Synthesis Techniques of Mixed-Doped Carbon Quantum Dots

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In nanotechnology, the synthesis of carbon quantum dots (CQDs) by mixed doping with metals and non-metals has emerged as an appealing path of investigation.

mixed-doped CQDs chemical sensing biosensing

1. Introduction

Carbon quantum dots (CQDs) are a fascinating class of fluorescent nanomaterials with particle sizes of between 2–10 nm [1]. Carbon quantum dots may also be referred to as carbon dots (CDs) [2], carbon nanodots (CNDs) [3], carbon polymer dots [4], and carbon nanoparticles (CNPs) [5]. In 2004, Xu et al. discovered these nanoscale structures coincidently during the isolation of single-walled carbon nanotubes synthesized from arc-discharge dust [6]. Since then, CQDs have received considerable attention owing to their distinctive properties and emerging applications.

Lately, CQDs have been synthesized for modification, surface functionalization, and various other applications [7]. However, many review articles published since 2011 have focused on the advancements in synthesizing carbon quantum dots using economical and straightforward methods [8], primarily for their applications in chemical/biological sensing and bio-imaging [9]. Hence, there is an urgent need for developing CQDs (particularly doped/co-doped) to further explore their applications [10].

3 Synthesis of Fe-N-CQDs

The electrochemical oxidation method was adopted to synthesize Fe-N-CQDs by coating an electrode with carbon cloth. Fe-N-C and PTFE in the ethanolic mixture were coated on the carbon fabric to prepare an electrode. The electrode worked as an anode and was dried at 80 °C for 12 h for electrolysis. PTFE material and voltage conditions were controlled to assess its efficiency. The counter electrode was Pt foil with 8 mL H_2O, 30 mg NaOH, and 35 mL C_2H_5OH as an electrolyte which was further dialyzed for 24 h, and the final solution was lyophilized to obtain Fe-N-CQDs powder [11].

3. Synthesis of Fe-N-CNPs

The present study involved the production of a composite material, namely, Fe-N-CNPs, through a plasma process. The plasma was generated by applying a high-voltage pulse between a proportional pair of metallic Fe electrodes immersed in a homogeneous blended CNF and 2-cyanopyridine ($C_4H_4N_2$). The carbon nanoparticles were doped by nitrogen in situ and were synthesized through dissociation and recombination processes using 2-cyanopyridine as the precursor. Simultaneously, iron atoms were mixed from the electrode into the homogeneous mixture. Within the plasma region, Fe atoms may react with C_2 and CN radicals that possess high levels of energy, which are produced through the dissociation of 2-cyanopyridine molecules. This reaction results in the creation of Fe-N coordination. The Fe and N sites in the plasma region were intermingled into suspension, and, subsequently, adhered to the surface of the CNF, forming a composite structure comprising N-CNP. The synthesized substance underwent a second heat treatment at 900 °C for 2 h within an argon atmosphere [12].

4. Synthesis of the Fe,N-C Electrocatalysts

In this synthesis process, 0.4 g Hemin was initially dissolved in 40 mL N,N-dimethylformamide (DMF), and combined with 0.4 g BP2000. The resulting mixture underwent sonication for a duration of 1.5 h to produce a uniform solution, which was, subsequently, subjected to a temperature of 50 °C to facilitate solvent evaporation. Later, the powder was transferred into a tubular furnace for pyrolysis at 900 °C for 2 h while exposed to an argon atmosphere. The resultant substance was washed using 2 M HCl at ambient temperature to eliminate unstable iron atoms or compounds. After washing with the acid, the substance underwent a second heat treatment at a temperature of 900 °C for 2 h in an argon atmosphere, producing a modified Fe-N carbon electrocatalyst, Fe-N-C-AH [13].

