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CHARATERIZATION ON OPTICAL PROPERTIES OF CARBON NANODOTS FROM SCREW PINE LEAVES FOR COPPER ION DETECTION

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Abstract

Here, we fabricate, characterize and synthesize the highly fluorescent carbon nanodots (C-dots) from screw pine leaves (SPL). The objectives of this research are to fabricate C-dots from SPL using the carbonization method which is followed by sonication, centrifugation and filtering processes, to charactrize the optical properties of the as-prepared C-dots, and to applied these C-dots as sensor for detection of copper (Cu^{2+}) ions. The optical properties of the C-dots can be characterized from their absorption (Abs), photoluminescence (PL), and Fourier Transform infrared (FTIR) spectra. Whereas the application of the as-produced C-dots as sensor for Cu^{2+} ions detection can be investigated by the quenching of PL intensity of C-dots after coordination with Cu^{2+} ions. The analysis of Abs, PL, and FTIR spectra demonstrated the formation of C-dots from SPL in ethanol solutiion. These C-dots have an absorption peak at 276 nm corresponding to the $\pi \rightarrow \pi^*$ transition and PL peak at 401 nm corresponding to their blue fluorescence. Fortunately, the interaction between the as-prepared C–dots and Cu^{2+} ions displayed the PL quenching of the C-dots with the LOD value as low as 2.85 μM. Based on these results, these C-dots exhibited the sensing system for Cu^{2+} ions detection

Keywords: *Characterization; Optical properties; C-dots; Screw pine leaves; Copper ions*

PENDAHULUAN

Carbon nanodots (C dots) are the youngest member of the nanomaterial family with quasi-zero dimension and size regime of less than 10 nm that were serendipitously discovered in 2004 at the time of purification of single-wall carbon nanotubes[1,2]. The carbon core of C-dots is predominant sp and an oxidized carbon surface with carbonyl and hydroxyl groups[3,4]. C-dots are composed of heteroatoms such as carbon (C), hydrogen (H), Nitrogen (N), and oxygen (O). According to the elemental analysis, C-dots contain 55.93 % C, 2.65 % H, 1.2 % N, and 40.3 % O[5]. Generally, the ratio of C, H, N, and O in the C-dots is a little different and depends on different synthetic methods[6] Whereas conventional carbon contains 91.7 % C, 1.8% H, 1.8% N, and 4.4 % O[7]. Therefore, the preparation of C-dots is a process to reduce the mass of carbon atoms to become the mass of oxygen atoms so hydroxyl and carbonyl groups can be formed in the molecule structure of C-dots[8]. These groups induce C-dots to have good solubility in water. C-dots have been gaining importance in both fundamental research and industry owing to their unique and novel properties[9]. The most notable properties of C-dots are excellent photoluminescence (PL) in the ultraviolet-visible and near-infrared region[10,11] which make C-dots have a wide range of applications in different fields of human life, high photostability[12], high solubility in water [13,14], excellent biocompatibility [15] and bioimaging[16]. The other applications of the C-dots are photocatalysis[17], Light-emitting diode[9] and biomedicine[18].

Carbon as a source to generate C-dots is an element that can be easily found in every organic material so C-dots can be easily prepared from organic material and

their waste. So far, the fabrication of C-dots from various benign agro-based waste products has been conducted. For example, C-dots from coconut shell[1,2], mango peel[19,20], cassava peel[21], dragon fruit peel [22,23], orange peel[24], soursop juice[8], soursop peel[11], avocado peel[25] and rice husk[26,27]. These C-dots have been applied as sensors, bioimaging, and light-emitting diodes.

When the C-dots were coordinated with metal ions, the PL intensity of C-dots will quench [13]. The quenching of this PL intensity can be used to detect poisonous metal ions through the determination of the lowest concentration from metal ions or an analytical blank which is called the limit of detection (LOD). For instance, Yunfei Sha, et al., prepared C-dots from pipe tobacco using the hydrothermal method. When these were blended with copper (Cu^{2+}) ions, the PL intensity of the C-dots quenched and the LOD value of Cu^{2+} ions was evaluated as low as 0.01 μM.. Yingshuai Liu, et al., fabricated C-dots from bamboo leaves using the hydrothermal method. Thus, the PL intensity of these C-dots decreased after coordinated with Cu^{2+} ions and the LOD value is 0.115 μM which is much lower than the maximum level (∼20 μM) of Cu^{2+} in drinking water permitted by the US Environmental Protection Agency[28]. Vedamalai, et.al., synthesized C-dot from o-phenylene-diamine with Cu^{2+} ions and the LOD value of Cu^{2+} ions is 1.85 μM[29].

