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RESEARCH ARTICLE

ONE-POT SYNTHESIS OF CARBON QUANTUM DOTS AND THEIR APPLICATION AS A FLUORESCENT INKS

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ABSTRACT

A facile, low cost, and one-pot hydrothermal reaction method is utilized to synthesized highly stable and durable carbon quantum dots (CQDs) by using *laurus nobilis* leaves as a carbon source. *Laurus nobilis* leaves were subjected to hydrothermal reaction at 175 °C for 10 h. The color of obtained CQDs under UV-light is bright blue fluorescence. The excitation dependent fluorescent emission of the prepared CQDs was observed and the obtained CQDs gives maximum emission at 425 nm when excited at 344 nm. The absorption peak of the CQDs is located at 279 nm. Furthermore, the synthesized CQDs can be consumed as a fluorescent ink for security, encryption and information storage applications. Combining with good stability and water solubility, unique fluorescence properties and its low-cost, CQDs can also be used as a next generation fluorescent ink alternative to traditional fluorescent ink for anti-counterfeiting.

Keywords: Carbon Dots, Fluorescent ink, XRD, Laurus nobilis

1. INTRODUCTION

Carbon dots (CDs) were accidentally discovered by purification of Single Wall Carbon Nanotubes (SWCNTs) in 2004. Carbon quantum dots or simple carbon dots have low toxicity and they can dissolve very well in water [1]. In addition, it has attracted great attention because of its stability, photoluminescence feature, biological compatibility and environmental friendly nature [2]. Moreover, CDs are a serious alternative to traditional fluorescent dyes and toxic semiconductor quantum dots due to their many properties such as excellent photoluminescence properties, photostability, adjustable excitation and emission wavelength, non-flashing emission [3]. The low synthesis cost of CDs lead to enhancement usage of it in different applications [4]. It is used in many application areas such as bioimaging [5], detection of ions [6, 7], light-emitting diodes [8, 9], drug delivery [10], self-healing fillers [11], photothermal therapy [12], optical sensing [13], photocatalysis [14], electrocatalysis [15] and last but not least security and counterfeiting [16].

CDs are synthesized from many different chemicals and organic sources. For example, urea [17], ammonium citrate [18], glycerol [19], citric acid [20], phytic acid [21], graphite [22], boronic acid [23], ethylenediaminetetraacetate [24] , zinc oxide [25], *ocimum sanctum* [26], onion waste [27], sweet potato [28], aloe vera [29], mandarin juice [30], ginkgo leaves [2], tangerine juice [31], sumac [32], prunus mume [33], starch [34], milk [35], coffee bean shell [36], black tea [37], *eleusine coracana* [38], *plectranthus amboinicus* [39], *borassus flabellifer* [40] and rosemary leaves [41] are some example of precursors.

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Carbon quantum dots can be synthesized in two main approaches, top-down and bottom-up. Arc discharge, electrochemical oxidation and laser ablation are particular important instances of top-down methods and combustion, thermal, supported synthetic and microwave are examples of bottom-up methods [42]. Among these different methods, hydrothermal carbonization is known as an environmentally friendly and conventional method for the synthesis of CDs in aqueous media [43].

Laurus nobilis is an evergreen aromatic tree with green and hairless leaves belonging to the Lauraceae family [44]. It is an ornamental plant native to the Mediterranean and *Laurus nobilis* is a known species since ancient times. It was a symbol of peace and victory in ancient Greek [45]. Laurel is one of the significant aromatic and medicinal plant grown in Turkey [46]. *L. nobilis* (bay laurel) leaves are used as a spice and flavoring agent in dishes [47]. Its oil is used in many areas such as perfume, cosmetics, phytotherapy. Its leaves have been used in a variety of medicinal treatments [48] It is also known that the oil obtained from the leaf of *Laurus nobilis* has antibacterial activity [49]. This tree, which grows in Turkey, corresponds to 90% of the laurel trees in the world. For this reason, the mention tree is important for Turkey [50]. In the present work, research, carbon quantum dots (CQDs) were synthesized from green precursor, *Laurus nobilis* leaves, by hydrothermal reaction process. The obtained CDs were analyzed using UV-Vis and fluorescence spectroscopy, X-ray diffraction (XRD) method, transmission electron microscopy (TEM) and Fourier transform infrared spectroscopy (FT-IR), The water-soluble carbon dots were utilized as the fluorescent ink as a one of the important applications of this unique nanoparticles.

2. MATERIALS AND METHODS

2.1. Materials

Laurus nobilis leaves were purchased from local producers. Solvents were obtained from standard manufacturers. All other materials were used in their current form without further purification. The ultra-deionized water was used throughout all experiments.