5. Synthesis of Pristine Fe-N-GQDs

A solution was prepared by dissolving 0.25 g citric acid, 0.16 g urea, and 0.2 g $FeCl_3$ in 5 mL ultrapure deionized water. After an hour of stirring, the solution was transferred into a 100 mL autoclave and was heated up to 150 ℃ for 12 h. Then, the mixture was centrifuged at a speed of 10,000 RPM for 5 m to eliminate solid residue. The aqueous solution underwent dialysis using a dialysis membrane for one night. The Fe-N-GQDs were obtained as the outcome [14].

6. Synthesis of Fe/N-CDs

The solution was prepared by dissolving 0.06 g $FeCl_3 \cdot 6H_2O$, 0.10 g EDTA, and 0.06 mL DETA in 10 mL ultrapure water. Then, the mixture was introduced into an autoclave lined with 30 mL Teflon, which was then subjected to a temperature of 200 °C for 6 h. The resultant solution was subjected to a 2500-fold dilution using PB buffer (10 mM, pH = 7.4) and, subsequently, preserved at 4 °C for further use [15][16].

7. Synthesis of Fe,N-CDs

The experimental procedure involved the combination of 1 g trisodium citrate, 2 mL ethylenediamine, and 1 g $FeCl_3$ with 30 mL ethylene glycol. The solution underwent vigorous stirring for 15 m until achieving clarity. Subsequently, the mixture was introduced into a stainless-steel autoclave lined with 100 mL Teflon. Then, the solution was subjected to a temperature of 200 °C and maintained for 8 h. After cooling at an ambient temperature, the products with a dark-brown hue underwent centrifugation at 10,000 RPM for 10 m to eliminate the least dense particles. Later, the rotary evaporator was employed to eliminate ethylene glycol. Fe,N-CDs in solid form were dissolved into deionized water and subjected to dialysis to eliminate low molecular weight species. Afterwards, the Fe,N-CDs solution was lyophilized to obtain its powdered form. The powder was stored at a temperature of 4 °C to ensure its preservation for later use [17].

8. Synthesis of Fe@N-CDs

The Fe@N-CDs were synthesized using *P. edulis* as an eco-friendly carbon precursor in a single-step hydrothermal method. The husks of *P. edulis* were first subjected to natural drying and then pulverized into a fine powder. After that, a mixture comprising 1.0 g powder, 1.0 mL EDA (agent for surface passivation), and 0.6 g $Fe_2(SO_4)_3$ as an iron source was homogeneously blended with 35 mL distilled water. Later, the solution was introduced into an autoclave lined with 50 mL Teflon and subjected to thermal treatment at 180 °C for 4 h. After the reaction, the autoclave was allowed to undergo natural cooling to achieve ambient temperature. The resulting carbonaceous solution was filtered using a 0.22 µm membrane filter to remove particles of significant size. Afterward, the solution was purified using a dialysis bag with deionized water for 24 h. A solution of Fe@NCDs exhibiting a homogenous brown hue was successfully obtained and preserved at a temperature of 4 °C for further use [18].

9. Synthesis of N,Cu-CQDs

In a standard protocol, a solution containing 19 mmol of EDTA and 19 mmol of $Cu(NO_3)_2{\cdot}3H_2O$ was combined with 105 mL of ultrapure water within a 150 mL Teflon-lined autoclave. The resultant mixture was subjected to a thermal treatment at a temperature of 180 °C for 12 h after ultrasonic irradiation for 15 min. After achieving the ambient temperature, the solution mixture was subjected to overnight dialysis using a bag with a molecular weight retention threshold 1000 Da, for the complete removal of salt ions. The solution was later filtered using a 0.22 µm filter to eliminate particles of significant size. Then, the solution was subjected to re-dissolution using ultrapure water until a total volume of 100 mL was achieved following lyophilization. The N,Cu-CD concentration was measured at 0.73 mg mL^{-1} [19].