In this work, we prepared C-dots from screw pine leaves (SPL) using the carbonization method at 300ºC for 10 minutes and applied them for detection of Cu^{2+} ions. The Copper element is one of the most important elements in living systems and industry. The toxicity of metal ions including Cu^{2+} ions has been found in food to affect the activity of coenzymes such as superoxide dismutase and the signalling process. The lower and/or higher concentration of Cu^{2+} ions in the human body will induce Alzheimer's disease and other diseases (VD)… The concentration measurement of Cu^{2+} ions in the human body is, therefore, necessary to be efficiently and effectively conducted for healthcare concerns. The Cu^{2+} ions were selected due to their positively charged nature which poses a high probability of interacting with the surface of the C-dots containing carboxyl groups. The SPL are a good candidate as starting precursor since they are considered a renewable resource, considerably cheap, and easily obtained because of their abundance as well as environmentally friendly raw material. This work suggests an option to use cheap agricultural in advanced optical sensing nanomaterial of high commodity value.

Under illumination with a UV lamp at 365 nm, the as-prepared C-dots emitted a blue emission color, and their absorption (Abs) peak at 276 nm corresponding to the $\pi \rightarrow \pi^*$ transition. Fortunately, by using an excitation wavelength (λ_{exc}) of 340 nm, the PL peak at 401 nm corresponds to their blue emission color. Interestingly. After the as-prepared C-dots coordinated with Cu^{2+} ions, the PL intensities of the C-dots quenched with the LOD value for Cu^{2+} ions was evaluated as 2.85 μM.

EXPERIMENTAL SECTION

1. Materials and apparatus

SPL were purchased from the traditional market in Tambolaka City, southwest Sumba Regency, East Nusa Tenggara Province, Indonesia. The chemical materials such as ethanol, methanol, copper (II) chloride dihydrate ($CuCl_2.2H_2O$), phosphate buffer sulfate (PBS), and so on were bought from Sumber Ilmiah Persada in Surabaya city and Multiguna shop in Kupang city. All these chemical materials were used without any further purification. The fluorescent color observation of C-dots was conducted by illumination with a UV lamp at 365 nm. The Abs and PL spectra of C-dots were investigated by using spectrophotometry of model JASCO UV-570 and SHIMADZU RF-6000, respectively. Fourier Transform

Infrared (FTIR) spectrum was recorded at 25°C with a JASCO model FT/IR-4200 Fourier transform infrared spectrophotometry.

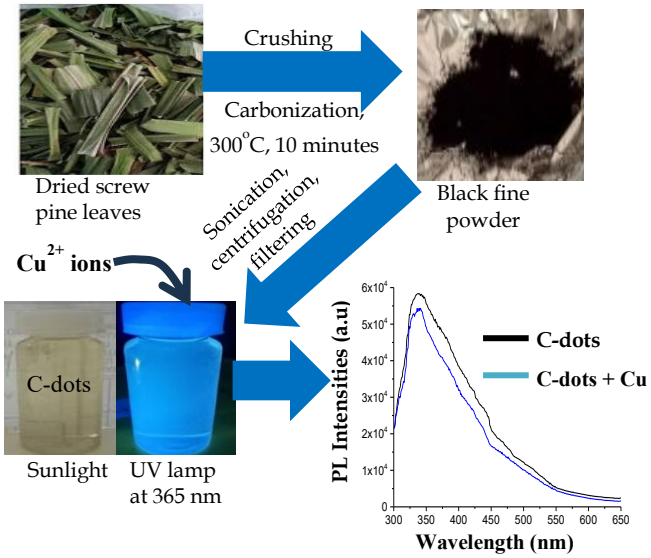
2. Fabrication of C-dots from screw pine leaves

