Synthesis and X-Ray Photoelectron Spectroscopic analysis of carbon dots from strach

Dissertation submitted in partial fulfillment of the requirements of the Degree of Bachelor of Science in Chemistry Kerala University Trivandrum, Kerala.

Submitted by

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CERTIFICATE

This is to certify that the project report entitled " **Synthesis and X-Ray Photoelectron Spectroscopic analysis of carbon dots from strach** ” submitted by Ajay Arjun, Amritha S, Amrithalekshmi is a bonafide record of the work carried out by them under my guidance and supervision in partial fulfilment of the requirements for the award of the degree of **Bachelor of Science in Chemistry**.

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ACKNOWLEDGMENT

I take this opportunity to express our sincere gratitude to Mrs Siji K Mary, Department of Chemistry, Bishop Moore College Mavelikkara for herr guidance for completing the project successfully. I also like to express my gratitude to all other professors of the department of chemistry. I am deeply thankful to all the non-teaching staffs for their help and sincere co-operation. I would like to express our heartfelt gratitude to my parents as well as to my colleagues for their support and encouragement to complete this project.

DECLARATION

We hereby declare that the project work entitled " **Synthesis and X-Ray Photoelectron Spectroscopic analysis of carbon dots from strach "** submitted to Kerala university in partial fulfillment of Bachelor's Degree in Chemistry is Boniface record of the work carried out by the supervision and guidance of Mrs.Siji K Mary, Assistant professor, Department of Chemistry, Bishop Moore College, Mavelkkara. We further declare that, the dissertation has not formed the basis award of any Degree, Diploma, Fellowship or Associate ship or similar title of any University or institution

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1. Introduction

Carbon Dots(CDs) possess attractive properties like low cost, high biocompatibility, water solubility high stability, tunable fluorescent emission, luminescent, environment friendly and simple synthetic procedure[1]. CDs are attracting considerable attention for various applications in solar cells, light emitting diode, electronic displays and other optoelectronic devices, bioimaging, biosensing, nanoagricultural feild and disease detection. Several top down and Bottom up approaches like electrochemical oxidation , Chemical oxidation, ultrasonic treatments, microwave heating ,hydrothermal treatment, thermal decomposition have been developed for the synthesis of CDs[2–4][1]. Among different methods, hydrothermal synthesis is one of the cost effective technique which produces CDs with advances in tunable emission, high quantum yield and purity. Several green synthetic approach have been developed for the synthesis of fluorescent CDs by using inexpensive renewable resources as the precursor[5][6].

1.1 **Starch** ,

Starch is a white, granular, organic chemical that is produced by all green plants. Starch is a soft, white, tasteless powder that is insoluble in cold water, alcohol, or other solvents. The basic chemical formula of the starch molecule is $(C_6H_{10}O_5)_n$. Starch is a polysaccharide comprising glucose monomers joined in α 1,4 linkages. The simplest form of starch is the linear polymer amylose amylopectin is the branched form.Starch is manufactured in the green leaves of plants from excess glucose produced during photosynthesis and serves the plant as a reserve food supply. Starch is stored in chloroplasts in the form of granules and in such storage organs as the roots of the cassava plant; the tuber of the potato; the stem pith of sago and the seeds of corn, wheat, and rice. When required, starch is broken down, in the presence of certain enzymes and water, into its constituent monomer glucose units, which diffuse from the cell to nourish the plant tissues. In humans and other animals, starch from plants is broken down into its constituent sugar molecules, which then supply energy to the tissues.

Figure 1. Different sources of starch

Potato starch is starch extracted from potatoes. The cells of the root tubers of the potato plant contain leucoplasts. To extract the starch, the potatoes are crushed, and the starch grains are released from the destroyed cells. The starch is then washed out and dried to powder.

1.2 CARBON DOTS

Carbon dots (CDs), as a new type of carbon-based nanomaterial, have attracted broad research interest for years, because of their diverse physicochemical properties and favorable attributes like good biocompatibility, unique optical properties, low cost, eco-friendliness, abundant functional groups (e.g., amino, hydroxyl, carboxyl), high stability, and electron mobility. In this Outlook, we comprehensively summarize the classification of CDs based on the analysis of their formation mechanism, micro-/nanostructure and property features, and describe their synthetic methods and optical properties including strong absorption, photoluminescence, and phosphorescence. Furthermore, the recent significant advances in diverse applications, including optical (sensor, anti-counterfeiting), energy (light-emitting diodes, catalysis, photovoltaics, super-capacitors), and promising biomedicine, are systematically highlighted. Finally, we envisage the key issues to be challenged, future research directions, and perspectives to show a full picture of CDs-based materials.

