Effect of post-treatment drying processes on the optical and photothermal properties of carbon nanodots derived via microwave-assisted method

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Abstract. Carbon nanodots (CNDs) are considered as potential materials for photothermal applications and can be used as solar absorbers to enhance the absorption and conversion efficiency of solar energy to heat. To meet the criteria as solar absorbers, CNDs were synthesized via microwave heating and dried by two different drying processes, namely freeze drying and oven drying, obtaining CNDs powder with the labeled of CND-FD and CND-OD, respectively. The effect of these two drying methods on the optical and photothermal properties of CNDs was investigated. It was observed that soft and light powders were obtained from freeze drying, while oven drying resulted in shiny and agglomerate particles. Oven drying did not alter the absorption profile of CNDs, but freeze-drying resulted in broader and slightly red-shift absorption compared with that of CNDs colloid before drying. Photoluminescence intensity of CND-FD was only half of that of CND-OD. FTIR analysis revealed that CND-FD contained fewer hydroxyl and hydrophilic amine groups, leading to less hygroscopic nature of CND-FD than CND-OD. Because of its better absorption capability, the presence of CND-FD in water significantly increased the water evaporation rate and evaporation efficiency up to 2.2 kg/m²·h and 84%, respectively, three times higher than that of water without CNDs. With a similar photothermal testing condition, CND-OD resulted in a evaporation rate of only 0.9 kg/m²·h and evaporation efficiency of 36%. It can be noticed that freeze drying is more suitable to dry CNDs powder over oven drying to obtain excellent optical and photothermal properties of CNDs.

Keywords: carbon nanodots; clean water; freeze drying; solar evaporation; microwave;

1. Introduction
Carbon nanodots (CNDs) are emerging carbon nanomaterials that have been used in an extensive range of applications. Owing to their small size (less than 10 nm), low toxicity, good biocompatibility, and high dispersibility in water, CNDs have been employed in biomedical fields, including drug delivery, bioimaging, biosensors, and cancer therapy [1–3]. Their outstanding photoluminescence and intrinsic
catalytic properties have been utilized in photocatalytic and dye-sensitized solar cells [4–6]. In the last four years, CNDs have also been investigated as solar absorbers for solar steam generation or solar water evaporation system [7–10]. Not only consist of carbon, hydrogen and oxygen atoms, the core of CNDs can also comprise of nitrogen, sulphur, boron, and phosphorus atoms, depending on the precursors [10,11]. On the surface, CNDs also have abundant functional groups such as amine, carbonyl, carboxyl, hydroxyl, and epoxy groups that determine the optical and electronic properties of CNDs [12].

In general, the synthesis of CNDs can be categorized into top-down and bottom-up approaches based on carbon raw materials. The former approach involves breaking down the higher dimensional carbon-based materials into a nanoscale particle through a physical or chemical synthesis, including laser ablation, electrochemical, or arc discharge [13–15]. On the other hand, bottom-up techniques, which include microwave, hydrothermal, solvothermal, plasma and chemical treatment, form CNDs from a smaller carbon-based molecule through carbonization and polymerization processes [16,17]. Among the methods mentioned above, the microwave was chosen due to rapid and uniform heating, energy saving, lower processing cost, and ease to scale up. Several works have revealed the successful synthesis of CNDs using microwave-assisted synthesis [10,17–19].

During the preparation of CNDs, drying process plays a critical role in controlling surface and internal structure of CNDs. Drying means remove water from a colloid system that can be thermal (e.g. oven drying) or non-thermal process (e.g. freeze drying) [20]. Control over surface and internal structure are key means when CNDs are used as light harvesters and photothermal materials [21,22]. Many studies have reported the influence of precursor materials and processing condition (temperature, time, solvent medium, concentration, etc.) on the characteristic of CNDs [23,24], but limited study that specifically investigate and directly compare the effect of different drying process. Therefore, the evaluation of different drying process to obtain excellent properties of CNDs, such as optical and photothermal properties, is still challenging.

