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The hydrothermal effect of time and temperature on the synthesis of carbon dots (CDs) from chicken feathers

E Hastuti*, C Salsadilla, AY Sari, U Hikmah

Department of Physics, Universitas Islam Negeri Maulana Malik Ibrahim Malang Jl Gajayana 50 Malang, Indonesia

*E-mail : erna@fis.uin-malang.ac.id

Abstract. Carbon dots (CDs) are carbon-based nanoparticles with a predominantly zero-dimensional structure and a diameter of typically less than 10 nm. CDs are desirable due to their high stability, low toxicity, high solubility in water, and accessible synthesis methods. The hydrothermal method is extensively used in the synthesis of CDs because it is simple and environmentally friendly, and particle size and shape can be controlled by initial materials and various hydrothermal conditions. This research involved the synthesis of CDs from poultry chicken feathers with hydrothermal time and temperature variations of 160 °C, 180 °C, and 200 °C, and treatment durations of 5 and 7 hours. The results indicate that the yield of CDs increased with the higher hydrothermal temperatures. The C-dots emitted a blue light when stimulated by ultraviolet light, and the band gap values of the CDs ranged from 3.45 eV to 3.28 eV.

1. Introduction

Carbon dots (CDs) are carbon structures that are zero-dimensional and mostly consist of carbon atoms. These structures possess diameters that are smaller than 10 nm. CDs are often produced by bonding carbon atoms with neighboring carbon atoms, such as oxygen or other heteroatoms, operating sp^2 or sp^3 hybridization [1]. CDs are a type of nanomaterials with a variety of potential structures and varying optical properties that possess the ability to fluoresce or emit light. The shape is generally quasi-spherical, and the structure can be amorphous, graphitic, or defined by a C_3N_4 crystalline core [2]. Extensive research has demonstrated that CDs are chemically inert and exhibit non-toxic characteristics [3]. Moreover, CDs display approving fluorescence qualities, making them suitable for photocatalytic, optoelectronics, nanomedicine, sensing, and bioimaging applications [4].

Biomass waste has the potential to function as a feasible source of carbon dots [5]. Biomass waste affects a characteristic carbonaceous resource comprising cellulose, hemicellulose, lignin, protein, keratin, and others. A typical example of biomass waste is created from plants that have 30-60% cellulose, 20-40% hemicellulose, and 15-25% lignin. Biomass waste is an environmentally friendly and sustainable carbon source that is easily obtainable, making it a feasible choice to produce CDs without causing adverse significances. However, the current practices for managing biomass waste primarily consist of disposal by dumping, landfilling, or open burning. These techniques lead to the loss of essential resources and give rise to several environmental concerns that found a substantial risk to human well-being [6]. The utilization of chicken feathers as a biomass waste material with the potential to be utilized as a viable source for carbon dots has been established. Chicken feathers are primarily composed of approximately 91% protein, predominantly in the form of keratin. Additionally, it contains approximately 1% fat and 8% water. Keratin is comprised of many constituents, encompassing carbon

(51%), nitrogen (17%), oxygen (21%), hydrogen (6%), and sulphur (5%), alongside small quantities of iron (Fe), magnesium (Mg), and other elements [7]. The potential utilization of biomass waste derived from feathers has been explored as an attractive option to produce carbon dots.

There are several methods for the production of CDs, including chemical exfoliation, laser-based methods, ultrasonic, microwave, solvothermal, carbonization, and hydrothermal methods [8]. The hydrothermal method is widely used in CDs because it is environmentally friendly and easy to make. The particle size and shape can be controlled using starting materials and hydrothermal conditions. The hydrothermal method is heating in a closed system with high pressure and low temperature (< 300°C) using water as a medium.

The temperature used in the hydrothermal process affects the particle diameter and crystal size, whereas if the synthesis temperature increases, the size of the crystals formed and the particle diameter will become smaller [9]. The temperature of the hydrothermal process also shows an influence on homogeneity and particle size. The crystallinity of the material increased with increasing hydrothermal reaction time due to the creation of a larger particle size. Longer reaction times allow more ions to diffuse and generate bigger nanoparticles [10].

In this research, carbon dots (CDs) were made from chicken feather waste using the hydrothermal method with variations in time and temperature. CDs from chicken feathers were characterized using FTIR (Fourier Transform Infrared), which was used for qualitative functional group analysis, UV-Vis (Ultraviolet-Visible) spectrophotometer to measure the absorbance wavelength and investigate the energy gap of the CDs samples, UV light was used to determine the colour of the fluorescence of the CDs samples used in this study. The carbon dot (CDs) material is then applied as a photocatalyst material, namely the degradation process of methylene blue dye.