2.2. Synthesis of Carbon Dots

Laurus nobilis leaves were preferred as carbon source. It was cleaned by washing with tap water and ditilled water. It was place in oven to dry at 55 ° C for 72 hours. The dried materials were sifted after being ground in a mortar. 1 gr of leaf powder were mixed in 50 ml of ultrapure water. It was kept in an ultrasonic bath for 10 minutes for homogeneous distribution. The mixture was transferred to a container made of teflon with a volume of 50 ml. The teflon container was placed in a stainless-steel autoclave. The autoclave was subjected to hydrothermal reaction at 175 °C for 10 hours. The autoclave was left alone after the reaction and cooled to room temperature naturally. The resulting solution mixture was filtered. It was centrifuged at 14,000 rpm for 15 minutes. It was kept at 4 °C for further experimentation and characterization. The schematization of synthesis of CDs and its usage as an invisible fluorescent ink is given in Figure 1.

2.3. Characterization

Fourier Transform Infrared (FTIR) spectra were recorded with Perkin-Elmer Spectrum 400 system in the range 4000–400 cm^{-1}. UV–Vis absorption spectra were recorded by using Shimadzu-1800 UV–Vis spectrometer. Philips X`Pert PRO brand XRD device (Cu Kα radiation (λ =0.154056 nm, set at 40 kV and 30 mA)) was utilized to measure X-ray diffraction (XRD) pattern of samples. The PL spectra were monitored with Varian Cary Eclipse spectrometer. Transmission electron microscopy (TEM) images were obtained from DAYTAM (Erzurum, Turkey) by using Hitachi HT-7700 electron microscope.

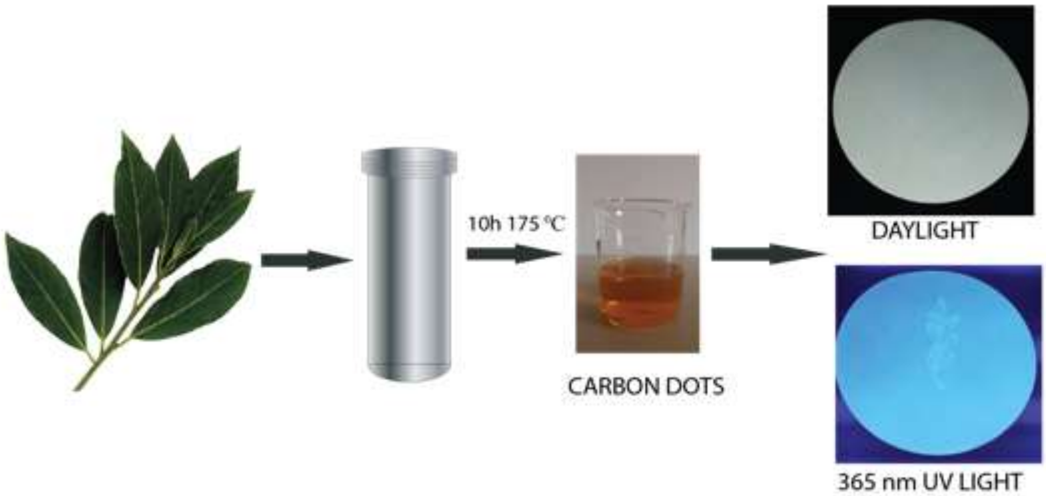

Figure 1. Schematization of synthesis of carbon dots from *Laurus nobilis* leaves and application as an invisible fluorescent ink.

3. RESULTS AND DISCUSSION

The UV-Vis spectrum of the obtained nanoparticle from *Laurus nobilis* leaves is given in Figure 2. The spectrum was measured between 200 to 600 nm wavelength and a distinct peak at 279 nm implying that the π-π* transitions of aromatic sp^2 domain [51]. The long tail of the observed peak also indicate the presence of a carbon quantum dots [52]. The behavior of UV–Vis spectrum of synthesized CDs is consistent with literature [53, 54].