In this study, CNDs were prepared by a facile microwave-assisted treatment derived from citric acid and urea. Two different drying processes, namely freeze drying and oven drying, were employed and compared. The objective of this work was to evaluate the effects of different drying process on optical and photothermal properties of CNDs. Optical properties were studied through the absorption and fluorescence profile, while photothermal behaviour was evaluated by irradiating CND-contained water using a solar simulator as the light source. This research describes the relationship between absorption and photothermal behaviour of CNDs, providing a potential application of CNDs as solar absorber in solar water evaporation system.

2. Experimental Method

2.1. Materials
Citric acid monohydrate and urea as the precursors of CNDs were purchased from Merck. Ultrapure water (UPW) (~0.061 µS/cm at 25 ºC) was obtained from a Millipore water purification system (Milli-Q, Germany).

2.2. Preparation of carbon nanodots
Citric acid monohydrate (2.1 g) and urea (3.0 g) were dissolved in an Erlenmeyer flask containing 20 mL of ultra-pure water. Subsequently, the solution was irradiated in the microwave oven (Electrolux EMS3087X, China) with M-HI mode (irradiation 26 s, stop 7 s) for 3 min. After cooling down to room temperature, the resulting product was dissolved in 20 mL of water using sonication for an hour to remove any lumps. The dark brown solution was then filtered using Whatman filter paper (Grade 1), and the filtrate was centrifuged (15,000 rpm, 30 min, room temperature, Himac CS150NX) to remove unreacted materials and large particles. After that, about 10 mL of supernatant was put in a petri dish with a diameter of 10 cm and dried by freeze drying and oven drying (100 ºC). Prior to freeze drying, the petri dish was placed in a deep freezer (Arctiko, Denmark) at -80 ºC for one day and subsequently put in the tray of freeze dryer (Labocon, United Kingdom) for three days. It took about 3-h evaporation
in a Universal Oven (Memmert UN260, Germany) to remove the water. Throughout this paper, CNDs powder obtained from freeze drying and oven drying will be named CND-FD and CND-OD, respectively.

2.3. Characterization of carbon nanodots

UV-visible absorption spectra of the CNDs were detected using Cary 60 UV Vis spectrophotometer (Agilent, USA) from 200 to 800 nm. Photoluminescence (PL) spectra were recorded using Cary Eclipse Fluorescence Spectrometer (Agilent, USA). Prior to optical measurement, the CNDs powder was re-dispersed in water at 500 ppm. Identification of functional group and chemical structure were performed using Fourier Transform Infrared (FTIR) in attenuated total reflectance (ATR) mode (Thermo Fisher Nicolet iS5, USA) at a resolution of 4 cm\(^{-1}\).

2.4. Water evaporation test

A water evaporation test to evaluate the photothermal behaviour of CNDs was conducted as illustrated in Figure 1. The samples were re-dispersed as much as 20 mg of powder in 60 mL of water. The water container was surrounded by thermal insulating polystyrene foam. Using a portable solar simulator (KGC Saintifik PEC-L01, Malaysia) with an intensity of 1 kW/m\(^2\), the samples were irradiated for 60 min in a controlled atmosphere at 27 °C and RH 58% and closed environment. The water loss weight and the water temperature were recorded using an electronic mass balance and a dual-channel thermocouple, respectively, for every 10 min. For the comparison, only water (without any CNDs) was irradiated with the solar simulator.

Figure 1. Schematic diagram of water evaporation test.

The water evaporation rate is determined as the value of the mass of evaporated water (kg) divided by time (h) and surface area (m\(^2\)) as stated in equation (1), while evaporation efficiency is calculated as the ratio of Qe (power for water evaporation) and Qs (power of solar illumination) stated in equation (2), where \(h_{LV}\) is the total enthalpy of the liquid vapour phase change (2257 kJ/kg), \(C_{opt}\) is the optical concentration, and \(q_i\) is the intensity of solar irradiation (1 kW/m\(^2\)) [25].

\[
\dot{m} = \frac{dm}{A \times dt} \quad (1)
\]

\[
\eta = \frac{Q_e}{Q_s} = \frac{\dot{m} h_{LV}}{C_{opt} q_i} \quad (2)
\]
3. Results and Discussion

