SYNTHESIS OF CARBON DOTS (CDs) *(Experiment)*

Introduction:

Carbon dots (CDs) are the newly discovered fluorescent nanomaterials with excellent biocompatibility and photostability. It can be synthesized under laboratory conditions by microwave mediated reactions. In this method, chitosan can be used as a carbon source along with poly(ethylene glycol) (PEG) as a passivating agent using sulphuric acid as a dehydrating agent. Microwave irradiation of the above mixture forms highly fluorescent CDs in a single step. Typical structure of CDs has been represented in Fig. 1.

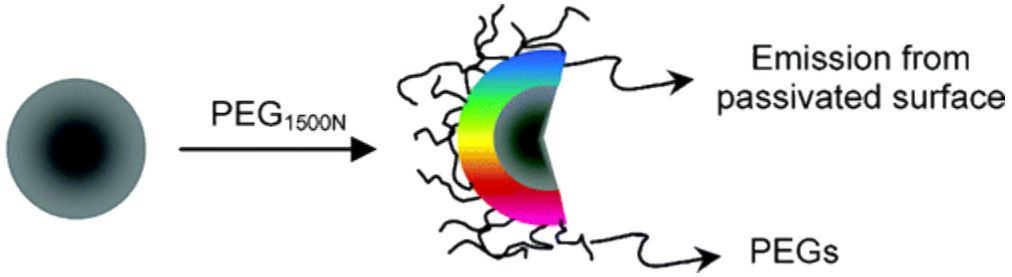

Figure 1 CDs with PEG species attached to the surface for passivation. (*Source: J. Am. Chem. Soc., 2006, 128, 7756–7757.)*

Chemicals required:

Chitosan, PEG-4000, Sulphuric acid (H_2SO_4) (98%), distilled water.

Synthesis of CDs by microwave pyrolysis method:

1. Add 0.2 g of chitosan was added to solution containing 25 mL of water and 4 mL of concentrated H_2SO_4.

2. Then add 0.2 g of PEG-4000 to the above solution and stir at 500 rpm for 15 minutes.

3. Subject the solution to microwave irradiation using a domestic microwave oven (IFB) operating at 100 % power level (700 W) for different cyclic times (20 s on,10 s off).

4. Allow the solution to cool naturally to room temperature.

5. Centrifuge the obtained dark brown solution at 14000 rpm for 15 minutes to separate the less fluorogenic, insoluble black deposit from fluorogenic, yellowish brown supernatant.

6. The yellowish brown supernatant is an indicate of formation of CDs

7. Check the presence of characteristic absorption peaks of CDs by UV- visible Spectroscopy, fluorescence of the CDs by fluorescence spectrophotometer, stability and size of the as-formed nanoparticles by Zeta sizer or Transmission Electron Microscope (TEM).

Characterization of AgNPs:

1.UV-Vis Spectroscopy:

Double click the "UV-VIS Analyst" software (Lasany UV-VIS LI-2800 double beam spectrophotometer, Germany).

⇩

Set scanning range for the sample as 200-700 nm at a scan speed of 480 nm/min.

⇩

Add 1mL distilled water to each cuvette and place inside chamber for blank calibration.

⇩

Go to UV Photometer icon. Select "Calibrate system baseline".

⇩

Select "Automatic blank calibration".

⇩

Replace one of the blank reference cuvette with 1ml diluted CDs solution.

⇩

Click on the "red color" start icon to record absorbance.

⇩

CDs show a characteristic absorbance between 200-400 nm, with characteristic peaks at 280 nm (π-π^* transition) and shoulder peak at 325 nm (n-π^* transition).

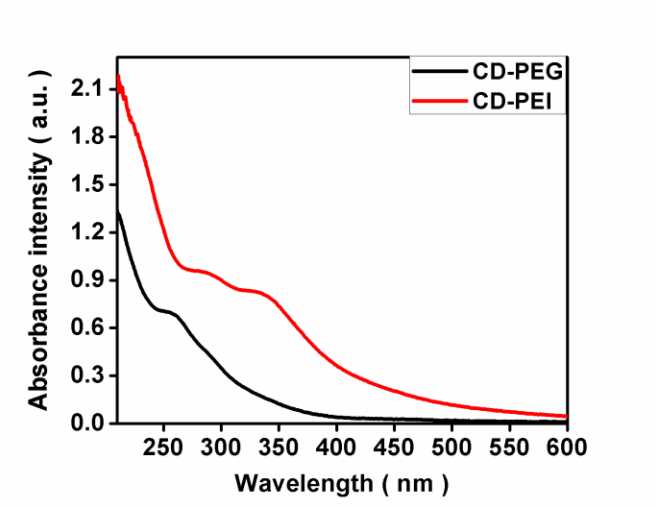

Figure 2: UV-Vis spectrum of Surface passivated CDs
(Source: RSC Adv. 2014, 4, 20915-20921.)

2. Zeta Potential and Size analysis:

The zeta potential of the silver nanoparticles can be measured according to the following procedure:

Double click the "DTS NANO Software" (Zetasizer nano-ZS90 series, Malvern Instruments Pvt. Ltd., Germany).

⇩

Add 750 µL of diluted CDs solution in a clear disposable zeta cell and place it inside the chamber at 25^0C.

⇩

Select "Zeta potential" and "Manual measurement" mode in the software.

⇩

Select "File" and click on "Save as" to save measurement file.