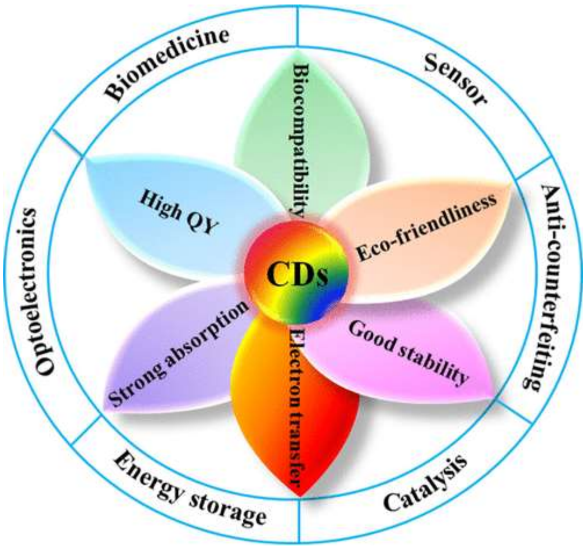


Figure 2. Applications of Carbon Dots

1.3 METHOD OFPREPARATION

Reaction of granular potato starch with urea and biuret resulted in the formation of products, which were soluble neither in cold nor boiling water. The net reaction was a monosubstitution of the hydrogen atom in one hydroxyl group in each D-glucose unit of starch with the either $CO–NH_2$ or $CO–NH–CO–NH_2$ moiety, respectively. Properties of the products, particularly these with urea, depended on the mode of reaction. Reactions were carried out in the microwave oven as well as with convection heating. The products retained the granular form of starch but a vast majority of granules were damaged. a-Amylolysis of those materials revealed that their susceptibility to the enzyme increasing in the order: starch < starch reacted with biuret < starch reacted with urea.. Susceptibility to α-amylolysis with simultaneous insolubility in water make these products suitable as ruminant fodder and, eventually, biodegradable material

1.4 PROPERTIES

CDs are fascinating nanomaterials with remarkable inherent physical and chemical properties that allow their application in diverse fields. Therefore, an in-depth study and understanding of the CD’s optical, physical, and chemical properties are essential to further improve its characteristics in order to broaden its applications.

1.4.1 Physical Property

1.4.2 Chemical Structure

Carbon dots usually are less than 10 nm with quasi-spherical nano-sized carbon particles. However, as mentioned earlier, synthesis routes dictate the various chemical structure of carbon dots. For instance, GQDs are anisotropic with a crystalline structure of one or more graphene layers. Different analytical techniques such as microscopic, spectroscopic, spectrometric, and diffraction methods are employed to confirm and ascertain the morphology, functional groups, size distribution, and crystalline nature of CDs. For example, the morphology of microstructure CDs-based lubricants following the ultrasonic approach was studied with TEM and HRTEM and found to be 2.38 nm on average and highly crystalline with 0.21 nm lattice spacing and

(100) graphene plane. The structural elucidation of defects and or graphitization could be further confirmed with Raman spectrometry by analyzing the G band and D band. By following the pulsed laser ablation synthesis method, nitrogen-doped GQDs were prepared and obtained a 3 nm particle

size by Neon and co-workers. The as- prepared N-GQDs exhibited 1565 cm^{-1} as G band and 1311 cm^{-1} as D band in Raman spectrum, confirming the disordered structure. XRD is a powerful instrument to characterize the physical state of the synthesized carbon dots. The amorphous character of CDs is observed as a broad hump, which is centered in and around~2θ = 26° in the XRD profile. Nearly spherical amorphous clusters of ~4–18 nm diameters were observed in the self-passivated CDs from dextrose by following the ultra- sonication approach .