3.1. Optical properties

Different drying process gives different result on the obtained CNDs powder, as shown in Figure 2a. From visual observation, direct sublimation of ice in the freeze-drying process gave softness and light powder for CND-FD that was easy to collect from the petri dish. On the other hand, oven drying led to the formation of shiny and dense layers on the petri dish, and the CND-OD powder can be collected by scraping the surface of the petri dish using the tip of the spatula. In addition, CND-OD transformed into sticky powder upon exposure to the atmosphere about 30 min after taking them out from the oven, resulting in dense and agglomerate powder due to the presence of water content in the CND-OD powder. Such hygroscopic nature was also observed by others [26,27]. In daylight, re-dispersion of CND-OD in water produced yellow colloid, while re-dispersion of CND-FD resulted in brown colloid (Figure 2b). Under UV illumination, CND-OD shows brighter fluorescence than CND-FD (Figure 2c) that can be quantified later using PL measurement.

![Figure 2. Optical images of CND-FD (left) and CND-OD (right) a) in powder form, b) in solution under visible light, and c) in solution under UV irradiation.](image)

UV-Vis spectra of the re-dispersion of CNDs are studied to determine the discrepancy in the absorption properties between CND-FD and CND-OD. As demonstrated in Figure 3a, CNDs generally exhibit broad optical absorption in the ultraviolet (UV) region (200-350 nm). The absorption peaks appeared at around 230-280 nm are due to π-π* electronic transition of aromatic C=C in the core of CNDs [28] and another peak at 335 nm is owned to n-π* transition of C=O/C-N/C=N bonds [29]. In addition, the visible absorption band (above 400 nm) is attributed to the amine groups on the CNDs surface [30] or surface defects that might occur during the synthesis. The expanding absorption to 550 for CND-OD and CND before drying as well as to 700 nm for CND-FD indirectly explains nitrogen-doping CNDs. Graphitic nitrogen dopants can inject excess electrons into the unoccupied π* orbitals, thus reducing the high occupied molecular orbital (HOMO) to lower unoccupied molecular orbital (LUMO) energy gap [31].

Surface engineering through freeze-drying and oven drying can modify the corresponding absorption spectra of CNDs. Slow evaporation through oven drying did not alter the absorption peaks of CNDs. On the contrary, direct sublimation of ice in freeze drying can prevent CNDs from agglomeration, thus broadening the absorption spectrum in the visible region. Moreover, a higher concentration of CND-FD owing to its lower density diminished the absorption band around 335 nm. At high CNDs concentration, the relative distance between CNDs individual particles is smaller, which might create new energy levels at CNDs due to the interactions between surface groups [32]. Comparing the absorption profile in the visible region, CND-FD has a peak at 414 nm, whereas CND-OD shows a peak at 408 nm. Figure 3b shows the photoluminescence emission spectra of CND-FD and CND-OD excited at 365 nm. The CND-OD emits brighter fluorescence than CND-FD under UV lamp 365 nm, as previously described in Figure 2c. The brighter fluorescence of CND-OD is confirmed by the higher PL intensity of CND-OD than CND-FD, about two times.
3.2. FT-IR analysis

To explore further the differences in the structure of CNDs obtained by different drying methods, FT-IR analysis was employed to identify the functional groups on CNDs. Figure 4 displays peaks in similar positions but with a more vigorous absorption intensity for CND-OD. The broad absorption bands present at 3100-3500 cm\(^{-1}\) are assigned to hydroxyl (O-H) and hydrophilic amine (N-H) stretching vibration. This broad peak contributes to the hydrophilicity and stability of CNDs in aqueous system [16,33]. Stronger absorption of CND-OD in that wavenumber causes CND-OD is more hygroscopic than CND-FD. Other broad peaks between 2750-3050 cm\(^{-1}\) suggest that stretching vibrations of C-H are formed [23]. The absorption peak at 1650 cm\(^{-1}\) along with a small peak at 1770 cm\(^{-1}\) correspond to the C=O stretching vibration of carbonyl groups [17,34]. The existence of amide bonding in CNDs was confirmed by the presence of primary amide and secondary amide groups. The peak at 1410 cm\(^{-1}\) is characteristic absorption of primary amide, which is the stretch vibration of the amine III C-N bond, whereas the secondary amide appears at 1573 cm\(^{-1}\) that is corresponded to the bending vibration of the amine II C-NH bond [35]. Those results corroborate the UV-Vis analysis. There are also an absorption band at 1185 cm\(^{-1}\) attributed to C-O stretching and a peak at 1060 cm\(^{-1}\) belongs to C-O-C stretching vibration [17,35]. According to a previous study, C-O-C stretching vibration could indicate that complete intermolecular dehydration and polymerisation were taken place [35].

