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Synthesis and Characterization of Carbon Dots (C-Dots) Derived from Tofu Dregs using Solvothermal Method

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ABSTRACT

Tofu dregs are an agroindustrial organic byproduct generated in large quantities and still contain carbon-rich compounds such as carbohydrates, proteins, and lipids. This carbon content makes tofu dregs a promising alternative precursor for the synthesis of value-added carbon-based materials, one of which is carbon dots (C-Dots). This study aims to synthesize C-Dots derived from tofu dregs using a solvothermal method and to characterize the optical properties, functional groups, morphology, and elemental composition of the resulting material. The synthesis of C-Dots was carried out using a solvothermal process involving heating in a closed system using tofu dregs and 96% ethanol as the solvent. Characterization was performed using UV–Visible (UV–Vis) spectrophotometry, Fourier Transform Infrared–Attenuated Total Reflectance (FTIR–ATR), and Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM–EDX).
The UV–Vis analysis revealed an absorption band at approximately 451 nm, which is associated with π→π* electronic transitions in conjugated carbon structures, as well as a shoulder around 524 nm that can be attributed to n→π* transitions originating from lone pair electrons in oxygen-containing functional groups. FTIR analysis indicated the presence of O–H/N–H, C–H, C=C, C–O/C–O–C, and C–N functional groups on the surface of the C-Dots. Meanwhile, SEM analysis showed agglomerated particle morphology, and EDX results confirmed that the main constituent elements of the material are carbon (C), oxygen (O), and nitrogen (N). The results of this study demonstrate that tofu dregs have strong potential to be utilized as a biomass-derived precursor in the solvothermal synthesis of C-Dots.

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INTRODUCTION

Indonesia is one of the countries with a high level of tofu production, driven by the substantial consumption of soybean-based products as a primary source of plant protein. The tofu industry is widely distributed across various regions and contributes significantly to the local economy [1]. However, the tofu production process generates large quantities of solid waste in the form of tofu dregs, which have not yet been optimally utilized. Most tofu dregs are still disposed of directly into the environment or used as animal feed with relatively low economic value. This condition poses potential environmental problems, such as water pollution and unpleasant odors, due to the high organic content of the waste [2].

Tofu dregs are known to contain considerable amounts of nutrients and organic compounds, including carbohydrates, proteins, lipids, and complex carbon compounds [3]. The abundant carbon content makes tofu dregs a promising raw material for the synthesis of carbon-based materials. The utilization of tofu dregs as an alternative carbon source not only adds value to organic waste but also represents a sustainable approach to managing food industry byproducts. This strategy is aligned with the principles of green chemistry, which emphasize efficient resource utilization and the reduction of environmental impact [4]. Therefore, it is necessary to develop innovative approaches to convert tofu dregs into functional products with higher economic and scientific value, such as value-added carbon materials, including carbon dots (C-Dots).

C-Dots are carbon nanoparticles with sizes below 10 nm that exhibit unique fluorescence properties, excellent chemical stability, high biocompatibility, and environmental friendliness [5]. These characteristics have led to their extensive development for various applications, including chemical sensing, bioimaging, photocatalysis, and optoelectronic devices [6]. The use of biomass waste as a carbon source for the synthesis of C-Dots is an attractive approach, as it supports the development of environmentally friendly materials while reducing dependence on synthetic chemicals. Various types of biomass have been employed as precursors for C-Dots synthesis, such as lime, corn husks, lemon peels, onion waste, and other agricultural residues. These biomaterials generally contain carbon-rich organic compounds, making them suitable as alternative carbon sources for the formation of nanoscale carbon materials [7].

