data Pertanian Provinsi Jawa Tengah, 2019). While tobacco leaves are mainly utilized for cigarette production, its stem waste remains underutilized. Currently, tobacco stems are processed into biopesticides (Nugraha & Agustiningsih, 2015) and liquid smoke to extend the shelf life of fish meat (Mu'tamar et al., 2018). Therefore, it can be said that its utilization is not optimal and efforts need to be made to minimize the amount of tobacco waste dumped into the environment.

This study aims to synthesize GO and C-Dots from tobacco stem waste using the Liquid Sonication Exfoliation (LSE) method combined with an oven process. To explore the potential of these nanomaterials, GO and C-Dots will be mixed in a certain ratio, and their characteristics will be analyzed using various characterization techniques. Through this approach, this study not only reduces agricultural waste but also produces high-value nanomaterials, which contribute to sustainable solutions in resource utilization and

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environmental conservation (Giwa et al., 2019; Sun et al., 2021).

GO is a derivative of graphene with a similar structure but functionalized with oxygen and hydrogen groups. Its exceptional properties include high electron mobility (200.000 cm^2/Vs), electrical conductivity (0.96 x 10^6/ohm), thermal conductivity (5000 W/mK), and tensile strength exceeding that of steel (Brownson & Banks, 2016; Ding et al., 2016). GO has been utilized in applications such as batteries, capacitors, and fuel cells, as well as biodegradable paints with conductive properties (Sartini, 2019; Tian et al., 2021). Carbon Dots (C-Dots) are spherical nanomaterials under 10 nm in size, characterized by an amorphous structure with an sp^2 carbon framework. These nanodots have gained significant interest for applications in optoelectronics, bioimaging, energy harvesting, and sensing (Dager et al., 2020).

Tobacco stem waste holds potential as a source of biomass for advanced technological applications. One promising approach is the conversion of this waste into high-value materials like Graphene Oxide (GO) and Carbon Dots (C-Dots). These nanomaterials are biocompatible and versatile, with applications spanning electronics to biomedicine (Wang & Hu, 2014). Studies have shown that tobacco stems are rich in cellulose, hemicellulose, and lignin, providing the carbon chain structures essential for GO and C-Dots synthesis (Bragatto, 2016; Handayani & Amrullah, 2018; Liu et al., 2015). This research seeks to utilize tobacco stem waste as a sustainable raw material for producing these nanomaterials.

Method

This research was conducted in several stages, as outline below.

Sample Preparation

The tobacco stem sheets were cut into small pieces weighing 40 grams and placed on a tray. The oven was preheated for 30 minutes at a temperature of 250˚C. The dried material was ground into a fine powder using a mortar and subsequently filtered to yield 25 grams of fine black powder.

Synthesis of C-dots

Graphite powder was heated again for 1 hour at a temperature of 200˚C. A solution of 250 ml distilled water and 2 grams of graphite was prepared, mixed using a magnetic stirrer at 8 rpm for 10 minutes. The resultant mixture was filtered using Whatman paper, yielding a C-dots solution.

Synthesis of GO

A solution containing 2 grams of graphite was mixed with 245 ml of distilled water (solution 1) and 2.5 grams of surfactant was mixed with 5 ml of distilled water (solution 2). Both solutions were combined and sonicated at a frequency of 42.13 kHz for 1.5 hours. The sonicated GO solution was filtered with Whatman paper until a powder is obtained, while the remaining solution was used for the synthesis of a mixture with C-Dots.

GO/C-dots Mixture

Two mixtures were prepared by varying the ratios of GO to graphite: 1:1 Ratio (240 ml of GO solution mixed with 2 grams of graphite) and 1:3 Ratio (240 ml of GO solution mixed with 6 grams of graphite). Each mixture was homogenized using a magnetic stirrer. From each mixture, 100 ml was reserved for UV-VIS-NIR spectrophotometric analysis. The remaining sample was subjected to furnace treatment at 1000˚C for 2 hours. The furnace product was allocated for characterization as follows: 0.2 grams for FTIR, 0.5 grams for XRD, and 0.4 grams for SEM analysis. Data from these analyses were processed using ORIGIN 2021 software.

