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A CASE STUDY

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Solvothermal synthesis of chitosan quantum dots using different solvents

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ABSTRACT

In this current research, carbon quantum dots were synthesized by solvothermal synthesis method, using different solvents. The different solvents which were used for synthesis of carbon dots were 5-Sulphosalicylic acid, and Salicylic acid.In this method 1 per cent salicylic acid and 2 per cent 5-Sulphosalicyic acid was used with ethanol in hydrothermal bomb for 4 hours at 140°C. After the synthesis period a luminescence was seen with both the solvents. The solvents produced green, violet and blue fluorescence at different wavelengths of UV light.

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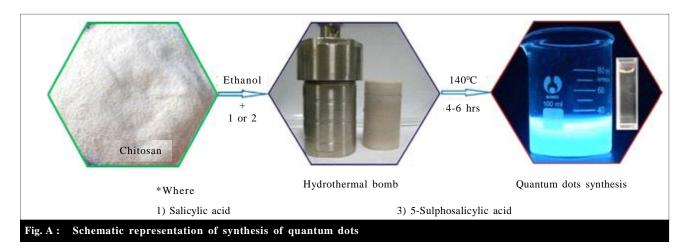
INTRODUCTION

Chitosan (CS) is a polysaccharide composed of glucosamine and N-acetyl glucosamine linked with a b-1-4- glycosidic linkage (Crini and Badot, 2008). Chitosan is non-toxic, biodegradable and biocompatible in nature (Govindan *et al.*, 2012 and Anitha *et al.*, 2014) and derivative of chitin which is found on the outer skeleton of aquatic animals, and abundantly present in fungi and algae (Anitha *et al.*, 2014).

The term "hydrothermal" consisted of two different words (Hydro+thermal) Hydro means water and thermal means heat. It is the technique of crystallizing substances from high-temperature aqueous solutions at high vapour pressures. If the solution is water then process is known as hydrothermal synthesis and when any other solution (like ethanol) is used for the synthesis of crystals the process is known as Solvothermal synthesis.

Fluorescent carbon nanoparticles or carbon quantum dots (CQDs) are a new class of carbon nanomaterials that have emerged recently and have garnered much interest as potential competitors to conventional semiconductor quantum dots. A wide range of applications are there of the Carbon Quantum Dots, including chemical sensing, biosensing, bioimaging, drug delivery, photodynamic therapy, photocatalysis and electrocatalysis (Lim *et al.*, 2014).

CQDs were used for chemical sensing due to their



low toxicity, water solubility, high photostability and superior chemical stability (Lim *et al.*, 2014).

MATERIAL AND METHODS

Preparation of Chitosan Solution :

Chitosan solution was prepared by dissolving 0.5 g of chitosan with 49.5 ml of distilled water.

Add 1 ml (2%) of glacial acetic acid, while stirring followed by sonication for 15 min. This was then filtered to obtain a clear solution. (Baek *et al.*, 2008), as chitosan is soluble in slightly acidic medium. (Badaway and Rabea 2011) Glacial acetic acid gives H^+ ions to chitosan for protonation to break its polysaccharide chain.

Chitosan solution was kept on magnetic stirrer for continuous stirring for 4-6 hours.

Viscous chitosan solution was obtained after 6 hours.

Filter the chitosan solution, using a dry muslin cloth, to remove its impurities.

Synthesis of carbon quantum dots :

There were 2 different solvents were used with chitosan solution for synthesis of carbon quantum dots, which are listed below as different cases.

With salicylic acid :

0.1 per cent of salicylic acid (10 mg) was dissolved in 10 ml of ethyl alcohol (ethanol), keeping the beaker on the magnetic stirrer. After this 10 ml of chitosan solution was added. Stirring was done for 3-4 hours (Archana *et al.*, 2013).

With 5-sulphosalicylic acid :

0.2 per cent (20 mg) of 5-sulphosalycylic acid was

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dissolved in 10ml of ethanol, keeping the beaker on magnetic stirrer (Fig. A). After this, 10 ml of chitosan solution was mixed with the solution (Archana *et al.*, 2013).

After complete duration of stirring, solution was transferred in hydrothermal bomb, which was consisted of a Teflon tube and a steel jacket. Then hydrothermal bomb was placed in hydrothermal oven for 4 hours at 140 °C. Take out the hydrothermal bomb After 4 hours from oven and keep it for cooling at room temperature for 2-3 hours.

Centrifuge the obtained solution at 7000 rpm for 6 minutes to settle down the carbon quantum dots.

Visualise the solution under the UV light to check the fluorescence.

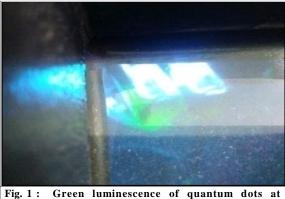
RESULTS AND DISCUSSION

The quantum dots were visualized under the UV lamp on different wavelengths. Appearance of quantum dots in Fig. 1 (with salicylic acid).

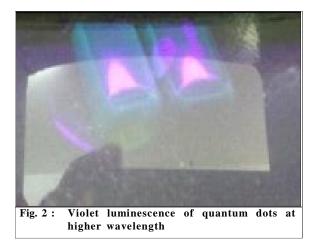
The chitosan solution with salicylic acid was brownish yellow in colour in visible light, (personal observation) and when projected under the short wavelength UV light (170 nm), it showed green luminescence. And further the same sample was visualized under higher wavelength UV light (300 nm), this time it produced violet luminescence.

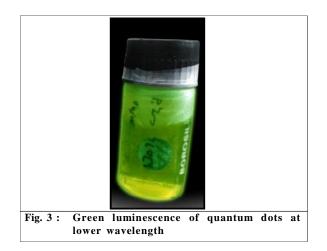
Appearance of quantum dots in sample 2 (with 5-sulphosalicylic acid):

The chitosan solution with 5-sulphosalicylicacid was brownish in colour in visible light, and when projected under the short wavelength UV light (170 nm), it showed



lower wavelength

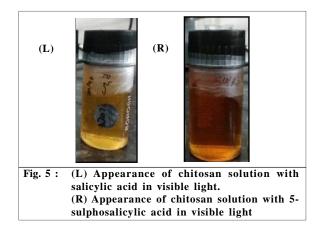




green luminescence. And further the same sample was visualized under higher wavelength UV light (300 nm), this time it produced Blue luminescence (Fig. 2). This luminescence property of the solution confirms the presence of quantum dots in the solution (Fig. 3, 4, and 5).



wavelength



This luminescence property of chitosan quantum dot solution confirms the synthesis of quantum dots in the solution.

Now for the further characterization process the sample can be analysed with SEM and TEM for the determination of quantum dots size.

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