The synthesis of C-Dots can be carried out through two main approaches, namely top-down and bottom-up. The top-down approach involves breaking down bulk carbon materials into nanoparticles, whereas the bottom-up approach involves the carbonization of simple organic molecules [8]. Bottom-up methods, such as the solvothermal approach, are widely used due to their relatively simple process, efficiency, and ability to produce C-Dots with optimal fluorescence properties. In the solvothermal method, carbon precursors are heated in a closed system under high temperature and pressure, leading to thermal decomposition and the formation of nanoscale carbon structures. Furthermore, this method generally does not require catalysts or hazardous solvents, making it safer and more sustainable than conventional chemical methods [9]. After synthesis, a characterization stage is required to confirm the formation of C-Dots and to identify the physicochemical properties of the resulting material. This characterization is essential for determining the optical properties, chemical structure, and surface morphology of the synthesized C-Dots.

RESEARCH METHODS

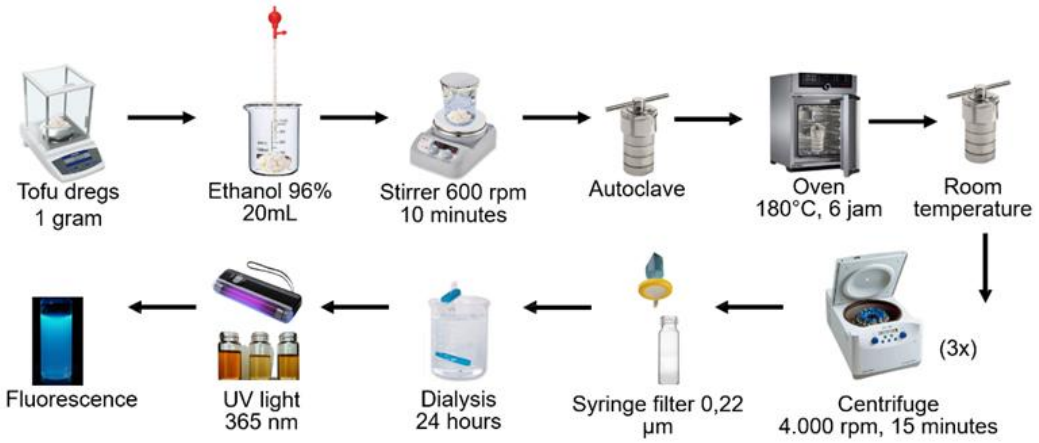

Figure 1. Schematic Illustration of C-Dots Synthesis using the Solvothermal Method

Method

The synthesis of C-dots was carried out using a solvothermal method with 96% ethanol as the solvent. Tofu dregs powder (1.0 g) was accurately weighed using an analytical balance and transferred into a 50 mL beaker. Subsequently, 20 mL of 96% ethanol was added as the solvent, and the mixture was homogenized. The homogeneous mixture was then transferred into a 50 mL Teflon-lined autoclave and heated in an oven at 180 °C for 6 hours to facilitate the solvothermal process. After the heating process was completed, the autoclave was allowed to cool naturally to room temperature. The resulting solution was transferred into centrifuge tubes and separated using a centrifuge at 4.000 rpm for 15 minutes. The obtained supernatant was then filtered through a 0.22 µm syringe filter to remove larger particles. The filtrate was subsequently placed into a dialysis membrane with a molecular weight cut-off (MwCO) of 1.000 Da and dialyzed against 96% ethanol for 24 hours, with the dialysis medium being replaced every 6 hours. After dialysis, the resulting C-dots solution was further characterized using a double-beam UV–Vis spectrophotometer (Jenway 6850), FTIR (Bruker Alpha II), and SEM–EDX (Quanta 650).