Data Analysis

The absorbance spectra of GO, C-dots, and GO/C-Dots samples from 190-1200 nm were processed using the Tauc Plot method to obtain the energy gap. The value obtained compared to the value of organic semiconductors. After that, FTIR spectroscopy was tested to identify the functional groups in the samples. XRD analysis was performed to determine the crystalline phases, full-width at half-maximum (FWHM), and crystal sizes. SEM imaging at 5000× magnification was used to observe surface morphology, while elemental composition was quantified in GO/C-dots samples. The use of PL tests is also used to determine the intensity value, as well as TRPL to determine the electron decay time.

Result and Discussion

Synthesis of Samples Using the Liquid Sonication Exfoliation (LSE) and Oven Heating

The synthesis process for graphite-based materials is depicted in the following images. The resulting GO/C-Dots powder, after filtration and synthesis, is shown in Figure 1.

Drying tobacco stem waste in an oven at 250°C produces a jet-black powder. The jet-black color of oven-dried tobacco stems, especially those that undergo pyrolysis or carbonization at high temperatures with minimal oxygen, indicates a high amorphous carbon (C) content. This carbon is the primary precursor in the production of GO and C-Dots (El-Nahas et al., 2021). The

synthesis outcomes for GO, C-Dots, GO/C-Dots (1:1), and GO/C-Dots (1:3) are shown in Figure 2.

Figure 1. GO/C-dots Powder

GO solution has the characteristics of thick black and liquid (Figure 2(a)), while C-Dots are yellowish, clear, and more liquid (Figure 2(b)). GO/C-Dots 1:1 solution has the characteristics of thick black like GO solution but slightly viscous, while GO/C-Dots 1:3 solution has a higher viscosity than GO/C-Dots 1:1 solution (Figure 2(c)). The thick black color in GO solution is produced from the presence of carbon material that is delaminated and oxidized extensively. Meanwhile, C-Dots solution has a clear yellow color due to the physical and chemical properties that are influenced by carbon precursors and synthesis parameters of temperature and heating time. The yellow color in C-Dots solution is also influenced by the presence of electron transitions in the phi plane structure and the surface of C-Dots at certain gap energies. In GO/C-Dots mixtures (1:1 and 1:3), the darker color is due to the dominant light absorption properties of GO, which masks the fluorescence emission of C-Dots. This effect is more pronounced at a ratio of 1:3, where higher GO concentrations further suppress the fluorescence of C-Dots (Chai et al., 2022; Dong et al., 2012).

(a) (b) (c)

Figure 2. Synthesis results of (a) GO; (b) C-Dots, GO/C-Dots 1:1; and (c) GO/C-Dots 1:3