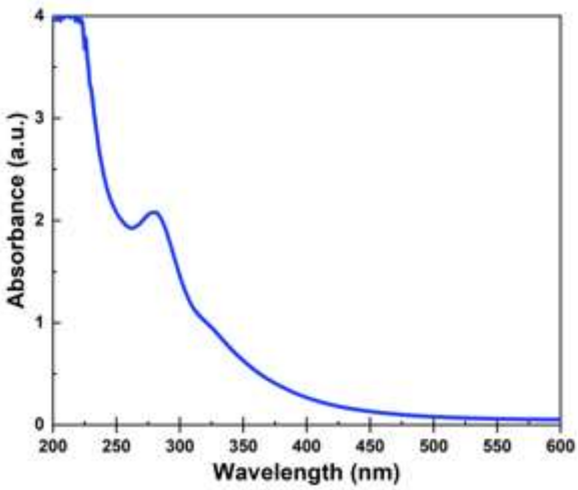

Figure 2. UV-Vis spectrum of synthesized carbon dots from *Laurus nobilis* leaves

The excitation-induced PL spectra of the obtained carbon quantum dots were detected by photoluminescence (PL) spectrometry at increasing excitation wavelengths from 320 nm to 400 nm. The maximum emission of CDs was observed at 425 nm when excited at 344 nm. Figure 3 illustrates the excitation and emission spectrum of produced CDs. When carbon dots were excited at 344 nm (λex), the maximum fluorescence intensity was obtained at 425 nm (λem). This imply optimum excitation and emission wavelength of the prepared CDs is found as 344 nm and 425 nm respectively.

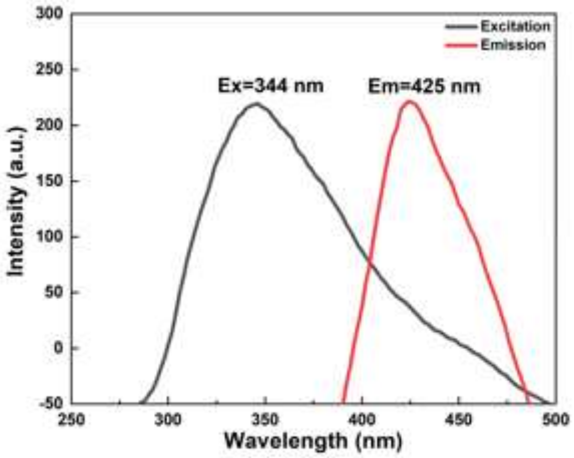

Figure 3. Excitation and emission spectrum of product CDs.

Excitation dependent photoluminescence spectra of carbon quantum dots was demonstrated in Figure 4. The excitation wavelength of sample starts from 320 nm and it is increased with 8 nm intervals to 400 nm. When the present emission peaks examined, it was observed that the intensity of the emission peak increased with the increase in the excitation wavelength and reached a maximum value at 425 nm excitation. After this point, it was observed that the emission peak decreased again. As the excitation wavelength is increased, the fluorescence emission spectra show a redshift [55] . This behavior is a common feature of wavelength dependent CDs fluorescence [56].

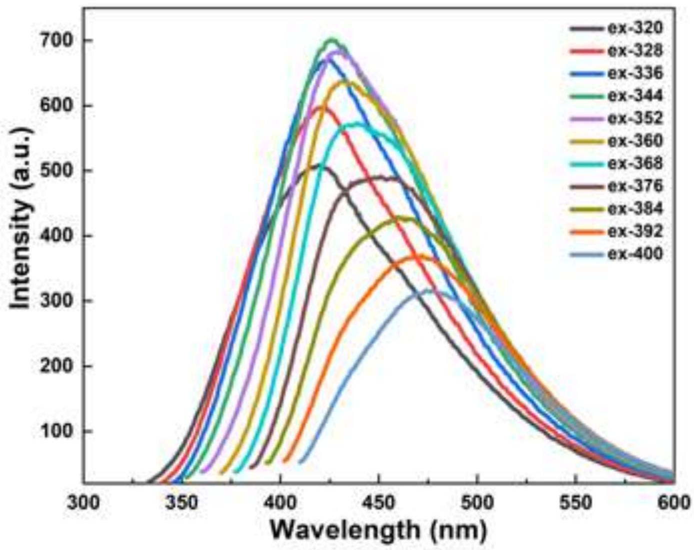

Figure 4. Excitation dependent emission spectra of synthesized CDs.

The FT-IR spectrum of the obtained CDs helps to identify the surface functional groups and it is illustrated in the Figure 5. As seen in the figure, broad peaks observed at 3235 cm^{-1} may indicate that the stretching vibrations of the -NH and -OH functional groups which are interconnected by hydrogen bonding of N/CQDs [57]. The peak about 2927 cm^{-1} is due to C-H stretching (sp^2) [56, 58]. However, the stretching vibration (asymmetric and symmetric) of the peak COO¯ band seen at 1590 cm^{-1} can be attributed to the molecular structure [29]. The peaks seen at 1201 cm^{-1} are due to the CO vibration band [59]. The last peak around 1044 cm^{-1} can be recognized to C-C stretching vibrations and C-N bending vibrations [60].

