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Facile Synthesis of Fluorescent Carbon Nanodots from Soursop Peel as a Carbon Source for Ferric Metal Ion Sensor

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ABSTRACT: In this work, we present a simple fabrication process of carbon nanodots (C-dots) from soursop peel as a carbon source. The aims of this research are to analyze the optical properties of the C-dots and to synthesize the C-dots as sensors for detecttion of ferric (Fe^{3+}) ions. The optical properties of the prepared C-dots were characterized by spectrophotometry of UV−Vis, photoluminescence, and FTIR. Whereas the synthesis process was conducted by blending with $FeCl_3H_2O$. The prepared C-dots emitted blue emission color. They had an absorption peak at 278 nm corresponding to the characteristic of $\pi \rightarrow \pi^*$ transition. When excited by an excitation wavelength of 340 nm, the PL peak at 430 nm corresponded to their blue emission color. Thus, the appearance of the O-H and C=O bonds in the FTIR spectrum demonstrated that the surface of C-dots was coated by hydroxyl and carbonyl groups, respectively. Interaction between C–dots and Fe^{3+} ion, investigated over quenching of the PL intensity of the C-dots after each addition of Fe^{3+} ion, demonstrated that the C-dots can be applied as a sensor for Fe^{3+} ion detection. The value of the limit of detection and limit of quantification from Fe^{3+} ion were determined to be 0.26 mM, These research results could, therefore, pave a large opportunity to explore C-dots from soursop peel for detection of Fe^{3+} ion in biosensing, in vivo bioimaging, biomedicine, light-emitting diodes, and so on.