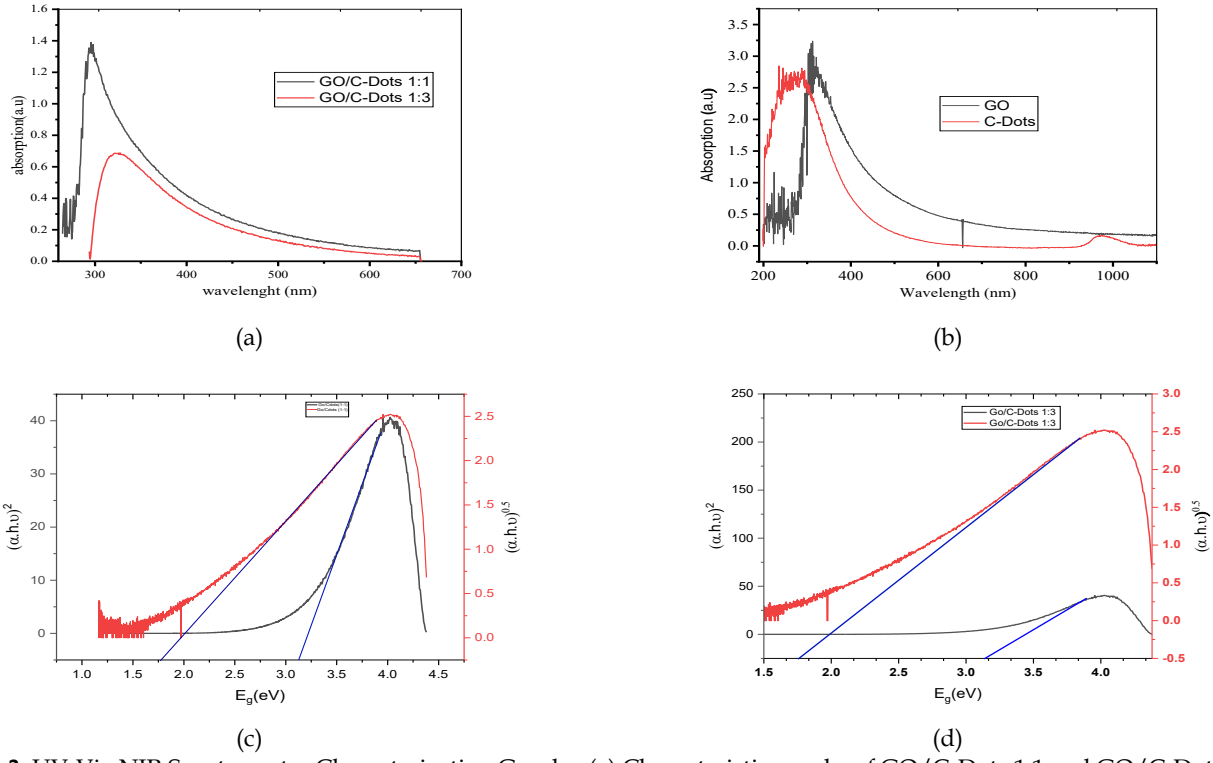


Figure 3. UV-Vis-NIR Spectrometer Characterization Graphs, (a) Characteristic graphs of GO/C-Dots 1:1 and GO/C-Dots 1:3; (b) Maximum absorption peak of GO/C-Dots; (c) Energy gap value graph for GO/C-dots 1:1 for direct transition and indirect transition; (d) GO/C-dots 1:3 for direct transition and indirect transition.

Sample Characterization

The sonication exfoliation process successfully separated the graphene layers from the carbon structure of tobacco stems. This was confirmed through characterization techniques including UV-Vis-NIR spectrophotometry, FTIR spectroscopy, SEM EDX, XRD, PL, and TRPL.