10. Synthesis of Cu-N@CDs

The Cu-N@CDs were synthesized by the thermolysis technique. A mixture of 1 g of Na_2-EDTA and $CuCl_2$ mixture was introduced into a 50 mL beaker containing deionized water. Then, the solution was stirred mechanically for 20 m. The product was isolated via filtration and heated in an oven at 200 °C for 5 h. Subsequently, the oven was

allowed to cool at RTP, and the resulting product was collected, pulverized, and dissolved in deionized water. The suspension underwent ultrasonic treatment for 2 h at an ambient temperature. The filtration membrane (with a pore size of 0.22 μm) was utilized to eliminate the sizable particles via filtration. The acquired Cu-N@C-dots solution was preserved at a temperature of 4 °C to facilitate subsequent analysis and utilization in PGL estimation [20].

11. Synthesis of N/CuCDs

The N/CuCDs were synthesized by a one-pot hydrothermal approach. A solution was prepared by adding 1.2 g citric acid monohydrate (CA) and 0.15 g copper acetate monohydrate in 20 mL of deionized water. Diethylenetriamine (DETA) was dissolved in the resultant solution at 0.15 mL. The solution underwent ultrasonic dissolution for 10 min, and, then, it was introduced into a Teflon-lined stainless-steel autoclave with a volume capacity of 30 mL. The autoclave was subjected to a temperature of 230 °C for 12 h. The product was dialyzed for 72 h after cooling at an ambient temperature to eliminate residual reactants. The hazel solution obtained was subjected to reduced pressure distillation to eliminate excess water. The N/CuCDs brown powder was acquired through vacuum drying [21].

12. Synthesis of Cu-N-CDs

The Cu-N-CDs were synthesized by solvothermal carbonization of folic acid (FA) and $CuCl_2$ in an ethanolic solution. A solution was prepared by dissolving 0.05 g FA and 0.12 g $CuCl_2$ in 15 mL ethanol. The solution was introduced into an autoclave lined with polytetrafluoroethylene and heated up to 180 °C for 6 h. The unrefined substance underwent filtration using a nylon membrane filter with a pore size of 0.22 μm and then eliminating ethanol through rotary evaporation. The obtained product was dialyzed in water for approximately 24, 36, and 48 h by adding deionized water. The findings suggest that variations in dialysis time do not have a significant impact on the fluorescence intensity. Consequently, a dialysis time of 24 h was the most effective. The obtained CDs are commonly denoted as Cu-N-CDs [22].

13. Synthesis of Cu,N@CQDs

The Cu, N@CQDs were synthesized using a one-pot hydrothermal approach. In summary, a solution was prepared by dissolving 0.65 g glucose and 0.45 g $CuSO_4 \cdot 5H_2O$ in 50 mL of double-distilled water. A 0.35 mL of ethylenediamine was introduced into the mixture above. Later, the mixture was agitated for 20 min and then followed by the thermal treatment at 200 °C for 10 h. The solution was obtained by adding the sample into centrifugation at 6000 RPM for 15 min. The resulting solution was filtered using a 0.2 μm filter membrane and was subjected to dialysis within a dialysis bag for three days. The dialysis water was replaced every 12 h. The synthesized Cu, N@CQDs were freeze-dried and stored at a temperature of 4 °C [23].

14. Synthesis of Zn-N-CQDs

A solution was prepared by dissolving 0.5 g $ZnSO_4$, 0.9 mL of conc. H_2SO_4, and 2.65 mL of ethylene diamine in 25 mL of water. The mixture was subjected to ultrasound for 5 min to ensure complete homogenization. The experiment was conducted for 20 min at a temperature of 150 °C, using a microwave digestion method with a power output of 1450 W. After removal, the substance was cooled to room temperature, gathered, and concentrated for further use. The QY% of Zn-N-CQDs was determined to be 14.26% using the relevant equation [24][25].