The fabrication of C-dots from SPL uses the carbonization method at 300ºC for 10 minutes which is followd with the sonication, centrifugation and filtering process. Completely, SPL (1kg) was washed by using water to remove dirt and other sediments present in the SPL. Then, it was dried in sunlight for 6h to remove the moisture content. After the drying process, it was crushed to get fine powder. This fine powder (60 g) was carbonized at the temperature of 300ºC for 10 minutes to get a black sample and cooled down to room temperature. Furthermore, the black fine powder sample (0.5 gram) was sonicated in ethanol (5 mL) for an hour to obtain a homogeneous dark solution and ethanol (8 mL) was added again to this sample for centrifugation process at 1000 rpm for 30 minutes. Finally, the resultant supernatant containing fluorescent C-dots in the transparent bottle was collected by removing larger particles through the filtration process. This supernatant was irradiated by a UV lamp at 365 nm to observe the fluorescent color of the C-dots. Finally, these C-dots were stored in a dark and cool place for further study, especially the measurement of their absorption, PL, and FTIR spectra using spectrophotometry of model JASCO UV-570, SHIMADZU RF-6000, and a JASCO model FT/IR-4200 Fourier transform infrared spectrophotometer, respectively.

3. Synthesis of C-dots with copper metal

The concentration detection of Cu^{2+} ions was conducted at room temperature. Briefly, C-dots (1 mL) dispersion in ethanol (3 mL) were added into 1 mL of PBS (1M, pH 7.0), followed by the addition of Cu^{2+} ions (50 µL) with various concentrations (0-10 µM). Furthermore, the PL spectrum of C-dots blending with Cu^{2+} ions was investigated by model SHIMADZU RF-6000.

DISCUSSION



Schema 1. The schematic illustration of simple preparation to realize C-dots from screw pine leaf and synthesis of C-dots with Cu^{2+} metal ion

1. Absorption, Photoluminescence, and FTIR spectra

In this work, the Abs, PL, and FTIR spectra of the as-prepared C-dots were characterized. Figure 1A shows the Abs (black line) and PL (red line) spectra of C-dots from SPL. Their Abs spectrum exhibits a wide Abs band with a peak at around 276 nm corresponding to the π→π* transition of the C=C bond in the heterocyclic ring[6,30] which was similar to the Abs area of the C-dots made from pomelo peel[31] and soursop peel[11]. The Abs peak at 276 nm confirmed the formation of C-dots from SPL [13]. When these C-dots are excited with excitation wavelength(λ_{exc}) at 340 nm, a strong PL peak around 401 nm is realized. This peak corresponds to the blue fluorescence of the as-prepared C-dots under illumination with UV lamp at 365 nm[1,22]. Fortunately, the bathochromic property of PL peaks of these C-dots is observed when the λ_{exc} increases as displayed in

Figure 1B. The PL peaks shifted to longer wavelength from 340 to 466 nm as the emission wavelength (λ_{em}) with the increase of λ_{exc} from 280 to 400 nm, accompanied by the decrease of PL intensity. These PL peaks are strongly dependent on the $\lambda_{exc.}$ This result exhibits the λ_{em} can be tuned by just controlling λ_{exc} with out changing the C-dots[11,13,22].