Keywords: Synthesis, carbon nanodots, soursop peel, ferric metal, sensor

INTRODUCTION

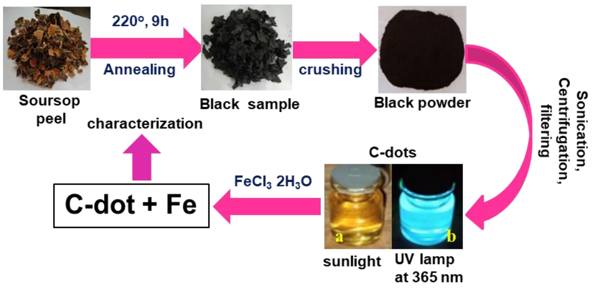
Carbon-nanodots (C-dots) have unique physical properties.[1] so they can be used as functional material in various electronic devices. The C-dots have been prepared from inorganic materials and have been applied as sensors, bioimaging, and other applications in several electronic devices such as light-emitting diodes, solar cell, and so on.[2] The C-dots from inorganic materials have, however, high toxicity[3] so they have limitations to being applied as functional material in various electronic devices[4–6] because they can cause environmental pollutants.[3] Fabrication of C-dots from organic materials is, therefore, one of the solutions to overcome the deficiency of C-dots from inorganic materials[4,7,8]due to organic materials having a low toxicity.[3,7,9,10] The C-dots include a new class of carbon nanomaterials with a size less than 10 nm[11,12] which were first accidentally discovered during purification of single-walled carbon nanotube (SWCNT) by Xiaoyou, et al. in 2004.[13] In the following two years, Sun, et al. found C-dots using the laser ablation method.[14] Generally, the carbon core of C-dots is predominant sp and an oxidized carbon surface with carbonyl and hydroxyl groups.[9,11,15]These two groups induce C-dots to have good solubility in water.[15] The other properties of C-dots are such as strong photoluminescence (PL) in the ultraviolet-visible and near-infrared area,[15–19] high photostability,[3,20,21] low toxicity,[3,7,9] favorable biocompatibility,[3,22] good solubility in water and resistance to photobleaching.[15] The strong PL of C-dots and high photostability benefit as a functional material for laser, sensor,[7,9,23,24] and bioimaging. [15–19] The other applications of C-dots are biological labeling,[12] photocatalysis,[20] and biomedicine.[23] When C-dots coordinated with metal ions, their PL intensities quenched. The quenching of these PL intensities takes place when the carboxylic (COOH), and amino (NH_2) groups on the surface of C-dots interacted with a specific metal through PL quenching.[24] Fortunately, the decreasing of this PL intensity can be used to find the lowest concentration from a material or an analytical blank,[15] which is called the limit of detection (LOD).[7,25]Nowadays, several types of research about the fabrication of C-dots from organic materials have been published. For instance, C-dots were fabricated from lemon juice,[1] pomelo peel,[7] watermelon peel,[26] garlic from China,[27] apple juice,[28] orange juice,[29] banana juice[30] from India, soursop juice,[8] rice powder,[31] and rice husk[32] from Indonesia. The C-dots with strong PL intensity can be applied as a sensor to find the LOD value of metal ions. So far, there are several recent kinds of research about the interaction between C-dots and metal ions, especially ferric (III) ions.[7,33–37] For example Ruhong, et al. demonstrated C-dots from ammonium fluoride and o-phenylenediamine as carbon sources to determine the selectivity and sensitivity to the Ferric (III) ion. Fortunately, the C-dots from ammonium fluoride and o-phenylenediamine emit red emission color.[33] Shuai, et al. exhibited fluorine and nitrogen co-doped carbon dots (FNCDs) to evaluate the selectivity and LOD value of Ferric (III) ions.[35] Chen, et al. used glutamic acid and ethylenediamine as carbon sources to investigate the selectivity and LOD value of ferric (III) ions.[36] Zulfajri, et al. prepared C-dots from cranberry beans by using hydrothermal method. These C-dots emit blue emission color and have been used to investigate the selectivity and LOD value of ferric (III) ions.[37] Durga, et al. fabricated C-dots from ethylene diamine. These C-dots were applied to the selectivity and LOD value of ferric (III) ions. Thus, the quantum yield (QY) of these C-dots is 64%.[34] Thus, there are several researches of C-dots blending with other metal, for example Wenbo, et al. presented a green synthesis of highly fluorescent C-dots from pomelo peel and had been used it as a sensor to find the LOD of mercury (Hg^{2+}) ions. They obtained that C-dots from pomelo peel emit blue emission color.[7] Zhou et al. demonstrated a facile approach for the synthesis of C-dots using watermelon peel as a carbon source with a QY of 7.1% and these C-dots had been applied as bioimaging. Theese C-dots emit blue emission color.[26] In our laboratory, Ngara, et al. demonstrated the preparation of C-dots from soursop juice using the microwave method[8] and rice powder using the hydrothermal method.[31] We obtained high fluorescent C-dots with blue emission color.[8,31] According to the above-mentioned research results, the fabrication and development of a simple, economical, and green preparative method to realize highly fluorescent C-dots are highly desired.[7]

Several heavy metal ions such as ferric (Fe^{3+}), Zink (Zn^{2+}), Hg^{2+} and Copper (Cu^{2+}) have put a serious danger to the environment, human health, plants, and animals.[7,12]The Fe^{3+} ion is one of the most abundant significant transition metal ions which play an essential role in environmental and biological systems.[2,33,38] In the human body, Fe^{3+} ions mostly accumulate within the liver, spleen, and bone marrow cells.[12] If the concentration of Fe^{3+} ion in the human body and/or in the environment is high, it can cause several diseases such as Alzheimer's, diabetes, hemochromatosis [2,12,38,39], and/or environment pollutant.[7,12,38] Whereas the deficiency of its concentration can induce anemia disease.[12] Therefore, the concentration measurement of Fe^{3+} and/or other metal ions both in the human body and in the environment is needed to be efficiently and effectively conducted for healthcare concerns.[33]