15. Synthesis of N,Zn-CDs

The synthesis process involved the addition of 0.9 g of citric acid, 2.6 mL EDA, and 0.2 g zinc acetate to 25 mL of distilled water. The resulting mixture was subjected to sonication for a brief period until a clear solution was obtained. The transparent solution was placed in a Teflon vessel with a volume of 60 mL, which was sealed tightly and inserted into a microwave oven. The microwave oven, operating at 850 W, raised the system's temperature to 150 °C within a minute. Under the specified reaction conditions, the reaction was conducted for 30 min by regulating the exposure time of microwaves (700 W). After 30 min, the reaction setup was fully deactivated and then allowed to equilibrate at an ambient temperature. The reaction solution was centrifuged at 10,000 revolutions per minute for 15 min at room temperature. Afterward, filtration was carried out using a membrane of 0.22 µm, followed by dialysis through a membrane for 8 h, using ultrapure water. The solid materials were acquired for subsequent analysis through freeze-drying [26].

16. Synthesis of Zn/Co-N-CQDs

A mixture of 0.3 g diphenyl semi-carbazide, 0.1 g cobalt sulfate, and 0.1 g zinc chloride was prepared in 30 mL aqueous solution. The solution mixture was transferred into a polytetrafluoroethylene auto-clave and subjected to a thermal treatment at 200 °C for 12 h. After cooling to room temperature, the solution was dialyzed through ultrapure water for 24 h, using a dialysis membrane with a molecular weight cut-off of 1000 Da. The ultrapure water used in the dialysis process was replaced every 4 h. Subsequently, the Zn/Co-NCDs solution was acquired and preserved at 4 °C for further examination [27].

17. Synthesis of Ce-N-CQDs

A solution in 40 mL ultrapure water was prepared by dissolving 2 g citric acid (CA) and 1 g cerium nitrate hexahydrate ($Ce(NO_3)_3{\cdot}6H_2O$). Later, 1 mL ethylenediamine (EDA) was added to the prepared solution and stirred continuously to achieve homogeneity. Following this, the mixture underwent a reaction within an autoclave at a temperature of 220 °C for 2 h. After cooling to room temperature, the brown–yellow substance was purified through dialysis. Subsequently, the Ce-N-CQDs powder acquired through lyophilization was enclosed and conserved at a temperature of 4 °C for further investigations [28][29].

18. Synthesis of Ni-N-C Materials

To prepare Ni-N-C materials, PVP and $Ni(NO_3)_2$ were completely dissolved into a concentrated solution of NaCl. The resulting mixture was, subsequently, frozen at −80 °C, and water was removed gradually from the frozen mixture through vacuum freeze drying. Then, the freeze-dried powder was pyrolyzed to obtain Ni-N-C materials under an N_2 atmosphere at a temperature of 900 °C [30].

19. Synthesis of Au/N-CQDs

The Au/N-CQDs were synthesized via hydrothermal technique. Initially, a solution comprising 0.1 g folic acid (FA), 1 mL glycerol, and 1 mL chloroauric acid (CA, $HAuCl_4 \cdot 3H_2O$) was dissolved in 30 mL deionized water. Then, the solutions were subjected to magnetic heating agitation at a temperature of 80 °C for 10 min. The solutions were introduced into a 50 mL autoclave lined with poly(tetrafluoroethylene) (Teflon) and subjected to a temperature of 180 °C for 12 h. Finally, the autoclave was allowed to cool at room temperature, resulting in formation of yellowish solutions. The solutions were filtered to eliminate aggregate using microfiltration membranes with a pore size of 0.22 µm. Finally, solutions of Au/N-CQDs were acquired (**Figure 5**d) [31].

20. Synthesis of Co-N-CDs

The conditions that yielded the most favorable outcome for the synthesis of Co-N-CDs were determined to be as follows: The experimental conditions involved the use of 2.0 g CA, 0.07 mL DETA, and 0.4 g $CoCl_2 \cdot 6H_2O$, with a reaction temperature of 160 °C and a reaction time of 8 h. A solution was prepared to synthesize Co-N-CDs by dissolving 2.0 g CA and 0.4 g $CoCl_2 \cdot 6H_2O$ in 20 mL ultrapure water. After that, 0.07 mL DETA was introduced into the solution mentioned above. The solute underwent complete dissolution by employing an ultrasonic technique for 10 min. Then, the solution was introduced into a Teflon-lined stainless-steel autoclave chamber with a 50 mL volume and subjected to a reaction at 160 °C for 8 h. After cooling to room temperature, the solution was subjected to dialysis using a dialysis bag with a molecular weight cutoff of 500 Da for 72 h. The Co-N-CDs solid powder was acquired through a 48 h vacuum freeze-drying process. The Co-N-CD powder was stored at a temperature of 4 °C for further use. [32][33].