1.4.3 Chemical and Optical Property

1.4.3.1 Ultraviolet-Visible Absorption

The basic chemical structure of CDs can be significantly elucidated by the typical UV- vis spectral analysis. The presence of π-π* (C=C) and n-π* (C=O, C-N, C-S, etc.) transition of the CDs skeleton indicates the type of surface functional groups, routes of CD synthesis, precursors, and chemical environment. For example, the absorption bands of CDs at around 273 nm and 342 nm may imply sp2 hybridization of the π electrons and n-π* transition, respectively. Again, the combination of the sam carbon but different nitrogen sources also influences the absorption' bands position**.** The presence of heteroatoms (such as N, O, P, B, S, and Se) in the CD's molecular structure also results in the fluctuations of the UV-vis peaks. The N, S-CDs fabricated from 3-aminothiophenol via a one-pot hydrothermal method showed two absorption shoulders at 298 nm, and 354 nm attributed to n-π* transition and heteroatoms N and S surface states defect, respectively .

1.4.3.2 Photoluminescence

The most impressive characteristic feature of CDs is the photoluminescent (PL) or the fluorescent property, which has allowed them to expand their field of applications. The PL of CDs is influenced by surface chemistry, quantum size effect, and molecular states of the carbon core. The up-conversion photoluminescence (UCPL) in the near infrared region carbon dots (NIR-CDs) may be because of the thermally activated electron transition from $S_{1\text{-edge}}$ to $S_{1\text{-Int}}$ in the excited state as shown in Figure 1I. The various synthetic approaches (as shown in Tables 1 and 2) along with different starting materials result in the generation of CDs with unique structures and different luminescent behaviors such as white, blue, green, yellow, red, and deep ultraviolet emission. Since the CDs are fabricated from variable carbon sources via different routes, the PL behavior also depends on the size, solvent, pH, and many more. Generally, due to the diverse electronic transition pathways, the CDs exhibit broad, symmetrical luminescence spectra across the whole wavelength scale. Various energy levels may be created by O-containing groups (named as O-related defect state), P- containing groups (named as P-related defect state), and N-containing groups (named as N-related defect state) on sp^2 hybridized carbons of the synthesized CDs as shown in Figure 1II. Moreover, compared to the quantum dots and other organic dyes, the CDs usually exhibit large Stokes shifts. The QY of the CDs depends on the surface chemistry and preparation methods. Yellow-emissive CDs from anhydrous citric acid and 2,3-phenanzinediamine were prepared that exhibited large Stokes's shift (188 nm), excellent stability, and 24% quantum yield . In another study, multi-color PL emissive CDs were synthesized from m-phenylenediamine (m-PD) and o- phenylenediamine (o-PD) and in the presence and absence of tartaric acid as the starting materials. Tartaric acid played a crucial role in tuning the surface state of CDs, such as the increase in surface oxidation and carboxylation. In the presence of tartaric acid (TA), the CDs from m-PD and o-PD exhibited green color and red color respectively whereas, in the absence of TA, CDs from m-PD and o-PD exhibited blue color and yellow-green color respectively. In particular, the red-CDs exhibited a high QY of up to 22.0%. The doping of CD changes the excitation-dependent PL. In a study, the emission of a bare CD that was found to be excitation-independent, showed a large red shift when it was doped with nitrogen. The reason is ascribed to the superimposition of blue (intrinsic molecular centers) and green emission bands (extrinsic molecular centers). In the emission process, the energy is transferred from the electron-hole pair formation at the intrinsic centers of the core to the extrinsic surface centers. Due to the surface defects and hybrid nanostructure in the mesoporous matrix, the contribution of the two bands modified, enhancing the tunability of the emission, thus promoting the green PL from blue emission. Besides, the QY of the CDs was found to vary, for the bare CDs (1.4%), N-doped CDs (22%),and purified N-CDs (28%). In another study by Kipnusu and co-researchers, nitrogen and boron doped carbon dots exhibited enhanced intersystem charge transfer (ICT) due to the presence of donor–acceptor moieties. Due to the ICT, the synthesized CDs produced triple colour emission as shown in the Figure 1III. The properties of PL are controlled by the surface functionalization of CDs as well. Recently, surface functionalization was shown to enlarge and narrow the band gap energy. The fluorescence of synthesized CDs could be quenched by tetracycline (TC) due to inner filter effect, whereas by introducing chlorotetracycline (CTC), blue-shift fluorescence was induced which may be due to enlarged energy band gap, and upon introducing oxytetracycline (OTC), fluorescence experienced red shift which could be because of the narrowed band gap as shown in Figure 1IV . In a recent report, the addition of a long-alkyl chain to the CDs promoted the emission of white luminescence under UV light (365 nm), which may be due to the inhibition of the aggregation- caused quenching effect. Additionally, the alkyl chains can effectively interact with the lipophilic fatty residues that can increase the potential applications of the developed white-CDs. Metal ion-dependent PL quenching of CDs has become a known phenomenon. In a report, the quenching mechanism has been identified as the photoinduced transfer of electrons from amine functional groups of the CDs to the respective metal ions.