In this work, we present the use of low-cost waste of soursop peel from Kupang regency, East Nusa Tenggara Province, Indonesia. In this case, C-dots were fabricated from soursop peel as natural plant materials as carbon source (precursors) using the annealing method. Soursop fruit (*Annona muricata L*., family Annonaceae) is a delicious fruit grown in almost all tropical and subtropical regions of the world including Kupang regency, East Nusa Tenggara Province, Indonesia. To date, the soursop fruit was only used to make juice as a fresh drink or was eaten directly by humans. Whereas the soursop peel is only generated as a by-product/waste because it has no economic value so it was discarded as waste material. However, If the soursop peel could be extracted by a certain method, one can derive a new material that has economic value. Therefore, the aim of our work is to convert soursop peel into added material. In this case, soursop peel was converted into highly fluorescent C-dots and to be applied as a sensor for Fe^{3+} ion. Briefly, the soursop peel will be annealed at a low temperature following sonication, centrifugation and filtering process to obtain fluorescent C-dots. Furthermore, C-dots were blended with Fe^{3+} ion to determine the value of LOD and the limit of quantification (LOQ) of Fe^{3+} ion.

Based on our knowledge, this is the first time, the C-dots were fabricated from soursop peel using the annealing method. Under illumination with a UV lamp at 365 nm, the prepared C-dots emitted blue emission color, and their absorption (Abs) peak at 278 nm corresponding to the $\pi \rightarrow \pi^*$ transition. Fortunately, by using an excitation wavelength of 340 nm, the PL peak at 430 nm corresponds to their blue emission color. Interestingly, by increasing the excitation wavelength (λ_{exc}) from 320 to 400 nm, their PL peaks shifted to a longer wavelength from 372 to 460 nm. After C-dots were blended with Fe^{3+} ion, the PL intensity of C-dots quenched and the value of LOD and LOQ for Fe^{3+} ion is 0.26 and 0..78 mM, respectively.

RESULTS AND DISCUSSION



Schema 1. The schematic illustration of simple preparation to realize C-dots from soursop peel and synthesis of C-dots with Fe^{3+} metal ion

Scheme 1 displays the schematic illustration of simple preparation to produce highly fluorescent C-dots from soursop peel and synthesis of C-dots with Fe^{3+} metal ion. The washed soursop peels were annealed at 220°C for 9 hours to realize a black sample. The black color of these samples demonstrated that the carbon source for the generation of C-dots has been formed[1]. These black samples were crushed with a blender to form a black powder. Subsequently, the highly fluorescent C-dots can be obtained by the following sonication, centrifugation, and filtering processes. By illumination with a UV lamp at 365 nm, the obtained C-dots emitted blue fluorescence color as displayed in Scheme 1. This blue color confirmed the presence of fluorescent C-dots and in agreement with previous results.[7,8,12,26,29,31] Furthermore, the prepared C-dots were employed as the sensor for Fe^{3+} ions through evaluating the value of LOD and LOQ of Fe^{3+} ions.

The Abs and PL spectra of the C-dots dispersion in ethanol were investigated by spectrophotometry of UV-Vis and model SHIMADZU RF-6000, respectively. Figure 1 exhibits the Abs (black line) and PL (blue line) spectra of the as-prepared C-dots dispersed in ethanol solution. According to Figure 1, the Abs range of these C-dots is from 250 to 420 nm with an Abs peak at 278 nm corresponding to the $\pi \rightarrow \pi^*$ transition of the C=C bond in the heterocyclic ring,[2,10] which was similar to the Abs area of the C-dots made from pomelo peel, soursop juice, rose-heart radish, and aloe.[7–9,40] This Abs spectrum confirmed that the formation of C-dots from soursop peel has taken place. By direct excitation of the wavelength of 340 nm, the peak of PL intensity from these C-dots is at 430 nm (Figure 1, blue line).

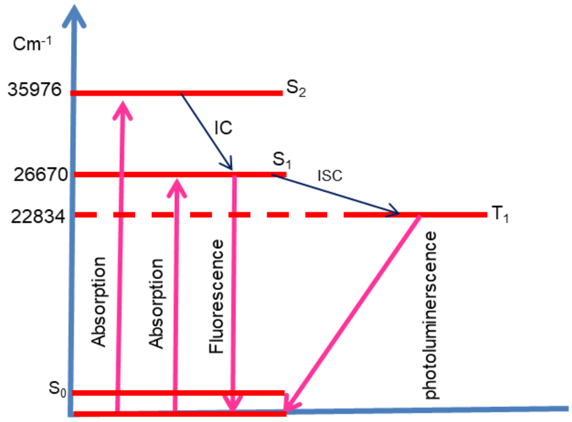


Figure 1 Abs (black line) and PL (blue line) spectra of the C-dots dispersion in ethanol. Inset: the photographs of the C-dots dispersion in ethanol with a) sunlight and (b) UV at 365 nm illumination