21. Synthesis of Bi-N-CQDs

DI water was used to wash the collected rice husk to remove the significant contaminants in the rice husks. Then, the rice husk was processed via a tabletop blender into tiny fragments. After pretreatment with 0.1 M HCl to eliminate any surface contaminants, the fine rice husk was continuously rinsed with DI water until the pH reached neutral. Then, the rice husk was dried at 60 °C in an oven for the rest of the day. For the doping of N and Bi, EDA with a volume ratio of 5 and 20 vol% and bismuth nitrate pentahydrate with a mass of 1 and 5 wt% were used. Following the various academic research for the synthesis of carbon quantum dots from rice husk, the hydrothermal temperature of 190 °C was chosen for synthesizing Bi-N-CQDs [34].

22. Synthesis of Mg-N-CQDs

Typically, 40 mL of distilled water was used to dissolve 10 g of citric acid (CA) and 4.2 g of $Mg(OH)_2$ to prepare a colorless, transparent, homogenous solution to avoid other anions. The hydrothermal method was used at a temperature of 200 °C for 3 h. Later, the product was subjected to filtration, dialysis, and lyophilization to obtain CQDs labeled as Mg-CQDs. Corresponding experiments were conducted to understand the role of magnesium chelator for high yield fluorescent yield of carbon quantum dots (CQDs), such as (1) citric acid pyrolysis, (2) use of ethylenediamine as surface modification agent in the absence of $Mg(OH)_2$, and (3) incorporation of ethylenediamine and $Mg(OH)_2$. The resulting product was labeled as Mg-N-CQDs [35][36][37].

23. Synthesis of Ag-N-CQDs

The Ag-N-CQDs synthesis involved mixing 1 mL of EDA, 0.1 g $AgNO_3$, and 1 g citric acid (CA). Then, the solution mixture was transferred to a Teflon-coated container at a temperature of 180 °C for 4 h. The mixture was dialyzed for 24 h to obtain Ag-N-CQDs by cooling to room temperature [38][39][40].

24. Synthesis of N/Al-CDs

The synthesis of N/Al-CDs involved the combination of finely ground durian shell waste (DSW) powder weighing 1 g and having a mesh size of 0.22 mm, with 0.1 g urea and 0.1 g aluminum nitrate. Later, the mixture was introduced into a Teflon-coated stainless-steel autoclave reactor with a 45 mL volume and 25 mL of deionized water. Then, the sealed reactor was subjected to a thermal treatment at a temperature of 210 °C for 12 h once the mixture had reached ambient temperature [41].

25. Synthesis of Zr-N-CDs

Zr-N-CDs were produced by dissolving 2 g citric acid and 1 g zirconium chloride in 40 mL ultrapure water, later supplemented with 1 mL ethylenediamine. After complete dissolution in water, the two mixtures above were transferred to a lined autoclave and subjected to a temperature of 150 °C for 2 h. The following procedure was conducted using identical parameters. Following the cooling of the mixture to ambient temperature, it underwent centrifugation for a duration of 15 m at a velocity of 10,000 revolutions per minute. The resultant supernatant was collected and subjected to additional filtration via a 0.22 mm filtering apparatus, yielding a homogeneously dispersed brown solution. The consistent particle size and a high quantum yield of the fluorescent CDs were ensured by transferring the solution to an interception bag with an interception quantity of 3000 Da. The interception bag underwent a 24 h purification process in ultrapure water at ambient temperature. Afterward, the solution was refrigerated for 12 h, followed by drying the samples through a freezer dryer, producing solids that exhibit commendable water solubility [42].

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