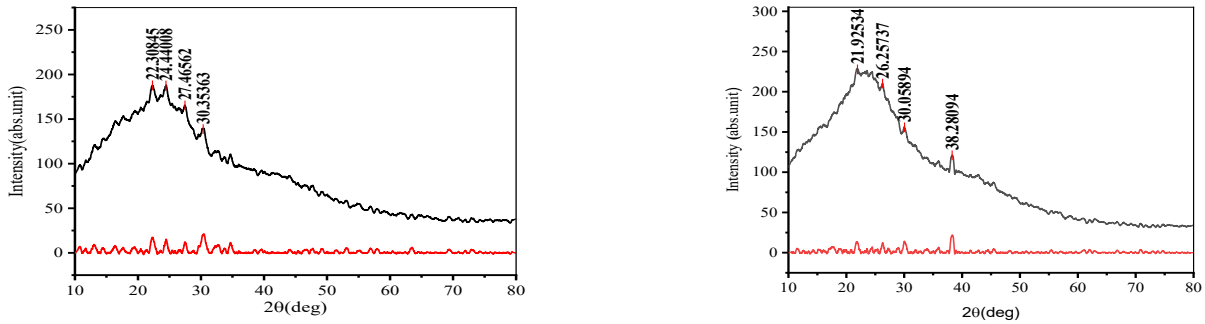
UV-Vis-NIR spectrophotometry

The UV-Vis-NIR test shows that at figure 3(a) the GO/C-Dots 1:1 sample exhibits an absorbance peak at a wavelength of 295.66 nm, while the GO/C-Dots 1:3 sample displays an absorbance peak at 326.52 nm. The combination of GO and C-Dots allows C-Dots to act as a reducing agent for GO (Sartini, 2019), facilitating the reduction of oxygen groups and the exfoliation of GO layers. Figure 3(b) shows the absorption peak of GO is at 312.51 nm, while C-dots have two absorption peaks, namely at 235.38 nm and 973.75 nm. Figure 3(c) demonstrates that the GO/C-Dots 1:1 sample has an energy gap of 1.75 eV for direct transitions and 3.13 eV for indirect transitions. Meanwhile, Figure 3(d) shows the GO/C-Dots 1:3 sample with the same energy gap of 1.75 eV for direct transitions but a slightly higher energy gap of 3.16 eV for indirect transitions.

Based on these results, the GO/C-Dots samples can be categorized as organic semiconductors. This aligns with Nagar (2018) who stated that materials with energy gaps in the range of 1.5–3.5 eV exhibit semiconductor properties.

XRD (X-Ray Diffraction)

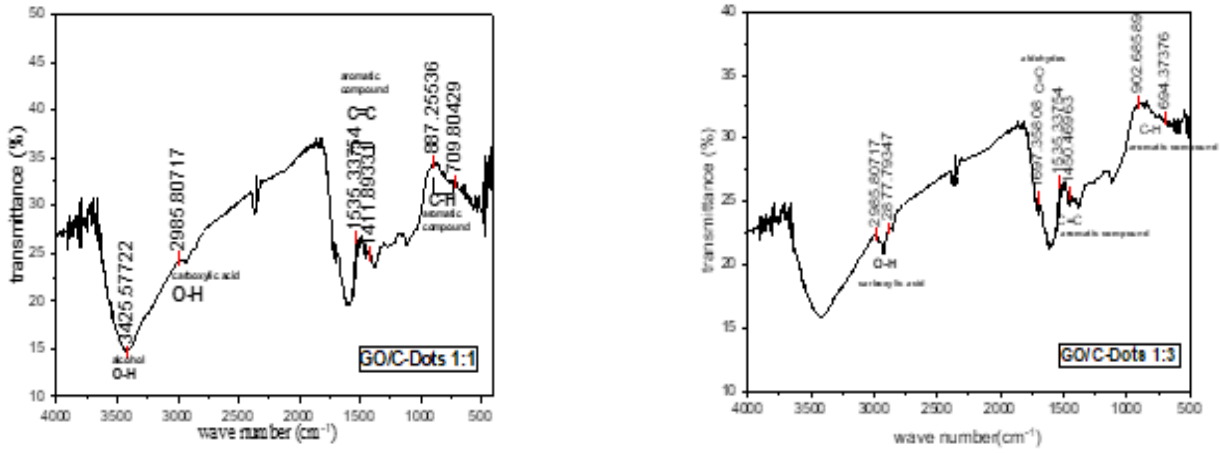
The XRD characteristics of GO/C-Dots using 2 sample ratios of 1:1 and 1:3 produce XRD spectra indicating that the intensity of 2θ is the diffraction angle. The XRD results for both GO/C-Dots 1:1 and GO/C-Dots 1:3 are presented in Figure 4.



(a) (b)

Figure 4. XRD characterization results of GO/C-Dots synthesis (a) 1:1 and (b) 1:3

The change in the XRD angle pattern observed in Figure 4, which shows peaks that broaden and shift to the left at higher angles, indicates an increase in the crystallinity of the graphite oxide material. The peaks that appear in Figure 4 indicate the presence of a crystal structure, which is organized and suggests the existence of crystalline domains in the GO/C-Dots composite material.



(a) (b)

Figure 5. FTIR Characterization Results for Go/C-dots (a) 1:1 and (b) 1:3

FTIR (Fourier Transform Infrared Spectroscopy)

The FTIR test identifies various functional groups, including hydroxyl (O-H), carbonyl (C=O), and carbon-carbon double bonds (C=C) in aromatic structures, highlighting the material's hydrophilic nature and high reactivity potential. This research supports previous

studies that show the presence of oxygen functional groups on the surface of point C.