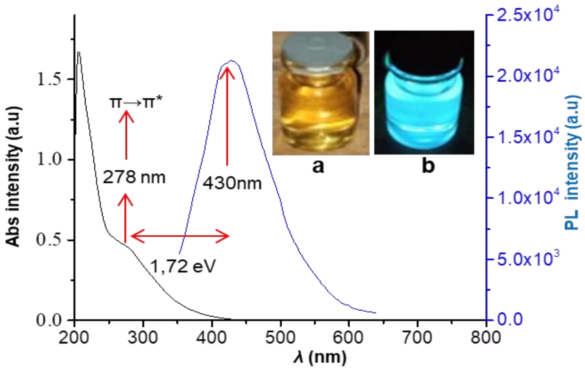


Figure 2. Jablonski diagram of C-dots. IC; internal conversion, ISC: intersystem crossing

The photographs of the C-dots dispersed in ethanol show colorless and strong blue emission color under without (a) and with (b) UV lamp at 365 nm illumination, respectively (Figure 1, inset). The blue emission color of C-dots indicated that C-dots had blue fluorescent color. Based on the Abs and PL peaks of these C-dots, the value of the Stokes shift is 1.72 eV. From the Abs spectrum of C-dots (Figure 1, black line), the energy gap (E_g) of C-dots is at 3.3 eV. To further investigate whether direct or indirect the energy gap from C-dots, the optical absorption coefficient (α) near the absorption edge for direct and indirect transitions is given by equation[41] $(\alpha\hbar\omega)^2 = A_d\left(\hbar\omega - E_g\right)$ and $(\alpha\hbar\omega)^{1/2} = A_i\left(\hbar\omega - E_g\right)$, respectively, where A_d and A_i are the absorptions constant for a direct and indirect transition, respectively. The graph of the energy gap for direct (black line) tand indirect (blue line) transitions was displayed in Figure S1. The energy gap value for direct and indirect transitions was determined to be 2.65 and 4.85 eV, respectively. From these values, one can conclude that the energy gap of C-dots is an indirect transition due to close to 3.3eV. The Jablonski diagram of the C-dot from the soursop peel was displayed in Figure 2. Broadly speaking, the Jablonski diagram can be evaluated by using Abs, fluorescence (FL), and PL spectra of a material. According to Figure 1, the energy of first (S_1) and second (S_2) excited single states was evaluated to be 26670 and 35976 cm^{-1}. Whereas the energy of the excited triplet state (T_1) is 22834 cm^{-1} as displayed in Figure 2.

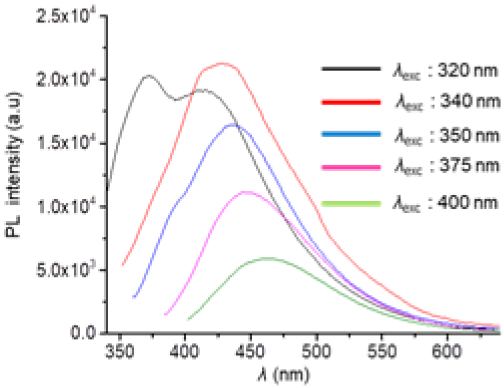


Figure 3. PL spectra of the C-dots dispersion in ethanol depending on λ_{exc}

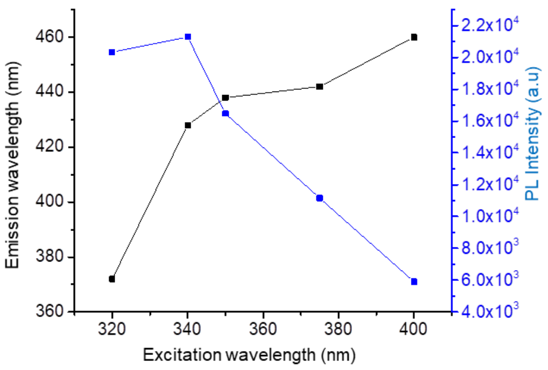


Figure 4. Emission wavelength (black dot) and PL intensity (blue dot) of C-dots versus excitation wavelength