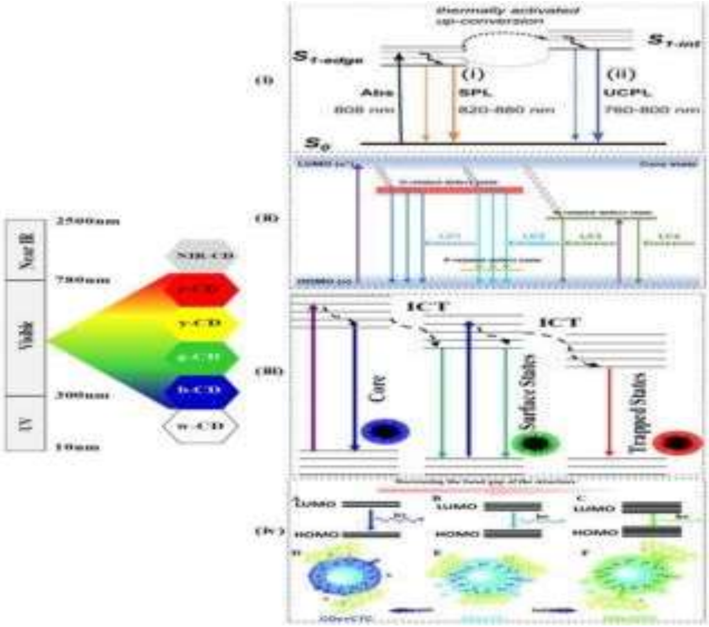


Figure 3. The band gap and transition of various color exhibited by CDs (near infrared CD: NIR-CD, red-CD: r-CD, yellow CD: y-CD, green CD: g-CD, blue CD: b- CD, white CD: w-CD). (**i**) Proposed mechanism of up-conversion PL in Near IR (NIR) CDs (Reproduced with permission), (**ii**) Proposed mechanism of CDs with O- defects, P-defects and N-defects states . (**iii**) Energy diagram of the CDs indicating the role of intersystem, charge transfer. (**iv**) Band gap transitions of CDs by introducing CTC, TC, and OTC .

1.5 APPLICATIONS OF CARBONDOT

The fascinating characteristics of C-dots has been uti lized in diverse applications. Carbon dots have various properties like chemical inertness, soluble, biocompatible and photoluminescence and multipurpose applications. Here we described some of the applications with effective results.

1.5.1 Detection of metals ions

Recently. Chen's group prepared a new type of C-dots for sensitive and selective detection of quercetin byusingN-(-aminoethyl)--aminopropylmethyl- dimethoxysilane and citric acid as starting materials 80 Phytic acid selec tively detected with linear range of 0.68-18.69 uM and a limit of detection of 0.36 uM utilizing the fluorescence off-on probe of C-dots 1. Tetracyclines can be selectively and efficiently detected upto a limit of 3.3 x 10-3 mol/L by C-dots derived by rose flower via microwave irradia tion method. Detection of TCs was based on the fact that fluorescent intensity of C-dots is considerably quenched by tetracycline

Ions of biological importance such as Fe^{3}+, Zn^{2}+, PO, S_20_2 have been sensitively detected employing on the carbon-dots (C-dots) based fluorescent off-on (Fe 5,0.2) and on-off (Zn^{2}+, PO,-) sensors for the detec tion of metal ions and anions 82. Besides it metal ions of biological importance and of environmental concern such as Cu2+ 67, Au(I)76, Hg2+ 83, Pb2+ (detection limit as low as 15.0 nM 184, Ag+, Pt(IV)85 can also be detected.