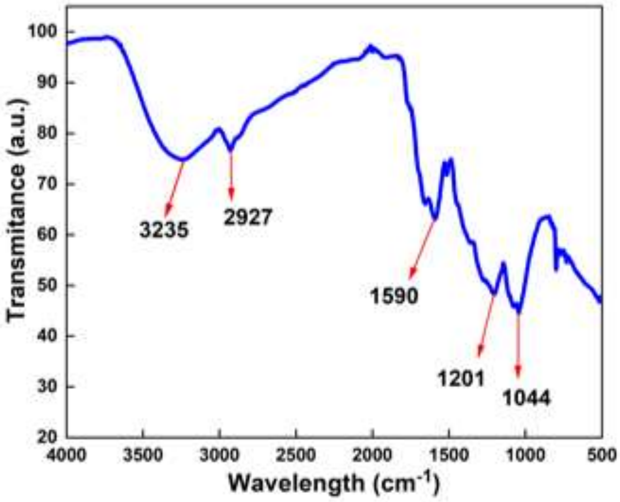

Figure 5. FT-IR spectrum of the CDs.

The crystalline structure of produced CDs synthesized from *Laurus nobilis* leaves is shown in Figure 6. Graphene like structure was seen from this figure [51, 56], the broad amorphous peak at 2θ=23˚ of CDs in the XRD diffraction pattern was observed [61]. The broad peak in this situation are commonly observed in XRD model of amorphous carbon dots [62, 63].

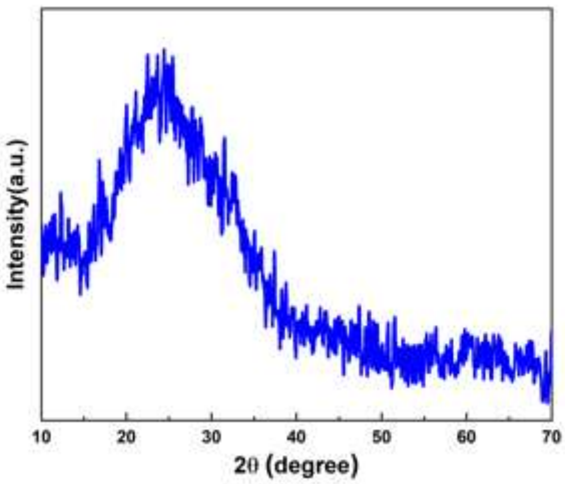

Figure 6. XRD pattern of the prepared CDs

The morphological features of the synthesized carbon quantum dots from *Laurus nobilis* leaves are examined with TEM micrograph and it is given in Figure 7. As seen from this figure, the prepared CDs exists in clusters. CDs in clusters are obvious with darker points. In the middle part of the figure, independent of the clusters, carbon dots with spherical geometrical shape are clearly seen. The particle size of these mentioned small spherical particles is below 10 nm similar particle sizes are also confirmed by previous studies in literature [30, 64]**.**

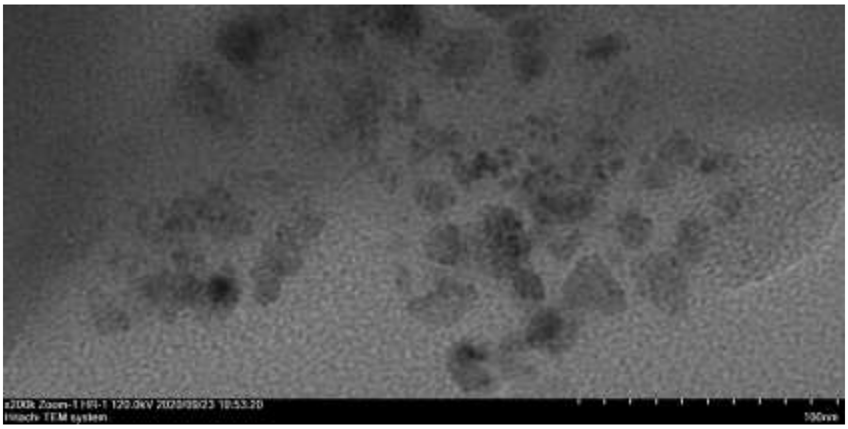

Figure 7. TEM image of synthesized CDs

CDs have been used in different application areas from bioimaging to sensing mainly because of their superior fluorescent properties. CDs are also utilized as fluorescent ink to identify to counterfeit printings, information storage and encryption [65]. In this study, fluorescent ink was prepared by using 15 mL synthesized CDs and 0.5 g polyvinyl alcohol (PVA). They were mixed by the help of magnetic stirrer for 1 hour. The obtained fluorescent ink was poured into empty fountain pen [65]. As showed in Figure 8(a), there were no significant patterns seen on the filter paper but blue fluorescent pattern was apparent under 365 nm light in Figure 8(b). This verify that obtained CDs from *Laurus nobilis* leaves can be used in anti-counterfeiting applications. Furthermore, the synthesized CDs are cost efficient and eco-friendly could be used instead of traditional fluorescent ink. Also, the prepared CDs have potential to use at high quality anticounterfeiting applications by inkjet printing [66].