Similar to the common C-dots, the PL peak positions of our C-dots depend on excitation wavelength (λ_{exc}). As the increase of λ_{exc}, the PL peaks shifted to longer wavelengths, accompanied by the decrease of PL intensity as demonstrated in Figure 3. When the λ_{exc} is varied from 320 to 400 nm, the PL peaks as emission wavelength (λ_{em}) shifted from 372 to 460 nm as shown in Figure 3 and Figure S2. These results indicate that λ_{em} can be tuned by just controlling λ_{exc} without changing the C-dots.[7,12]Thus, the PL intensity of the C-dots is strongly dependent on the λ_{exc}. When the λ_{exc} is 320, two PL peaks at 372 and 412 nm appear in the PL spectrum. Whereas the use of other λ_{exc} only produces one PL peak in the PL spectra (Figure 3). These results are in agreement with previous results.[10,17] Additionally, the photostability of C-dots was investigated. Remarkably, when C-dots dispersion in ethanol was illuminated with a UV lamp at 365 nm in the air at room temperature, the blue emission color intensity did not change significantly and no observation of aggregated nanoparticles at the beginning (a), after one month (b), after three months (c), and after six months (d) as displayed in Figure S3. The high photostability and easy dispersion in several solutions such as ethanol, aquadest, and dichloromethane of C-dots were affected by their small particle sizes.[42] These results indicated that C-dots had an excellent photostability, demonstrating that they are promising for sensor and bio-imaging in various electronic devices. From Figure 3, one can obtain the relationship between λ_{em} versus λ_{exc} (black dot) and PL intensity (blue dots) versus λ_{exc} as exhibited in Figure 4. The longer the λ_{exc}, the higher the λ_{em} and the lower the PL intensity of the C-dots.

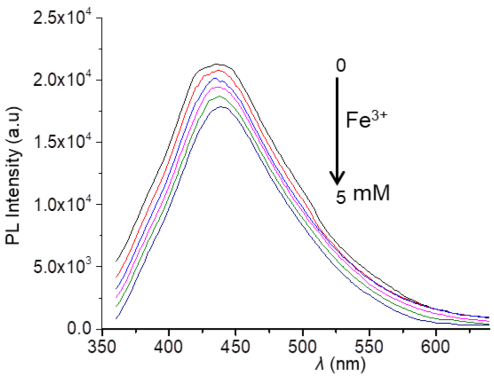


Figure 5. FTIR spectrum of the C-dots. Inset exhibits molecule structure of C-dots

Figure 5 shows the FTIR spectrum of the C-dots. Inset in Figure 5 displays the molecule structure of C-dots.[11] The peak at 3332.39 cm^{-1} is attributed to the O-H stretching vibration of the phenolic group and N-H, while several small bands from 2871.49 to 2968.88 and 878.42 cm^{-1} could be identified as the stretching vibrations of C-H bonds. The peaks at 1373-1402 cm^{-1} exhibited the symmetric existence of COO−. Moreover, the peaks at 1630.52 cm^{-1} and 1263.15 cm^{-1} indicated the presence of the carbonyl group of C=O and C-O-C stretching vibrations of aromatic functional groups.[12] The band at 1603 cm^{-1} was attributed to the stretching vibration of C=C. The broad band around 1035-1040 cm^{-1} was ascribed to the bending vibrations displaying C-O. The characteristic band of C−OH stretching was at 1305.15 cm^{-1}.[7,9] The intensity of the C-O bond in the FTIR spectrum from C-dots is highest indicating that C-dots had plenty of carbon and oxygen element containing functional groups resulting in excellent water solubility.[12,24] The appearance of O-H and C=O groups in the FTIR spectrum of the C-dots demonstrates that the surface of the C-dots was coated by hydroxyl and carbonyl groups, respectively as displayed in the molecule structure of C-dots. The hydroxyl and carbonyl moieties may originate from carbohydrates in the soursop peel.[8] When these C-dots coordinated with Fe^{3+} ion, the transmittance intensity of C-dots + Fe (red curve) at hydroxyl and carbonyl groups is higher than that of pure C-dots (black curve) as demonstrated in Figure S4. This result means that