Figure 4. FT-IR spectrum of CNDs dried by freeze drying (CND-FD) and oven drying (CND-OD).
3.3. Photothermal properties

For evaluating the photothermal behaviour of CNDs, the weight losses and temperature rise of water with and without CNDs under solar illumination of 1 kW/m² (1 sun) were recorded, and the results are shown in Figure 5a and 5b. In those figures, the temperature rise and mass change of CNDs-based samples are higher than that of bulk water only, indicating the photothermal effect of CNDs. Furthermore, when using CND-FD, the temperature rise in water is double the temperature rise in water with CND-OD (Figure 5a), thus contributing to fast evaporation in that water system. After 60 min of 1 sun solar illumination, the mass change in water with CND-FD system reached up to 2.2 kg/m², while it was only about 0.9 kg/m² for water with CND-OD (Figure 5b). These results indicate better photothermal conversion ability of CND-FD than CND-OD. One reason for this phenomenon should be the better absorption of CND-FD since more electrons transfer from HOMO to LUMO, then more possibilities to quench fluorescence and produce heat from lattice vibration or non-radiative relaxation of photoinduced electrons [36]. Moreover, some literatures demonstrated that aggregation might occur during oven drying process and induce radiative emission [28,37] that diminishes the possibility of producing heat through non-radiative relaxation.

![Figure 5](image_url)

**Figure 5.** Photothermal properties of CNDs dispersed in water and water only as control under 1 sun illumination (1 kW/m²). a) Mass changes of water over time, b) Temperature rises of water as a function of time.

To further study the photothermal performance of CNDs in solar evaporation systems, water evaporation rate and evaporation efficiency were calculated using equation (1) dan (2). Compared to CND-OD and water only, CND-FD shows a superior water evaporation rate of 2.20 kg/m².h under one sun, which is 2.3 times that of CND-OD and 3.3 times that of water only under the same evaporation condition. The evaporation rate in this work is comparable with some of the recently developed carbon dots for solar evaporation systems that report water evaporation rates in the range of 1.82-2.31 kg/m².h [9,32,38]. The calculated evaporation efficiencies of CND-FD and CND-OD were 84% and 36%, respectively. It is to be noted that higher absorption could result in achieving a higher photothermal effect as well as efficient solar evaporation. Similar efficiency was reported by Pal et al. when CNDs co-doped with nitrogen, sulfur, and phosphorus were dispersed in an aqueous medium and irradiated with NIR-laser irradiation (2 W, 808 nm IR laser) [10].

4. Conclusion

Here, we report a facile synthesis of CNDs by using microwave-assisted method derived from citric acid and urea. We found that post-treatment drying process affected optical and photothermal properties of CNDs. Owing to the ability to remove bound water, freeze drying technique resulted in soft and light powder, hence more particle’s concentration at the same weight compared to oven-dried CNDs. UV-Vis absorption profile demonstrate that oven drying gave similar absorption peak with CNDs colloid before drying. Higher concentration of CND-FD due to lower density induced red-shift and broad
absorption intensity in the visible region. Based on FT-IR analysis, oven-dried CNDs have more hydroxyl and amine groups that led to more hygroscopic powder over CND-FD. Owing to better absorption of CND-FD, this material shows superior photothermal properties when CND-FD is dispersed in water and irradiated by a solar simulator. An efficient solar evaporation system with an evaporation rate of 2.2 kg/m$^2$.h and an evaporation efficiency of 84% was achieved when using CND-FD. The CND-FD powder with better absorption and simple preparation provides a promising solar evaporation system for clean water production.

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Author contribution
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