⇩

Click Measure and set parameters- '3' rounds of measurements; '0' equilibration time.

⇩

Press 'Green color' start button to measure zeta potential.

⇩

Analyze the zeta potential value of CDs.

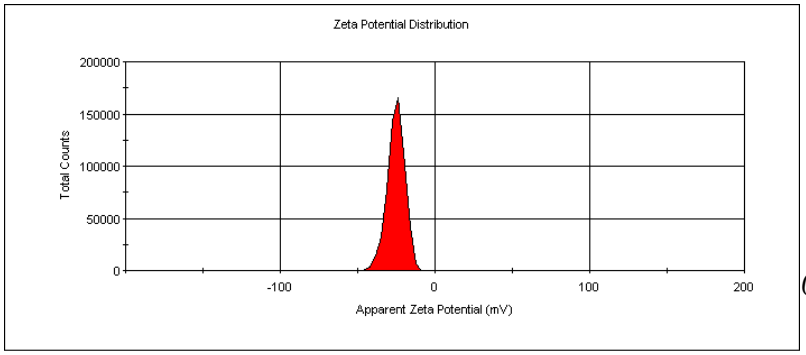

Figure 3: Surface zeta potential of CDs. The average zeta potential was measured to be −25.2 mV. *(Source: Chem. Commun., 2013,49, 8605)*

For size analysis, a similar procedure was followed and the sample was taken in polypropylene size cuvettes.

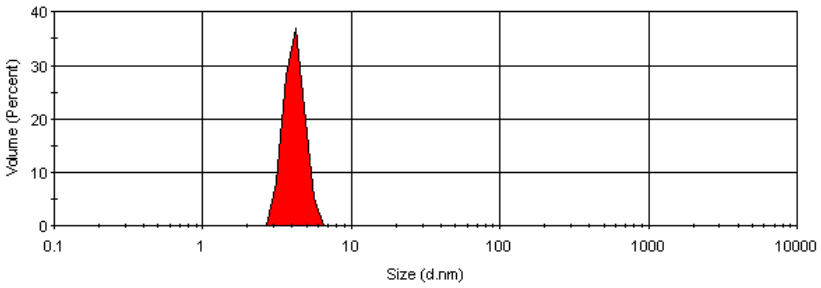

Figure 4: Size distribution by DLS. Average hydrodynamic size was calculated to be 4.15 nm. *(Source: Analyst, 2015, 140, 4260)*

3. Fluorescence spectroscopy:

Fluorescence emission of CDs can be recorded according to the following procedure**:**

Click the “FL-4600” software (Hitachi F-4600 fluorescence spectrophotometer).

⇩

Take 1 mL of diluted CDs solution in a fluorescence quartz cuvettee.

⇩

Set excitation wavelength as 320 nm and the emission range as 335-700 nm at a scan speed of 240 nm/min.

⇩

Adjust the excitation and emission slit widths accordingly.

Click on “PRESCAN”.

Select “MEASURE” to record the emission spectra.

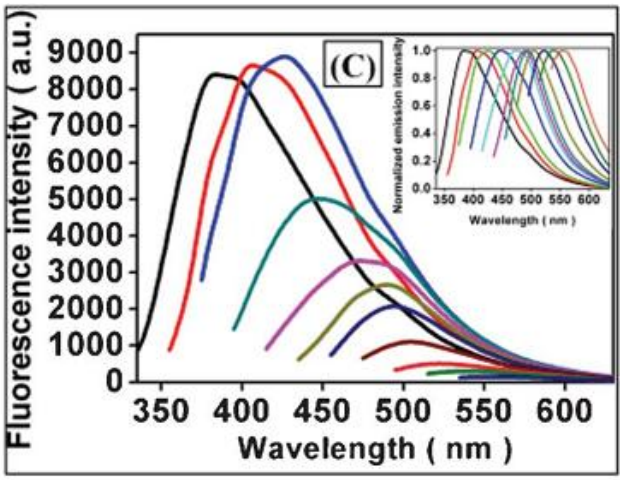

Figure 4: Emission spectra of CDs with increasing excitation wavelength from 320-520 nm in 20 nm increments. (inset: normalized emission spectral intensity).
(Source: RSC Adv.,2013, 3, 16958-16961)

4. Transmission Electron Microscopy (TEM) Analysis:

The average particle size of CDs can be determined by TEM.

Procedure:

1. Drop cast suitable dilutions of the samples onto the carbon supported copper grids.
2. Air dry the sample.
3. Images can be acquired by FEI Technai G2 TEM operating at 200 kV.

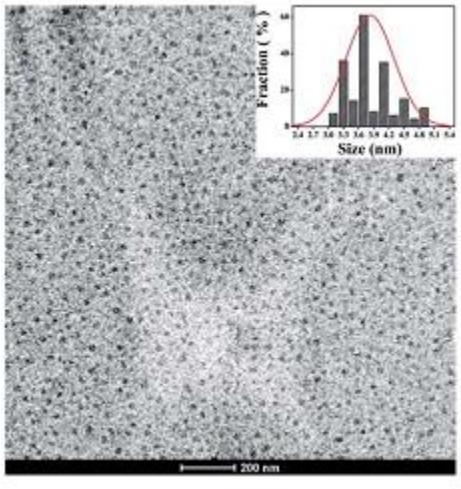

Figure 5: TEM image of CDs. Inset is the size distribution histogram
(Source: RSC Adv. 2014, 4, 20915-20921.)

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