2. Experimental

2.1. Preparation of activated carbon derived from chicken feather

Synthesis of CDs material from chicken feathers using the hydrothermal method. Clean chicken feathers are ground and sifted using a 50-mesh sieve. 1 gram chicken feather powder mixed with 60ml distilled water. After mixing, the solution was put into a Teflon line autoclave and processed with variations in time and temperature of 160°C, 180°C, and 200°C for 5 and 7 hours. The hydrothermal reactor is left at room temperature. The samples obtained were then centrifuged and filtered using a 0.22 μm membrane.

2.2. Characterization

The surface functional groups and structure of CDs were investigated using Fourier Transform Infrared Spectroscopy (FTIR) (FT 1000 Varian). The measurements were conducted with a resolution of 2 cm^{-1}, and a wavenumber range from 400 to 4000 cm^{-1} to obtain spectra lines. Investigation of the optical character of CDs was analysed using a UV-Vis spectrophotometer (UV-Vis Cary50 Conc Varian) and UV light (395 nm). The Methylene Blue (MB) degradation test process was carried out using a simple UV-LED-based light photocatalytic test reactor and the decrease in concentration was measured every 15 minutes.

3. Result and Discussion

In this research, CDs were made from chicken feathers using the hydrothermal method with variations in time and temperature. The percentage yield of CDs samples produced is influenced by variations in time and hydrothermal temperature. The yield value produced is directly proportional to both the duration and the temperature employed. According to the data presented in Figure 1, it can be observed that the sample exhibiting the lowest yield value corresponds to the hydrothermal treatment conducted at a temperature of 160°C for 5 hours, resulting in a yield of 39%. Conversely, the sample demonstrating the highest yield value is associated with the hydrothermal treatment performed at a temperature of 200°C for 7 hours, yielding 57%. Dissolution happens early in the hydrothermal process and increases with time and temperature employed. The number of CDs yield produced is determined by hydrothermal time and temperature [11].

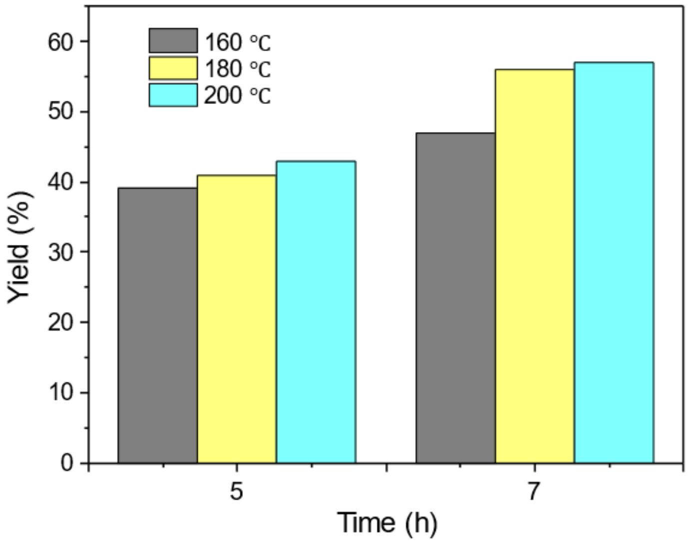

Figure 1. Yield percentage of the obtained carbon dots

Fourier Transform Infrared Spectroscopy (FTIR) data analysis was used to show the functional groups of the CDs samples. Figure 2 shows that chicken feathers resulting from hydrothermal processes contain the functional groups O-H, C≡N, C=O, C–N, C=C, C–O–C, and C–H. The hydrothermal process causes the appearance of the C–N, C=C, C–O–C functional groups and changes in the intensity of the higher absorption of the C=O functional groups, which indicates that the chicken feathers have decomposed.

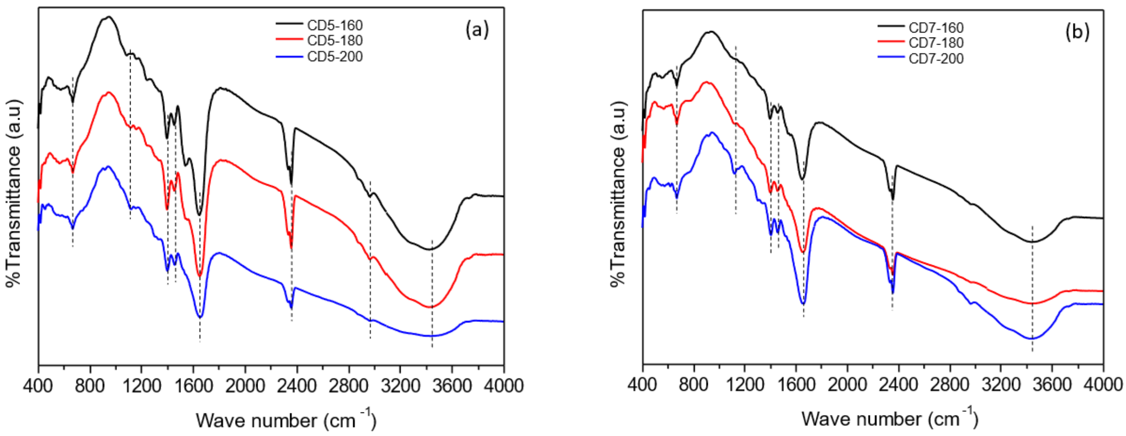

Figure 2. FTIR spectra of carbon dots with hydrothermal time (a) 5 h; and (b) 7 h