As shown in Figure 5, the FTIR spectra of the GO/C-Dots samples reveal distinct functional group compositions. The GO/C-dots ratio 1:1 sample contains hydroxyl functional groups (O-H) for alcohols and carboxylic acids, as well as aromatic compounds (C=C) and (C-H). Meanwhile, the GO/C-dots ratio 1:3 sample contains hydroxyl functional groups (O-H) for carboxylic acids, carbonyl groups (C=O), and aromatic compounds (C=C) and (C-H).

The spectral trends indicate notable differences between the two ratios. The GO/C-Dots 1:1 sample shows an increase in hydroxyl (O-H) groups, accompanied by a decrease in carbonyl (C=O) groups, suggesting enhanced alcohol and carboxylic acid functionalities. In contrast, the GO/C-Dots 1:3 sample exhibits a reduction in hydroxyl (O-H) groups and a marked increase in carbonyl (C=O) groups, indicating a shift toward higher aldehyde content.

SEM EDX

SEM analysis revealed the presence of crystalline fragments and porous sheet-like structures, indicating that the larger composition of C-Dots influences the surface morphology of graphene oxide. This structural variation correlates with differences in the elemental composition of the samples, as illustrated in Figure 6.

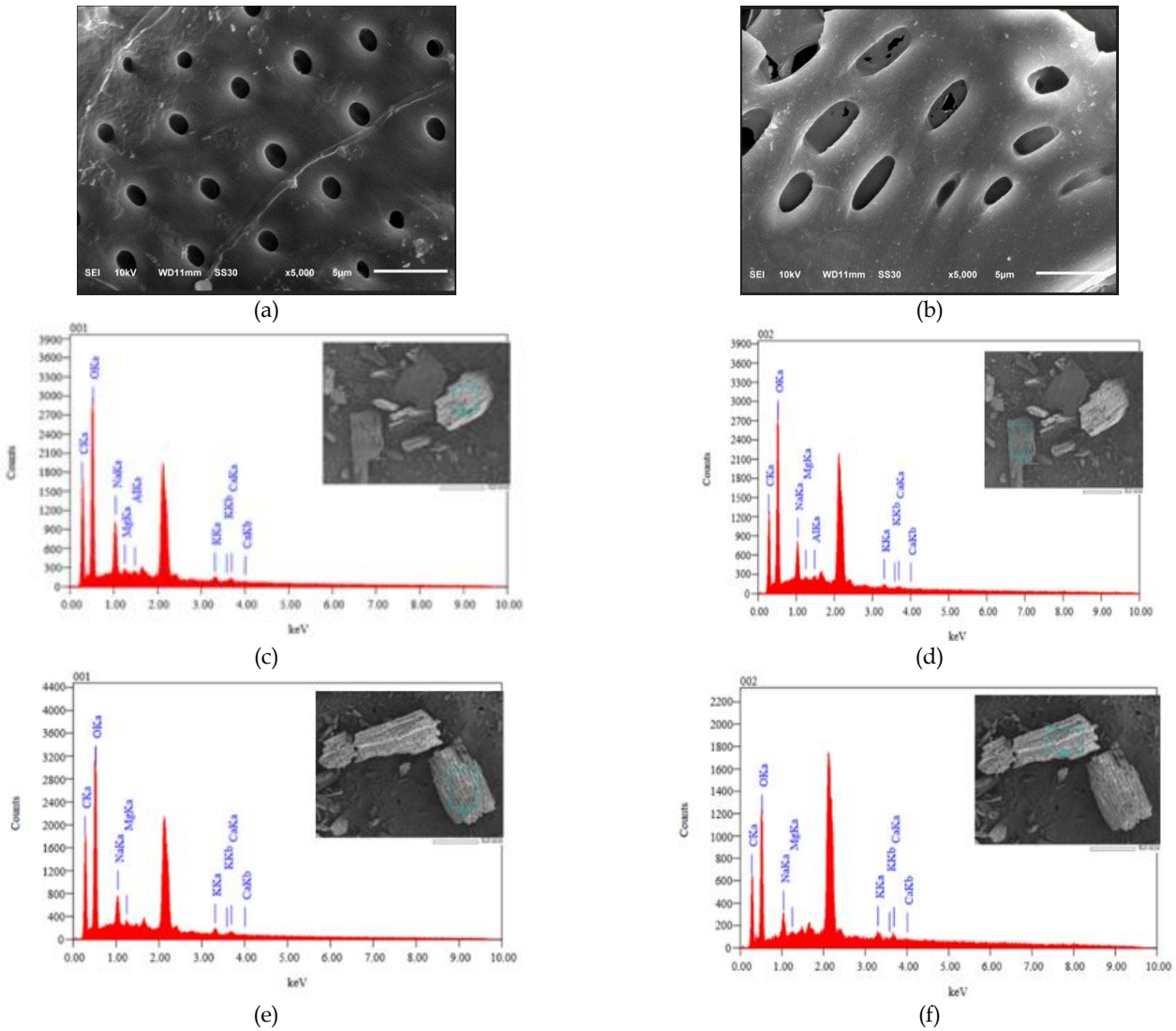


Figure 6. Surface Appearance Results of GO/C-Dots (a) 1:1 Ratio at 5000x Magnification; (b) 1:3 Ratio at 5000x Magnification; (c) Element Composition Graph of GO/C-Dots 1:1 Spot 1; (d) Element Composition Graph of GO/C-Dots 1:1 Spot 2; (e) Element Composition Graph of GO/C-Dots 1:3 Spot 1; (f) Element Composition Graph of GO/C-Dots 1:3 Spot 2