RESULTS AND DISCUSSION

In this study, C-Dots were synthesized from tofu dregs using a solvothermal method. This method utilizes high-temperature and high-pressure conditions in a closed system to facilitate chemical reactions among the organic components present in the biomass [10]. During the solvothermal process, the organic compounds in tofu dregs undergo a series of chemical transformations, including hydrolysis, dehydration, fragmentation, polymerization, condensation, and aromatization. In the initial stage, hydrolysis occurs, breaking down complex biomass components into simpler molecules. This is followed by dehydration reactions that generate more reactive intermediate compounds. These intermediates subsequently undergo fragmentation into smaller molecules, which then experience polymerization and condensation to form conjugated carbon structures. In the final stage, aromatization takes place, resulting in the formation of nanometer-sized aromatic carbon domains that constitute the carbon core of the C-Dots. These cores are typically surrounded by various surface functional groups, such as hydroxyl, carbonyl, and carboxyl groups, formed due to partial oxidation during the carbonization process [11]. The success of the C-Dots synthesis process can generally be indicated by observable changes in the solution characteristics, particularly the formation of a brown to yellowish-brown solution, which suggests the formation of nanoscale carbon materials. In addition, C-Dots solutions typically exhibit fluorescence when exposed to ultraviolet (UV) light [12]. To confirm the formation of C-Dots structures and to determine the physicochemical properties of the resulting material, further characterization was carried out using several analytical techniques.

Analysis Using a UV Lamp at 365 nm

Visual analysis using a 365 nm UV lamp was conducted to directly observe the fluorescence properties of the synthesized C-Dots. This test aims to evaluate the ability of C-Dots to emit visible light when excited by UV radiation at a wavelength of 365 nm. In general, observation under UV light serves as a simple yet effective qualitative method to confirm the presence of the characteristic optical properties of C-Dots, which are typically indicated by the emission of specific colors resulting from electron transitions from excited energy states back to the ground state. This phenomenon is closely related to the aromatic carbon core structure and the functional groups present on the surface [13].

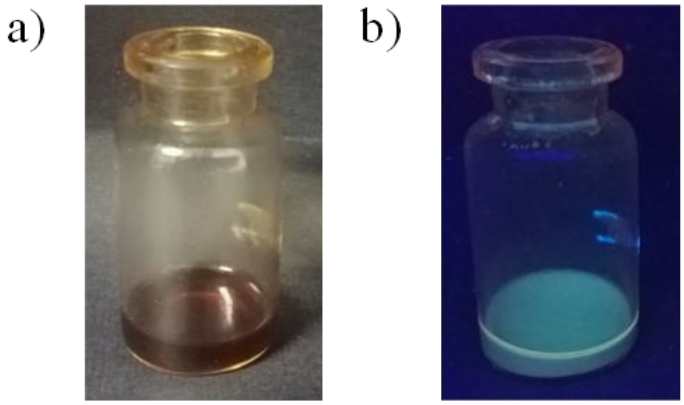

Figure 2. (a) C-Dots Before UV Irradiation at 365 nm; (b) C-Dots After UV Irradiation at 365 nm

Based on the observations presented in Figure 2, the solution obtained from the synthesis of C-Dots derived from tofu dregs exhibits a clear change in optical characteristics when observed under ultraviolet (UV) irradiation at a wavelength of 365 nm. Visually, the solution appears light brown under visible light but displays a bright blue emission when exposed to UV light at 365 nm. This blue emission is a typical characteristic of C-Dots and serves as a qualitative indication of the successful formation of nanoscale carbon materials [14]. The fluorescence observed in C-Dots arises from the excitation of electrons from the ground state to higher energy levels upon absorption of UV radiation, which is primarily associated with $\pi \rightarrow \pi^*$ electronic transitions within sp^2-bonded aromatic carbon domains. This transition involves the promotion of electrons from bonding π orbitals to antibonding π* orbitals and requires relatively high energy, resulting in strong absorption in the UV region. Following excitation, the electrons undergo relaxation back to lower energy states, accompanied by the emission of visible light [15]. The resulting blue emission is attributed to the extremely small particle size of the C-Dots, which leads to the quantum confinement effect. This effect causes the discretization of energy levels and an increase in the band gap, thereby shifting the emission toward shorter wavelengths, specifically within the blue region of the visible spectrum [16].

