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Preparation of carbon quantum dots from cigarette filters and its application for fluorescence detection of Sudan I

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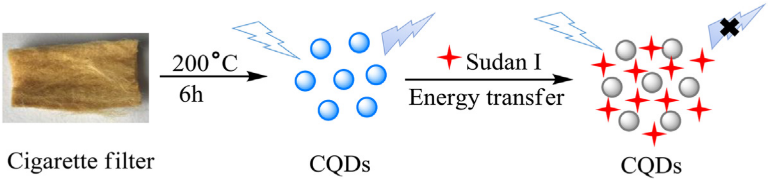
HIGHLIGHTS

- CQDs were synthesized by hydrothermal method using cigarette filters as carbon source for the first time.
- The obtained CQDs were used as a selective turn-off probe for Sudan I.
- Satisfactory results were achieved when the method was employed to the determination of Sudan I in food samples.

GRAPHICAL ABSTRACT



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ABSTRACT

Carbon quantum dots (CQDs) with quantum yield of 14% were successfully synthesized via a simple, low-cost, and green hydrothermal treatment using cigarette filters as carbon source for the first time. The obtained CQDs showed a strong emission at the wavelength of 465 nm, with an optimum excitation of 365 nm.Sudan I with maximum absorption wavelength at 477 nm could selectively quench the fluorescence of CQDs. Based on this principle, a fluorescence probe was developed for Sudan I determination. Furthermore, the quenching mechanism of the CQDs was elucidated. A linear relationship was found in the range of 2.40–104.0 μmol/L Sudan I with the detection limit (3σ/k) of 0.95 μmol/L. Satisfactory results were achieved when the method was submitted to the determination of Sudan I in food samples.

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1. Introduction

Food colorants, either natural or synthetic, are a very important class of food additives. Compared with natural colorants, synthetic colorants are widely used in food industry because of their advantages such as good stability, excellent water solubility, strong coloring power and low cost. However, because synthetic colorantspose a potential risk to human health, the use of food colorants is strictly controlled by laws and regulations in all countries. To date, Various techniques including UV–Vis spectrometry [1,2], fluorescence emission spectrometry [3,4], thin-layer chromatography [5,6], high-performance liquid chromatography [7–9], capillary electrophoresis [10] and electrochemical method [11] have been established for the detection of synthetic colorants. Sudan I (1-phenylazo-2-naphthol) is a synthetic azo-colorant dye, which was previously used for coloring of various foods. Because of its high carcinogenic nature [12], Sudan I as a food additive is now strictly forbidden in many countries. Unfortunately, there are still many organizations that use Sudan I in their products due to its intense color and low cost. Furthermore, Sudan I still has been widely used in foods by some countries [13]. Therefore, monitoring the content of Sudan I in foods is of great significance to ensure food

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safety and consumer confidence.

Carbon quantum dots (CQDs) is a new kind of carbon nanomaterial, which exhibits strong fluorescence with tunable emission depending on the size and excitation wavelength. Compared with traditional semiconductor quantum dots and organic dyes, CQDs are superior in terms of water solubility, photostability, toxicity and biocompatibility. Various approaches have been developed for the preparation of CQDs with desired possible applications in chemical analysis [14–16], biosensing [17,18], fluorescent imaging [19,20], etc. Among reported methods, the oxidation of graphite powder need a large amount of strong oxidants like concentrated sulfuric acids or concentrated nitric acids which are hazardous, and multi-step operations involved [21]. Recently, much effort has been made in the preparation of CQDs via high temperature or microwave-assisted hydrothermal carbonization of carbon precursors such as citric acid [22], ascorbic acid [23] and glucose [24]. So far, simple and green approaches for the large-scale preparation of CQDs from discarded materials are still challengeable.

In the present study, we report a facile and economical hydrothermal process for the preparation of CQDs by using discarded cigarette filters as carbon sources. The as-prepared CQDs have a size around 8 nm and exhibit excitation wavelength-dependent photoluminescence with a high quantum yield of 14% at the strongest emission wavelength of 465 nm. Most importantly, because the emission spectra of CQDs was effectively overlapped with the absorption spectra of carmine, significant fluorescence quenching of the CQDs was found upon addition of different concentrations of Sudan I. Thus, the CQDs can serve as a fluorescent probe for detection of Sudan I with high sensitivity and selectivity. To the best of our knowledge, there are no previous reports on the preparation of CQDs using discarded cigarette filter. And the use of CQDs as fluorescence probe for determination of Sudan I has also not been reported.