SEM-EDX characterization confirmed the presence of key atomic components, including Mg, Al, Ca, K, Na, C, and O. Notably, the GO/C-Dots ratio of 1:3 exhibited a significantly higher Al content compared to the 1:1 ratio. This suggests that the proportion of C-Dots in the composite affects the incorporation or retention of

certain elements during synthesis. Overall, the synthesis method employed in this study was effective in producing GO/C-Dots materials with distinct structural and compositional characteristics. These findings provide a valuable foundation for further exploration of GO/C-Dots applications across various fields, including electronics, catalysis, and environmental remediation.

Photoluminescence (PL)

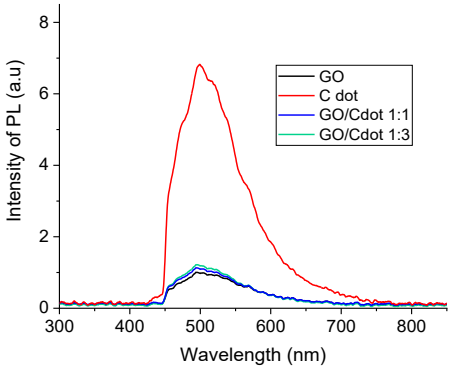


Figure 7. Results of the Photoluminescence (PL) characterization of GO, C-Dots, GO/C-Dots 1:1, and GO/C-Dots 1:3

Figure 7 shows the Photoluminescence (PL) spectrum of GO, C-Dots, GO/C-Dots 1:1, and GO/C-Dots 1:3. All samples have an emission peak at a wavelength of 499 nm. The PL intensity of Cdot is very high compared to GO, C-Dots, GO/C-Dots 1:1, and GO/C-Dots 1:3. Differences in the Go/C-Dots ratio may affect other characteristics such as total intensity, peak width, or peaks at other wavelengths, but for the specific peak at 493.922 nm, similar values indicate similar emission properties. Similar peak intensities indicate that the electron-hole recombination efficiency at that level is the same or comparable for both ratios.

In a study conducted by Georgiopoulou et al. (2023) showed similar peak intensities at 493.922 nm implying that the electron-hole recombination efficiency is comparable, indicating that both materials can function effectively in optoelectronic applications.