Analysis Using a UV–Vis Spectrophotometer

UV–Vis analysis was conducted over a wavelength range of 400–800 nm to evaluate the optical absorption properties of the synthesized carbon material. The UV–Vis spectrum of the C-Dots obtained using the solvothermal method is presented in Figure 2.

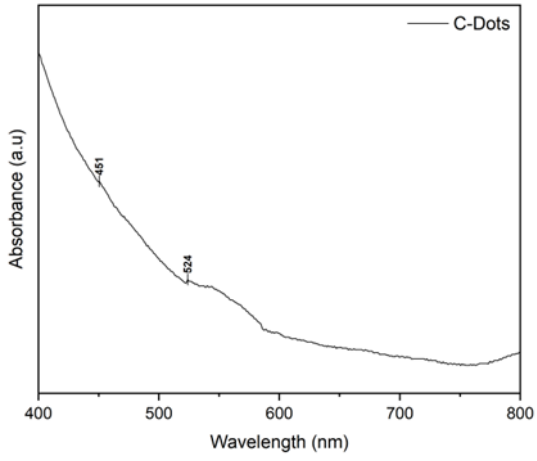

Figure 3. UV–Vis Spectrum of C-Dots

Based on the spectrum presented in Figure 3, a relatively strong absorption band is observed at approximately 451 nm, along with a weaker shoulder around 524 nm. The absorption band at 451 nm is attributed to π → π* electronic transitions originating from conjugated C=C double bonds within the aromatic carbon core of the C-Dots. During the solvothermal process, the organic components in the biomass give rise to small aromatic domains. These aromatic domains are capable of absorbing light energy, resulting in the excitation of electrons from π orbitals to antibonding π* orbitals [17]. Furthermore, the presence of an absorption shoulder at approximately 524 nm is associated with n → π* electronic transitions arising from lone pair electrons in oxygen-containing functional groups, such as C–O and C–O–C, located on the surface of the C-Dots [18].

The two-band absorption pattern observed in this study is consistent with previous findings reported by Zhao [19] who demonstrated that C-Dots synthesized using a solvothermal method from 1,2,4,5-tetraaminobenzene in DMF solvent exhibited absorption peaks at approximately 450, 483, and 523 nm, which are comparable to the absorption features shown in Figure 3. An increase in solvent polarity leads to a shift toward shorter wavelengths (blue shift) in the λ_max values due to the formation of interactions such as hydrogen bonding between the lone pair electrons of amino groups and the polar solvent. This interaction increases the energy gap between the n and π* orbitals. The agreement in the absorption band positions suggests that the C-Dots synthesized in this study possess structural characteristics comparable to those reported in previous studies.

Functional Group Analysis Using FTIR–ATR

Functional group analysis on the surface of the C-Dots was carried out using FTIR–ATR. Each functional group exhibits characteristic absorption frequencies that can be used to determine the chemical composition and structural changes occurring during the synthesis process. Through FTIR–ATR analysis, the presence of functional groups in the synthesized C-Dots can be identified [20].

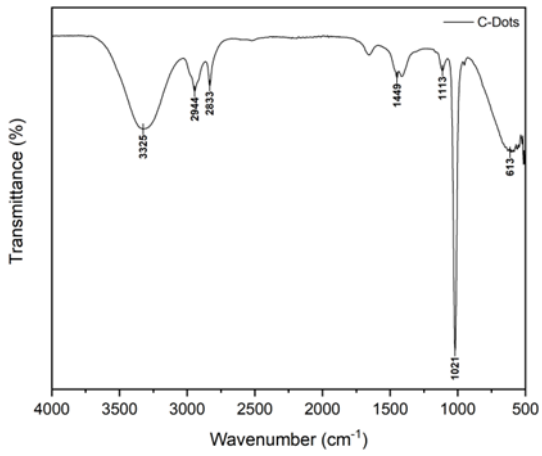

Figure 4. FTIR Spectrum of C-Dots Synthesized from Tofu Dregs using the Solvothermal Method

The results of the FTIR–ATR characterization are presented in Table 1, as follows.