2. Experimental section

2.1. Instruments and materials

The UV–vis absorption spectra and fluorescence spectra were obtained, respectively, with a UV-4802 (Unico, Shanghai, China) and a RF-5301PC spectrophotometer (Shimadzu, Kyoto, Japan). High-resolution transmission electron microscopy (Tecnai G2 F20 S-TWIN, FEI Company, USA) image was taken at an accelerating voltage of 200 kV. FTIR spectra were recorded on Nicolet iS50 FTR spectrometer (Thermo Scientific Co., Ltd. Madison, WI, USA). Size distribution of the CQDs was observed using a Zeta sizer Nano ZS instrument (Malvern, UK).

Sudan I was obtained from Adamas Reagent (Shanghai, Co., Ltd., China). The Britton-Robison (B-R) buffer solution with different pH values were prepared by adding various amounts of 0.2 mol/L sodium hydroxide solution into 0.04 mol/L mixture of phosphoric acid, ethylic acid and boric acid. All reagents were of analytical grade and used without further purification.

2.2. Preparation of CQDs

CQDs were prepared via a hydrothermal method with discarded cigarette filters as carbon source. In a typical procedure, the cigarette filters was first washed with water several times and then dried in oven at 60 °C till reaching weight no further change. 0.4 g pretreated cigarette filters were mixed with 40 mL of doubly deionized water (DDW) and then the mixture was transferred to a poly (tetrafluoroethylene)-lined autoclave (50 mL) and heated at 200 °C for a period of 6 h in an oven. After the reaction, the autoclave was cooled to room temperature. The solution was filtered through 0.45 μm membrane filter and diluted to 200 mL for further use.

2.3. Pretreatment of real samples

Chilli powder, chilli sauce and tomato sauce were selected as test samples because Sudan I may be added as a colorant into them. All samples were obtained from a local supermarket in Nanning, China. The samples were pretreated according to the method from a literature [25], with some modification. A sample of 6.0 g added with a certain amount of Sudan I (0, 2.5, 12.5, or 25.0 mg, respectively) were mixed with 50 mL of ethanol. After being sonicated for 20 min, the mixture was centrifuged at 10000 rpm for 10 min. The supernatant was collected and filtered through a 0.45 μm polytetrafluoroethylene filter membrane. The filtrate was diluted with ethanol to 100 mL, and 1.0 mL aliquot of the resulting solution was taken out for analysis according to the determination procedure.

2.4. Procedure for determination of Sudan I

The determination of Sudan I was performed at room temperature. In a typical assay, B-R buffer (pH = 6.09, 5.0 mL), CQDs solution (1.0 mL), and various amounts of Sudan I standard solutions (800 μmol/L, ethanol as solvent) were sequentially added to a series of 10 mL colorimetric tubes. Then the mixture solution was diluted with ethanol to the mark and thoroughly mixed. After 30 min, the fluorescence spectra was measured at $\lambda_{ex}/\lambda_{em} = 365/465$ nm, the slit widths of excitation and emission were set to 3 nm and 5 nm.

3. Results and discussion

3.1. Characterization of CQDs

The CQDs possess wavelength dependent fluorescent emission properties. Under different excitation wave-length, the corresponding surface state emissive trap will be dominant, giving excitation-dependent fluorescence spectra. As shown in Fig. 1, with the increase of excitation wavelength from 325 to 435 nm, emission peaks are red-shifted from 437 to 500 nm. This is believed that the giant red-edge effect is the reason for the strong dependence of the emission peak position upon the excitation wavelength [26]. And the emission intensity reached a maximum when excited at 365 nm.Fluorescence quantum yield (QY) of as-prepared CQDs is

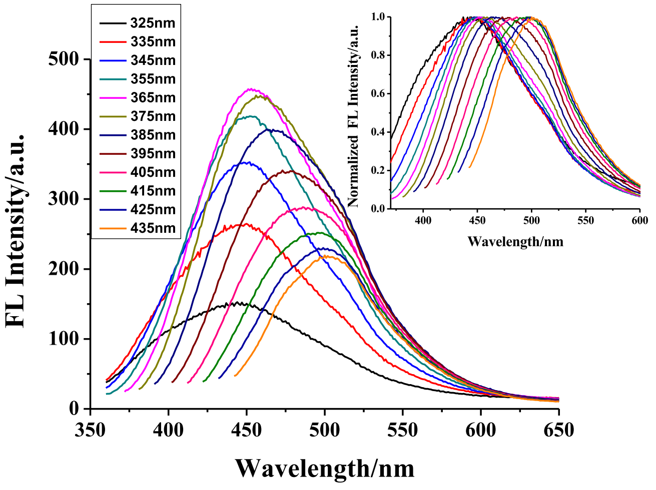

Fig. 1. The fluorescence spectra CQDs excited from 325 to 435 nm. Inset: normalized fluorescence spectra.

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