All samples have functional groups that accordance with the properties of CDs. CDs contain functional groups such as C=C, O-H, C-N, C-H, C=O, and C-O-C [12], [4]. The presence of the O-H functional group in CDs suggests the continued presence of water molecules. Carbon vibrations can be identified by the C=C functional group, which constitutes the central structure of CDs. The carbonyl (C=O) and C-H stretching functional groups a common functional group found on CDs [13].

The presence of the C-N functional group indicates that the CDs sample contains nitrogen which can influence the band gap energy [14]. The existence of the C-O functional group in the sample suggests the existence of carbon and oxygen bonds, which have the potential to impact the surface structure of the CDs. The C=N functional group shows that the sample includes nitrogen, which is one of the keratin components.

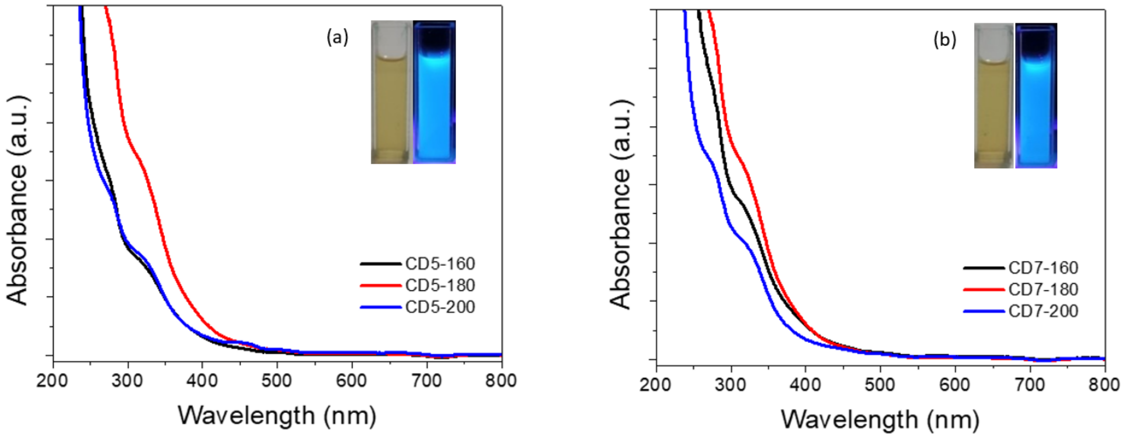

Figure 3. UV-Vis spectra and photoluminescence of carbon dots with hydrothermal time (a) 5 h; and (b) 7 h

The UV-Vis spectroscopy spectra (Fig. 3) revealed that the CDs samples exhibited absorbance, characterized by absorption peaks within the wavelength range of 276-321 nm. The absorption peaks within the wavelength range of 220-270 nm are attributed to the π-π* transition of the C=C and C=N bonds. The absorption peak occurs within the wavelength range of 280-350 nm, corresponding to the n-π* transition of the C-O and C=O bonds [15]. The UV-Vis test analysis reveals the existence of C=C, C=N, C-O, and C=O bonds, which aligns with the functional groups identified through FTIR spectra.