Table 1. FTIR–ATR Results and Functional Group Identification of C-Dots

Wavenumber (cm^{-1})	Functional Group
3325	O-H/N-H
2944	C-H alifatik
2833	C-H alifatik
1449	C=C aromatik
1113	C-O/C-O-C
1021	C-N
613	C-H

Based on Figure 4 and Table 1, the FTIR–ATR spectrum of the C-Dots exhibits several characteristic absorption bands representing the surface functional groups of the nanoscale carbon material. The broad absorption band observed at 3325 cm^{-1} indicates the presence of O–H/N–H groups. These groups are typically derived from biomass organic compounds and oxidation products formed during the carbonization process, and they contribute to enhancing the hydrophilic properties of the C-Dots [21]. The absorption bands at 2944 cm^{-1} and 2833 cm^{-1} are attributed to aliphatic C–H stretching vibrations, indicating the presence of amorphous carbon structures consisting of a combination of sp^2 and sp^3 domains [22]. Furthermore, the absorption band at 1449 cm^{-1} corresponds to aromatic C=C stretching vibrations, suggesting the formation of conjugated carbon structures that constitute the core of the C-Dots [23]. The absorption band at 1113 cm^{-1} indicates the presence of oxygen-containing functional groups such as C–O and C–O–C, which are typically associated with alcohol, ether, or ester groups on the surface of the C-Dots [24]. Meanwhile, the absorption band at 1021 cm^{-1} is attributed to C–N stretching vibrations, reflecting the incorporation of nitrogen originating from the tofu dregs biomass. In addition, the absorption band at 613 cm^{-1} corresponds to out-of-plane C–H bending vibrations of aromatic rings, further confirming the presence of aromatic carbon structures in the material [25].

Analysis Using SEM–EDX

SEM–EDX analysis was conducted to observe the surface morphology and elemental composition of the sample [26]. SEM characterization requires several sample preparation steps, as the liquid sample must be converted into a solid (powder) form to enable proper observation. In this study, the liquid sample was poured into a beaker and heated on a hot plate until the solvent evaporated, leaving a caramel-like residue. The sample was then examined using an SEM instrument (FEI Quanta 650) at a magnification of 200× with a scale of 200 µm. The SEM characterization results are presented as micrograph images of the sample, as shown in Figure 5.

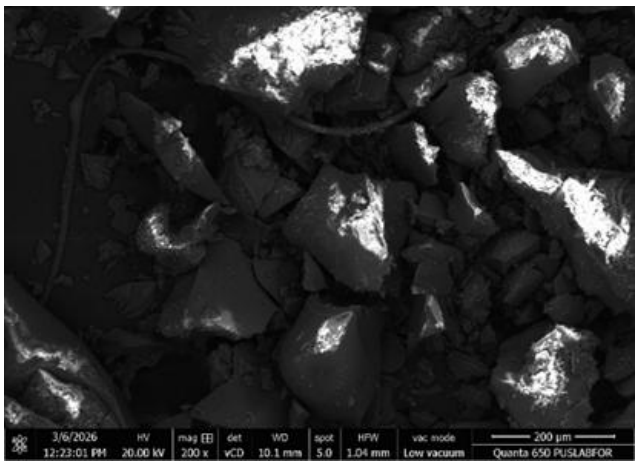

Figure 5. SEM Characterization Results of C-Dots

Based on SEM observations, the synthesized material exhibits an aggregated morphology with irregular shapes, in which the particles appear to coalesce into larger structures. The particle surfaces appear rough, and the aggregates are relatively large in size. This phenomenon is commonly observed in C-Dots materials that have undergone post-treatment processes such as drying, where nanoscale carbon particles tend to agglomerate due to interparticle interactions, including van der Waals forces and surface functional group interactions. Such agglomeration occurs as the reduction of solvent decreases the interparticle distance, thereby promoting aggregation and the formation of larger structures when observed under SEM [27].