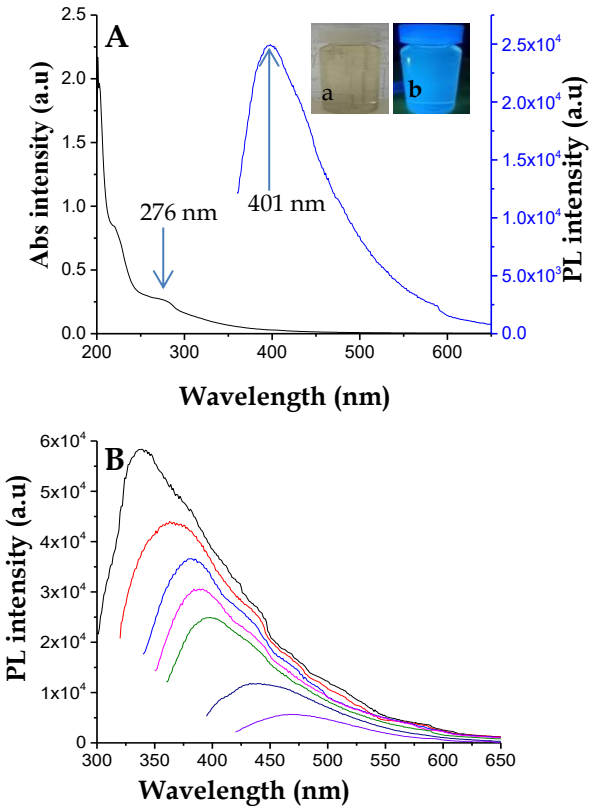


Figure 1 A. Abs (black line) and PL (blue line) spectra of the C-dots dispersion in ethanol (Inset: the photographs of the C-dots dispersion in ethanol with a) sunlight and (b) UV at 365 nm illumination, B. PL spectra of the C-dots dispersion in ethanol depending on excitation wavelength (λ_{exc}),

The PL mechanisms of C-dots are still a challenge for Scientists to explain in more detail. So far, four main factors can affect the PL mechanisms for C-dots, namely quantum size effect (intrinsic state emission), surface defect states, bandgap transition, and surface passivation[6]. The surface passivation of bare C-dots and the quantum size effect can enhance the PL of the C-dots[6,32]. With the enhancement of surface oxidation degree from C-dots, more surface defects are formed to trap more excitons, resulting in a bathochromic of the emission wavelength of C-dots[6].

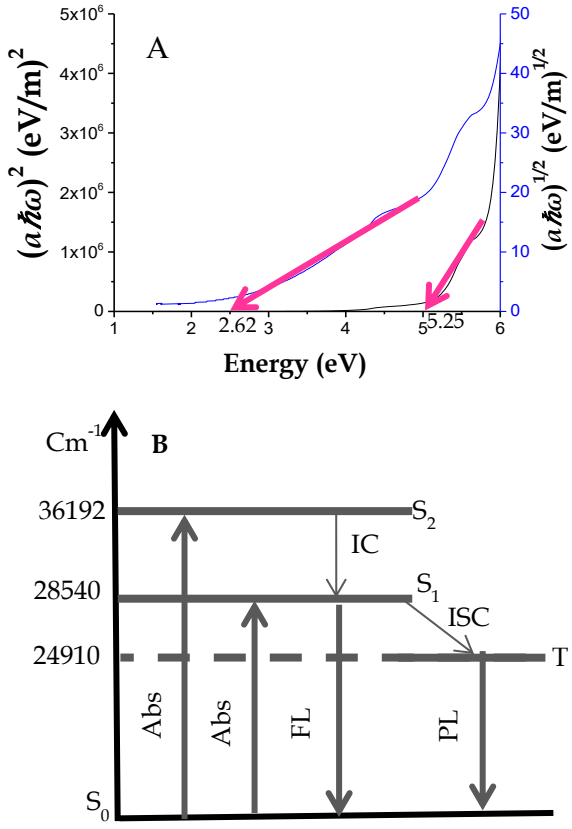


Figure 2. A) Energy gap for direct (black kurve) and indirect (blue kurve), B) jabloncki diagram

The inset in Figure 1A demonstrates the photographs of the C-dots dispersed in ethanol showing colorless and blue fluorescence under (a) without and with (b) UV lamp at 365 nm illumination, respectively[1]. From the Abs spectrum in Figure 1A (black line), the energy gap (E_g) of the as-prepared C-dots is evaluated as 3.26eV. The curve for direct (black line) and indirect (blue line) transitions was displayed in Figure 2A. The E_g value for indirect and direct transitions was determined as 2.62 and 5.15 eV, respectively and one can conclude that the E_g of the as-prepared C-dots is an indirect transition due to close to 3.26 eV. The E_g

value of the as-prepared C-dots is similar with the Eg of C-dots from Mringa oleifera [4]. According to Abs and PL spectra in Figure 1A, the energy of first (S_1) and second (S_2) excited single states was determined as 28540 and 36192 cm^{-1}. Whereas the energy of the first excited triplet state (T_1) is 24910 cm^{-1} as demonstrated in Figure 2B. Additionally, the photostability of these C-dots was investigated. These C-dots exhibited a long-term homogeneous phase without any noticeable precipitation at room temperature, and the blue fluorescence has no obvious change for long-time storage of 180 days[11]. These results showed that C-dots have excellent photostability, displaying they can be applied for sensor and bio-imaging in various electronic devices. Based on Figure 3A, one can obtain the relationship between λ_{em} (blue dots) and PL peaks (black dots) versus λ_{exc}. The bigger the λ_{exc}, the higher the λ_{em} and the lower the intensity of PL peaks from the C-dots.