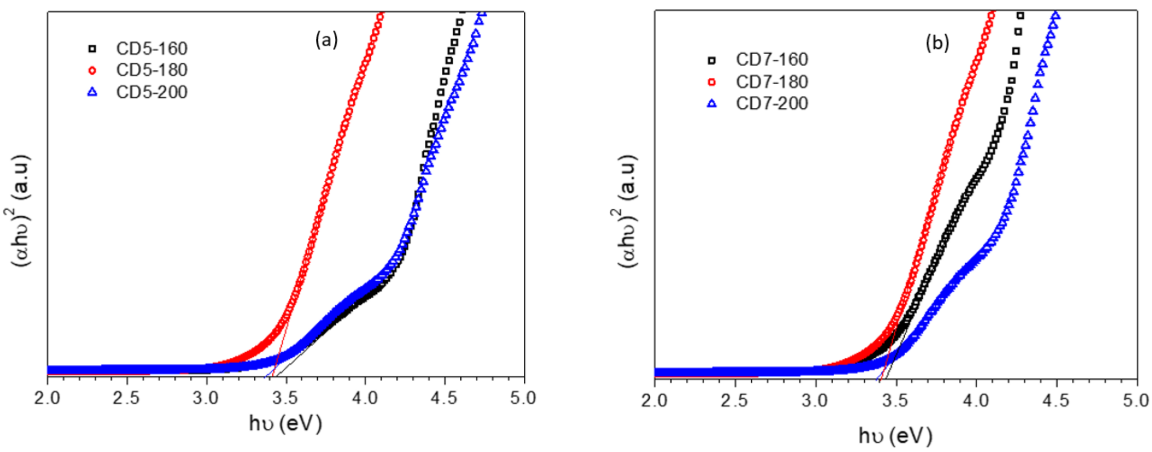

Figure 4. Band gap energy of carbon dots with hydrothermal time (a) 5 h; and (b) 7 h

Deeper analysis to determine the gap energy of the resulting CDs was calculated using the Tauc plot method. In Figure 4, all samples show gap energy values that are not much different, with a range of 3.45-3.28 eV. The CD7-200 sample had the lowest gap energy of 3.28 eV, whereas the CD5-160 sample had the highest gap energy of 3.45 eV. The gap energy of CDs is influenced by hydrothermal activities.

As the hydrothermal temperature increases in duration and intensity, there is a corresponding decrease in the band gap energy. This band gap energy greatly influences its ability to photocatalytic degradation.

The CDs material is designed to perform as a catalyst in the methylene blue (MB) photodegradation process as shown in Figure 5. The sample for the photocatalysis procedure consists of 47.5 ml of 5 ppm MB and 2.5 ml of CDs solution. The photocatalytic activity of CDs was evaluated in two distinct phases, specifically under conditions of darkness and light using UV light. The efficiency results of MB show without CDs that in the dark condition has an efficiency of 2.89%, and increased with the light condition. CDs samples with different hydrothermal times and temperatures exhibited variances in the degradation process with an efficiency of 7.43%-12.30%. The highest efficiency was shown in the CD7-200 sample. This aligns with the greater concentration of CDs (from yield data) and the most negligible gap energy (3.28 eV). This proves that samples in light conditions are better at degrading MB because of the presence of light energy, which can move electrons from the valence band to the conduction band.

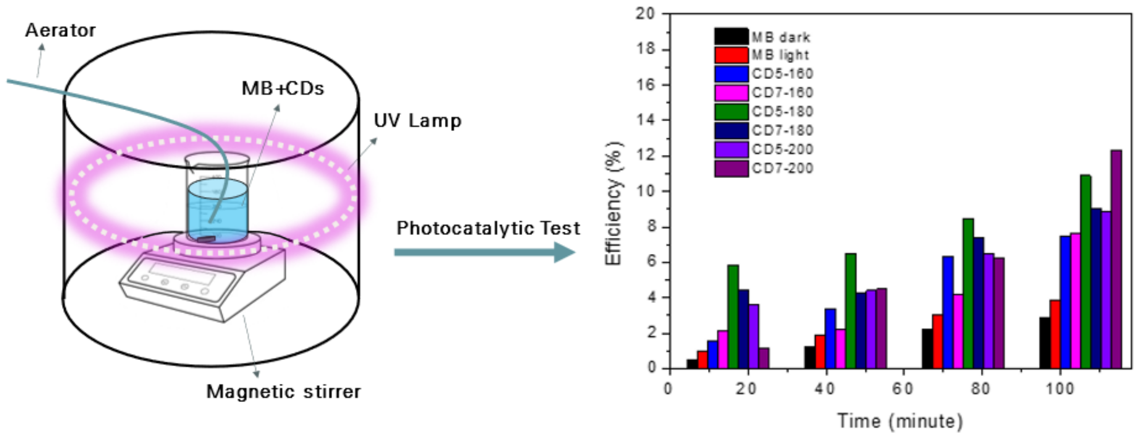

Figure 5. A Photocatalytic system with carbon dots and efficiency degradation of methylene blue

4. Conclusion

In this research, carbon dots (CDs) were successfully made from chicken feathers using the hydrothermal method. Hydrothermal time and temperature affect the sample yield value with the highest yield being 57% at CD7-200 sample. FTIR spectra reveal that all samples have C=C, O-H, C-N, C-H, C=O, and C-O-C functional groups and exhibited blue luminescence when irradiated by UV light. The results of bandgap analysis show a decrease in gap energy to 3.28 eV in the CD7-200 sample. Photodegradation test results show that the addition of CDs can reduce MB concentration with an efficiency of up to 12%.

Acknowledgment

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