Fe^{3+} coordinated with C-dots through carbonyl and/or hydroxyl groups.

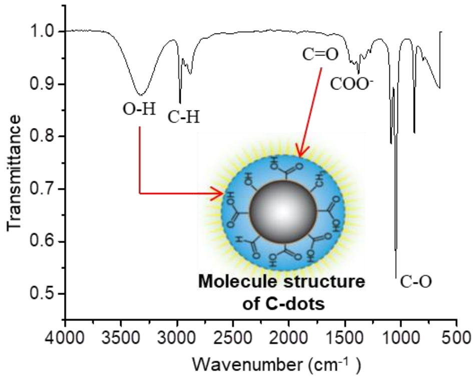


Figure 6. PL spectra of the C-dots in the presence of different Fe^{3+} concentration from top to bottom: 0, 1, 2, 3, 4, and 5 mM (excitation wavelength at 340 nm).

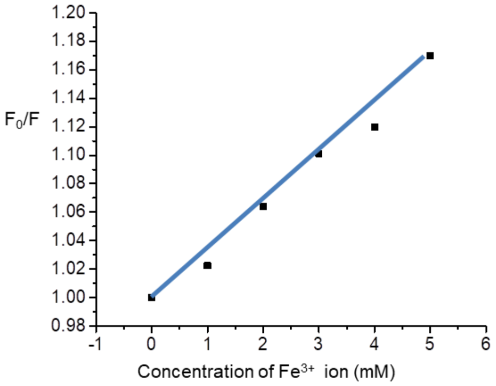


Figure 7. Plot of the values of F_0/F at 430 nm versus the concentration of Fe^{3+} ions.

Broadly speaking, the surface of C-dots has many hydroxyl and carbonyl groups so C-dots can bind metal ions to form a C-dots complex.[2,24,43] In this research, the C-dots coordinate with the Fe^{3+} ion in order to evaluate the LOD and LOQ values of the Fe^{3+} ion. Figure 6 demonstrated the PL spectra of C-dots blending with Fe^{3+} ions. Thus, the excitation wavelength of 340 nm was chosen to investigate the quenching of PL intensity from C-dots because the Abs area of the C-dots is from 250 to 400 nm (Figure 1)[44,45] as well as highest PL intensity of C-dots occurs on excitation wavelength of 340 nm. When excited by an excitation wavelength (λ_{exc}) at 340 nm, the PL intensities of the C-dots in the blue emission at 430 nm quenched with the increase of the Fe^{3+} ion concentration. These results mean that Fe^{3+} metal ion as a quencher to the C-dots. The quenching of these PL intensities might be due to the coordination reaction between Fe^{3+} ions and phenolic hydroxyl groups and/or carbonyl groups present on the surface edge of the C-dots.[2,12,46] This means that the ability of Fe^{3+} ions to quench the PL intensity of the C-dots is most probably via electron or energy transfer between the C-dots and Fe^{3+} ion. [7,38,43] Interestingly, the PL intensity peaks of the C-dots showed noticeable changes for Fe^{3+} ions at 430 nm. Therefore, these C-dots can be used as sensors for Fe^{3+} ion through the determination of the LOD and LOQ values of Fe^{3+} ions.