The FTIR spectrum of C-dots from SPL was displayed in Figure 3B. This FTIR spectrum was used to identify the functional groups of the as-produced C-dots. As displayed in Figure 3B, the transmission peaks at 3319.37, 2971.77, and 1045.23 cm^{-1} in FTIR spectra were assigned to O-H stretching vibration, C–H, and C–O, respectively[6,11]. This O-H group demonstrates the presence of hydroxyl groups, which makes the C-dots hydrophilic and improves their stability and dispersibility in water. Moreover, the peaks at 1752.29, 1659.11, and 1378.85 cm^{-1} indicated the presence of the carbonyl group of C=O, C=C, and COO^-, respectively. The presence of O-H and C=O groups implies that these C-dots may have excellent water solubility and the surface of C-dots was coated by hydroxyl and carbonyl groups. The intensity of the C-O bond is highest indicating that C-dots had plenty of carbon and oxygen element-containing functional groups resulting in excellent water solubility[11,13].

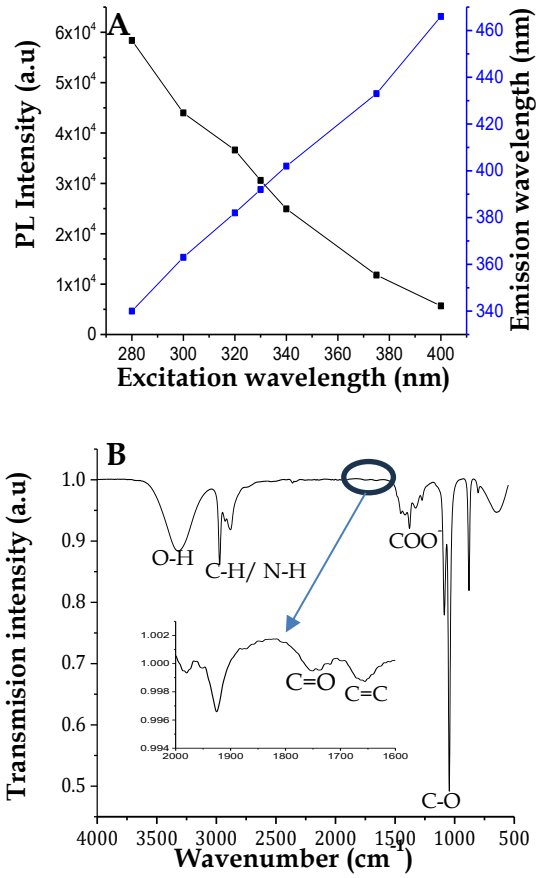


Figure 3 A. Emission wavelength (λ_{em}, blue dot) and PL intensity (black dot) versus excitation wavelength (λ_{exc}), B. FTIR spectrum of C-dots (inset: FTIR spectrum with wavelength number from 1500 to 2000 cm^{-1}

2. Quenching of PL intensity of C-dots and LOD value

Here, we investigate the quenching of PL intensity when the as-prepared C-dots were synthesized with Cu^{2+} ions. According to Figure 4A, the PL intensities of the as-derived C-dots at a peak of 340 nm quench when the concentration of Cu^{2+} ions increases. These results mean that the Cu^{2+} ions are a quencher to theese C-dots. The quenching of these PL intensities might be due to the reaction between Cu^{2+} ions and

C-dots through the O-H or C=O groups. The Cu^{2+} ions were bound by O-H or C=O groups from C-dots to form C-dots complex[13,33]. In order to confirm the interaction between these C-dots and Cu^{2+} ions, the FTIR spectrum of the as-derived C-dots after synthesis with Cu^{2+} ions was investigated. The transmittance intensity on O-H and C=O bonds of the C-dots complex is higher and broader than that of pure C-dots[22].