Time Resolve Photoluminescence (TRPL)

The measurement of Time-Resolved Photoluminescence (TRPL) from GO, C-Dots, GO/C-Dots 1:1, and GO/C-Dots 1:3 can be seen in the Figure. TRPL fitting was performed with a double exponential using the equation (1).

$$I = A_1 exp(-t/\tau_1) + A_2(-t/\tau_2) \quad (1)$$

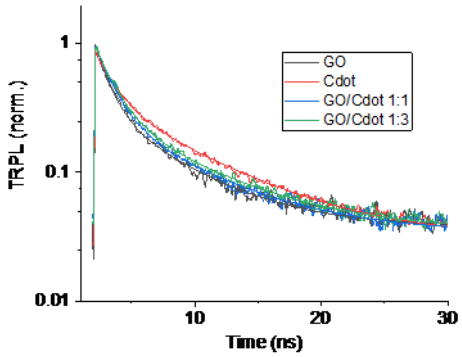


Figure 8. Results of the Time Resolve Photoluminescence (TRPL) characterization of GO, C-Dots, GO/C-Dots 1:1, and GO/C-Dots 1:3

Where I is the TRPL intensity, A_1 and A_2 are the amplitudes of the two exponential components, τ_1 and τ_2 are the lifetimes of the two exponential components. Meanwhile, the average lifetime (τ_{avr}) can be calculated using the equation (2).

$$\tau_{avr} = \frac{\tau_1 A_1 + \tau_2 A_2}{A_1 + A_2} \quad (2)$$

Table 1. Average lifetime calculation results of GO, C-dots, GO/C-Dots 1:1 and GO/C-Dots 1:3

Sampel	A_1	τ_1	A_2	τ_2	τ_{avr}
GO	5.23	1.04	0.40	5.57	1.36
C-Dots	2.22	1.41	0.46	7.21	2.46
GO/C-Dots 1:1	3.24	1.36	0.34	6.70	1.87
GO/C-Dots 1:3	2.93	1.45	0.35	6.97	2.04

The average value (τ_{avr}) of GO, C-Dots, GO/C-Dots 1:1, and GO/Cdot 1:3 is almost the same as seen in the table 1. C-dot has the highest τ_{avr} value and GO has the lowest τ_{avr} value, consistent with the trend of PL intensity where C-dot has the highest value and GO the lowest.

At a 1:3 ratio, the lifetime is longer (2.04 ns) than at 1:1 (1.87 ns), implying slightly longer electron-hole recombination, or better excitation retention efficiency. A longer lifetime typically indicates reduced non-radiative recombination (energy loss without light emission), so materials with a 1:3 ratio tend to have higher photoluminescence efficiency than those with a 1:1 ratio. This difference in lifetime could be due to different interactions between GO and C-Dots at these ratios, affecting the trap state mechanism, recombination rate, and energy transfer.

Time-resolved photoluminescence (TRPL) tests for graphene oxide (GO) and carbon dots (C-Dots) in 1:1 and 1:3 ratios, with lifetimes of 1.87 ns and 2.04 ns, respectively, provide insights into the carrier dynamics and recombination processes in these materials. Different lifetimes indicate variations in charge carrier

behavior influenced by the GO to C-Dots ratio, which may affect their potential applications in optoelectronic devices.

Conclusion

This study successfully synthesized GO/C-Dots composite material from tobacco stem graphite using Liquid Sonication Exfoliation (LSE) method and oven heating. The characterization results showed that the GO/C-Dots material has organic semiconductor properties, good crystalline structure, and active functional groups that vary depending on the composition ratio. PL and TRPL tests revealed that the 1:3 ratio provides a longer emission lifetime and higher photoluminescence efficiency compared to the 1:1 ratio. Overall, the GO/C-Dots material shows great potential for applications in the field of optoelectronics and other functional technologies.

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Author Contributions
ST as visualization, investigation, formal analysis, writing – original draft, resources, and writing - review & editing. RE as investigation, formal analysis, and writing - original draft. AM as visualization, investigation, and formal analysis. F as validation and funding acquisition. DA as conceptualization, supervision, metodology, and validation. AH as data curation and supervision.

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Conflicts of Interest
The authors declare no conflict of interest.

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