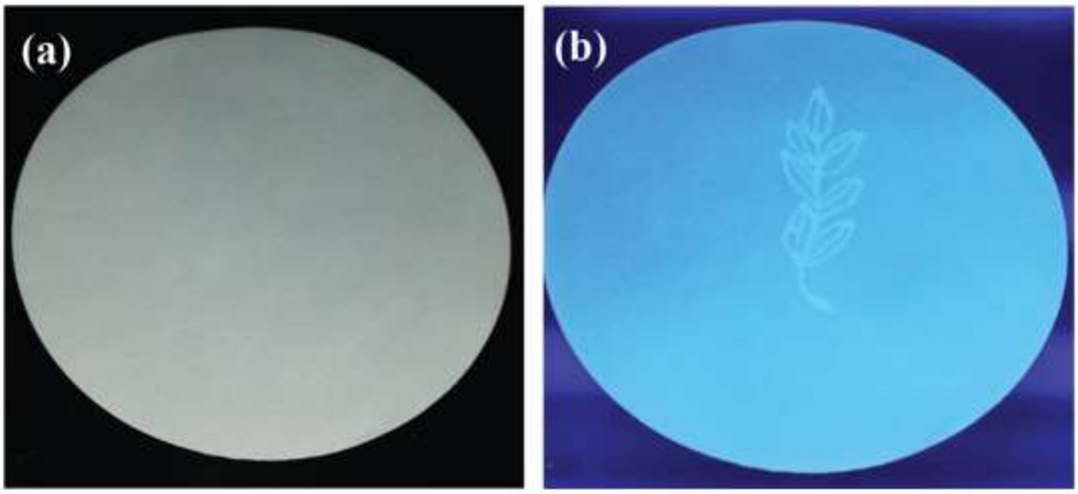

Figure 8. Invisible fluorescent ink application of the synthesized CDs.

The fluorescence stability of obtained CDs from *Laurus nobilis* leaves illustrated in Figure 9. As seen from this figure, the stability of CDs was decreased as a function of time but after 40 days the stability is quite high (over 85 % fluorescence intensity of the freshly prepared CDs). Finding of this data also verify that prepared carbon dots have good stability and great capability for different type of applications like sensing [55].

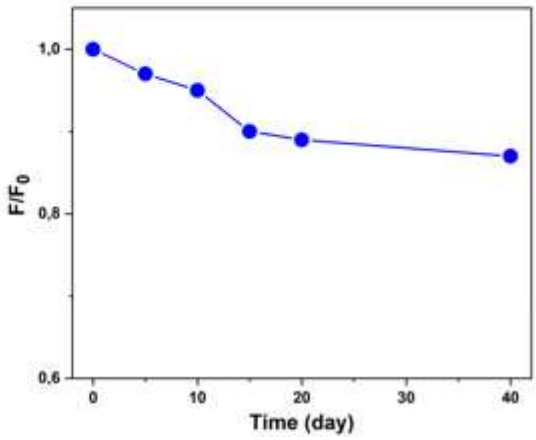

Figure 9. Fluorescence stability of the synthesized CDs as a function of time.

4. CONCLUSION

In the current work, a simple, cost efficient, non-toxicity fluorescent CDs were prepared via one-pot green hydrothermal approach using *Laurus nobilis* leaves as a precursor without any chemical. The color of resulting CDs is bright blue under UV-light (365 nm). According to XRD results, the hydrothermal carbonization of *Laurus nobilis* leaves leads to formation of amorphous carbon phases. The TEM observation reveal that obtained CDs are spherical morphology in shape. The excitation dependent emission feature of synthesized CDs was observed. Moreover, the prepared CDs were also utilized as fluorescent ink due to their excellent fluorescence stability. This novel CDs exhibit excellent fluorescent ink feature as a result of conducted experiment was observed. The obtained water-soluble CDs might potential for utilizing as a fluorescent ink in anticounterfeiting applications with green and cost-effective manner.

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CONFLICT OF INTEREST

The authors stated that there are no conflicts of interest regarding the publication of this article.

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