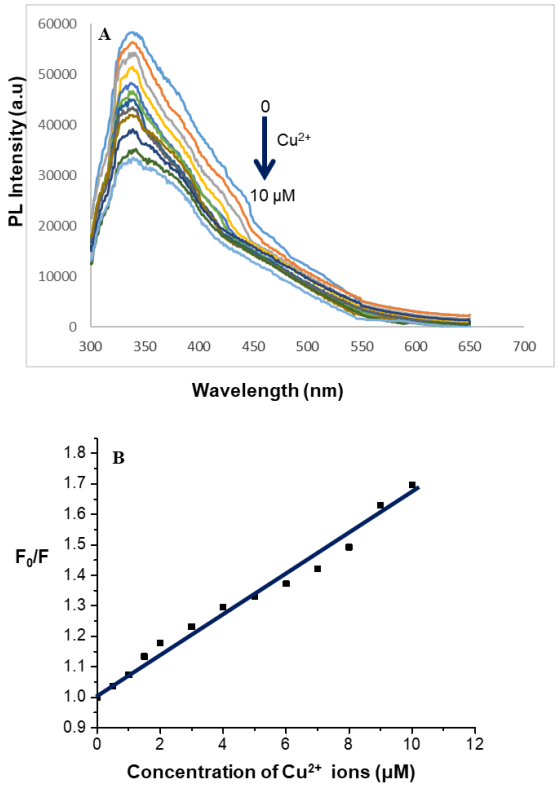


Figure 4. A. PL spectra of the C-dots in the presence of different Cu^{2+} concentrations, B. Plot of the values of F_0/F at a peak of 340 nm versus the concentration of Cu^{2+} ions

In this research, the quenching of PL intensity of the as-prepared C-dots after to be synthesized with Cu^{2+} ions was investigated. According to Figure 4A, the PL intensities of the as-derived C-dots at a peak of 340 nm quench when the concentration of Cu^{2+} ions increases. These results mean that the Cu^{2+} ions are a quencher to theese C-dots. The quenching of these PL intensities might be due to the reaction between Cu^{2+} ions and C-dots through the O-H or C=O groups. The Cu^{2+} ions were bound by O-H or C=O groups from C-dots to form C-dots complex[22]. The quenching of PL intensity of C-dots takes place via energy or electron transfer between the C-dots and Cu^{2+} ions and it demonstrates that C-dots can be used as sensors for detection of Cu^{2+} ions[34]. To investigate the sensing ability of the as-perpared C-dots, the value of LOD of Cu^{2+} ions is necessary to determine from the quench of PL intensity from these C-dots.

The quench of the PL intensity of the as-obtained C-dots as the increase of Cu^{2+} ions concentration can be fitted by using the Stern-Volmer equation, namely $F_0/F = 1 + K_{sv}[Q]$[11,13], where K_{sv} is the Stern-Volmer quenching constant (or slope of the graph from F_0/F versus $[Q]$, and $[Q]$ is the concentration of Cu^{2+} ions, F and F_0 are PL intensity in the presence and absence of different Cu^{2+} ions, respectively. Figure 4B exhibited the relationship between F_0/F and $[Q]$. According to Figure 2B, the linear relationship between F_0/F against $[Q]$ was obtained and it indicated the excellent sensing properties of the as-prepared C-dots. By using the Formulae[13], where σ^2 is the standard deviation, the value of LOD from Cu^{2+} ions was calculated as 2.85 µM, which is much lower than the maximum level (~20 µM) of Cu^{2+} ions in drinking water permitted by the US Environmental Protection Agency[28].

SUMMARY AND CONCLUSIONS

In summary, we have fabricated highly fluorescent C-dots from SPL as a precursor using the carbonization method and determined the LOD value from Cu^{2+} ions. The as-obtained C-dots emit a blue fluorescence and possess good solubitily in water and excellent photostability. The Abs peak at 276 nm confirmed the formation of C-dots and the PL peak at 401 nm corresponds to the blue fluorescence of the as-prepared C-dots. When the as-derived C-dots coordinated with Cu^{2+} ions, the PL intensity quenched with the increase of Cu^{2+} ion concentration. The value of LOD from

Cu^{2+} ions is 2.85 μM. The blue fluorescence, the excellent photostability, and the quench of the PL intensity from these C-dots can give several advantages for their application such as sensors, bioimaging, light emitting diode, and other optical electronics devices

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