The quench of the PL intensity can be fitted by using the Stern-Volmer equation, $F_0/F = 1 + K_{sv}[Q]$, [7,12] where K_{sv} is the Stern-Volmer quenching constant and $[Q]$ is the concentration of Fe^{3+} ions, F and F_0 are PL intensity in presence and absence of different Fe^{3+} ions, respectively. The relationship between the quenching ratio of PL intensity before and after the addition of Fe^{3+} ion (F_0/F) was displayed in Figure 7. Based on Figure 7, the linear relationship between PL intensity against the concentration of Fe^{3+} ion pervades two ranges of 0-2 mM and 2-6 m, respectively. The linearity of the Fo/F curve to the Fe^{3+} ion concentration in the range of 0-5 mM indicated the excellent sensing properties of C-dots in the detection of Fe^{3+} ion trace. By using the formulae $LOD = 3.3\sigma^2 / S$ and $LOQ = 3LOD$,[25] where σ^2, and S are standard deviation, the slope of the calibration graph, respectively, the value of LOD and LOQ from Fe^{3+} ion in the range of 0-5 mM can be calculated, that is 0.26 and 0.78 mM, respectively. The detection of Fe^{3+} ions through a visible fluorescent method would be of considerable benefit.[43]

CONCLUSION

In summary, we have demonstrated a simple method to prepare highly fluorescent C-dots using soursop peel as the carbon source and investigated their application to find the LOD and LOQ values of Fe^{3+} ion. These C-dots emitted blue emission colour and had an excellent photostability after storage for six months. The blue emission and the Abs peak at 278 nm confirmed that the formation of C-dots from soursop peel has been occurred as well as the PL peak at 430 nm corresponds to the blue emission colour of the C-dots. When the C-dots coordinated with Fe^{3+} ions, the PL intensity quenched with the increase of Fe^{3+} ion concentration. The value of LOD and LOQ from Fe^{3+} ion is 0.26 and 0.78 mM, respectively. The blue emission colour, the excellent photostability, and the quench of the PL intensity from these C-dots can give several advantages for the application for sensors, bioimaging, drug delivery, and so forth.

Experimental Section

MATERIALS AND APPARATUS

Soursop fruits were purchased from the traditional market in Kupang regency, East Nusa Tenggara Province, Indonesia. The chemical materials such as ethanol, methanol, ferric chloride dehydrate ($FeCl_3.2H_2O$), 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 irradiated with a UV lamp at 365 nm. The Abs and PL spectra of C-dots were measured 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. The morphology surface of C-dots was investigated by Scanning electron microscopy (SEM).

FABRICATION OF C-DOTS FROM SOURSOP PEEL

The preparation of C-dots from soursop peel was followed according to Reference[25] with a small modification. The C-dots from the soursop peel were fabricated by the annealing method using the furnace. In a typical fabrication, soursop fruit was washed several times under water to remove dirt and other sediments present in the soursop peel. Then, the peel of the washed soursop fruit was pared and dried in sunlight for 8h to remove the moisture content. A 60 g from dried soursop peel was annealed at 220^{o}C for 9h to get a black sample. After cooling down to the room temperature, this black sample was crushed by a blender to realize black powder. A 0.5 g from the black powder was added to 3 mL ethanol, followed by an ultrasonic process for an hour to obtain a homogeneous dark solution. Furthermore, a 10 mL of ethanol was added again to this sample for the centrifugation process at 1000 rpm for 30 minutes. After the centrifugation process, the sample was filtered to obtain the resultant supernatant containing fluorescent C-dots, followed by the illumination of a UV lamp at 365 nm to observe the fluorescent color of the C-dots. Furthermore, the spectra of Abs, FL, FTIR spectra, and morphology surface of the C-dots were measured.

SYNTHESIS OF C-DOTS WITH FERRIC METAL

The detection of Fe^{3+} ion was performed at room temperature. Briefly, A 500µL of C-dots dispersion was added into 1 mL of PBS (1M, pH 7.0), followed by the addition of Fe^{3+} ion (50µL) with various concentrations (2-6 mM). Furthermore, the PL, FTIR spectra, and morphology surface of these samples were investigated by model SHIMADZU RF-6000, a JASCO model FT/IR-4200 Fourier transform infrared spectrophotometer, and SEM, respectively

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