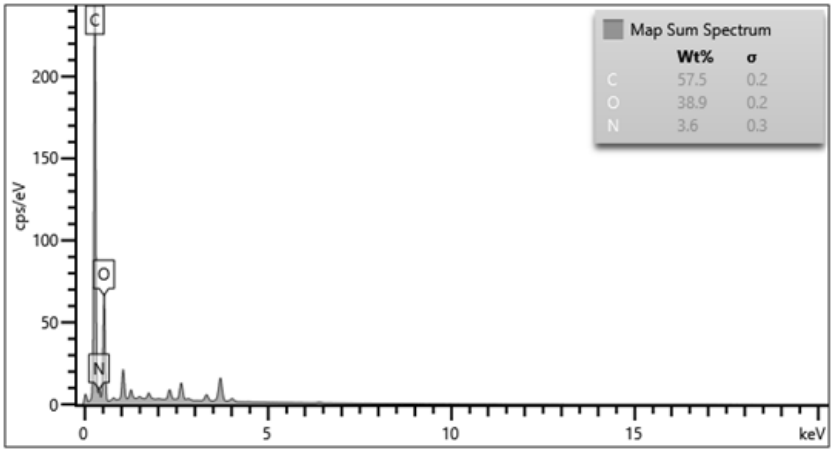

Figure 6. EDX Characterization Results of C-Dots

Elemental composition analysis using EDX indicates that the synthesized material consists primarily of carbon (C), oxygen (O), and nitrogen (N). Based on the EDX spectrum shown in Figure 6, the detected elemental percentages are 57.5% carbon, 38.9% oxygen, and 3.6% nitrogen. The dominance of carbon confirms that the material is carbon-based. Meanwhile, the relatively high oxygen content suggests the presence of oxygen-containing functional groups on the surface of the C-Dots, such as C–O and C–O–C. The presence of nitrogen in the C-Dots structure is typically associated with bonding configurations such as N–H or C–N. Nitrogen doping can modify the electronic structure by increasing electron density and introducing defect states, which may influence the optical and fluorescence properties of the C-Dots. As a result, C-Dots tend to exhibit emission in the blue to blue-green region, as observed in the UV 365 nm analysis shown in Figure 2b [28]. These findings are consistent with the FTIR analysis, which

revealed the presence of functional groups such as O–H/N–H, C–H, C=C, C–O/C–O–C, and C–N, indicating the incorporation of heteroatom-containing groups on the material surface. Therefore, the FTIR and EDX characterization results complement each other in confirming both the functional groups and the elemental composition of the synthesized C-Dots.

CONCLUSION

This study demonstrates that tofu dregs have significant potential as a precursor for the synthesis of C-Dots using the solvothermal method. The resulting material exhibits characteristic features of nanocarbon, as evidenced by its optical properties and chemical structure. UV–Vis analysis reveals the presence of a conjugated carbon system, as well as surface functional groups that play a crucial role in electronic transitions. These findings are further supported by FTIR analysis, which confirms the presence of functional groups such as O–H/N–H, C–H, C=C, C–O/C–O–C, and C–N on the material surface. Meanwhile, SEM–EDX results indicate that the material is primarily composed of carbon, oxygen, and nitrogen, with a particle morphology characterized by agglomeration. The successful formation of C-Dots in this study is more convincingly demonstrated by the material's ability to exhibit fluorescence under UV irradiation, which is a key characteristic of C-Dots. This is particularly important because SEM-based morphological analysis cannot accurately represent the true particle size due to agglomeration occurring during the drying process, making nanoscale dimensions difficult to observe directly. Therefore, for future work, it is recommended to conduct characterization using Transmission Electron Microscopy (TEM) on samples in the solution phase to obtain more accurate information regarding particle size.

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