1.5.2 Cellular imaging

Due to excellent fluorescence, low toxicity and biocompatiability, C-dots can be used in cellular imaging and multimodal bioimaging of cells and tissues. The pos- sibility of using C-dots as fluorescent labels for cellular imaging was first demonstrated by Sun et al. who used PEG1500N passivated C-dots to non-specifically stain Caco-2 cells. Since then, non-specific intracellular im- aging of uptaken C-dots in other cell types have been shown, MCF-7 fibroblast cells7 etc. The confocal micro- graph showed brightly yellow emission in MCF-7 cells. It has also been found that the emission mainly locate at cytoplasm region, suggesting the C-dots can pass through cell membranes and enter into cells25

N.S co-doped C-dots were applied for biological ap- plications, they were directly applied in the imaging of peritoneal macrophages of mice without any further functionalization which represents that C-dots had penetrated into the peritoneal macrophages of mice. Further applications.

more, the photoluminescence intensity of the labelled macrophages showed no obvious reduction after continu- ous excitation for 20 min, suggesting that N. S co-doped C-dots possessed a remarkable photostability and low photobleaching and thus revealing the promising applica- tion of N,5 co-doped C-dots in cell labelling

Photocatalysis and photovoltaics: Carbon dots can also serve as a catalyst in many reactionsof biological and environmental importance. It is well known that Carbon dots have a broad range of absorption spectrum ranging spectral region from ultraviolet (UV) to NIP radiation, which enables Carbon dots to efficiently utilize the full spectrum of sunlight 89. Since the size of C-dots is very low it has very large surface volume ratio which is beneficial for absorbing more light and providing surface coordination sites in photocatalytic process. It result in augmented catalytic activity of Carbon dots based carbo- catalysts 90.91, Photoactive C-dots are both excellent electron donors and excellent electron acceptors as revealed by a recent study and electron transfer with C-dots can be induced by photoexcitation.

1.5.3 Drug delivery

C-dots due to its biocompatiability, excellent solubil Ity and low toxicity is effective drug carrier for therapeu tic payloads. But there are only few reports on C-dots as an efficient drug carrier. Due to high interstitial fluid pres sure entry of most drugs is very daunting task. One of the finest solutions to avoid this situation is to synthesize smallest possible nanoparticles which are able to carry large amount of drugs and unload it to solid tumors. Hence C-dots can serve as a better targeted drug release carrier. Recently, Mewada et al.63 have synthesized C-dots by microwave assisted heating of sorbitol using folic acid as navigational molecule (fC-dot) due to its widespread asso ciation with most of tumor cells. Before the exploitation of synthesized C-dots for targeted delivery of doxorubicin (DOX) to cancer cells, surface of C-dots was protected with bovine serum albumin (BSA) and thereafter DOX was grafted on it (fC-dot-DOX). At pH 5.8, there was dramatic change in the percentage DOX release after 4 h till 70 h where -14% drug was found to be released. The therapeutic use of C-dots is very promising for future application.

2. Review

Among different methods, hydrothermal synthesis is one of the cost effective technique which produces CDs with advances in tunable emission, high quantum yield and purity. Several green synthetic approach have been developed for the synthesis of fluorescent CD by using inexpensive renewable resources as the precursor[6,7]. Luminescent properties of CD can be improved by functionalization. Several research works were reported on the functionalsed CD and have some promising application in different fields[8,9]. In this work we functionalised the CD by using the nitrogen doping (partial substitution of carbon with nitrogen) which is highly enhanced photoluminescence[10] and urea was added for the functionalization of the carbon materials(starch powder) with nitrogen. CD synthesised via hydrothermal method and the precursors for the synthesis of CD involves the solution containing equal weight of starch powder and urea.

Carbon

dots (C-dots), a relatively new and promising candidate in the fluorescent nanomaterial category, possess a perceptible impact on various bioapplications. Herein, we report a one-step facile hydrothermal synthesis that yields a novel surface-passivated carbon dot (CDP) from curcumin (as a green substrate) displaying high aqueous solubility. The physico-chemical characterization of thus synthesized C-dots was accomplished by an UV−visible spectrophotometer, fluorescence spectrophotometer[6].

Carbon dots (CDs) synthesized from natural products have drawn numerous attentions due to some unique properties. Here, Prunus cerasifera fruits were used as carbon source to synthesize high luminescent CDs by hy- drothermal method. The obtained CDs were characterized by TEM, FTIR and XPS methods, founding the CDs were near-spherical and contained abundant nitrogen element[7].

3. Objectives

- To prepare carbon dot from the starch powder by using hydrothermal method
- UV Visible and fluorescence spectroscopic analysis of carbon dot.
- X-Ray Photoelectron spectroscopic analysis of carbon dot.

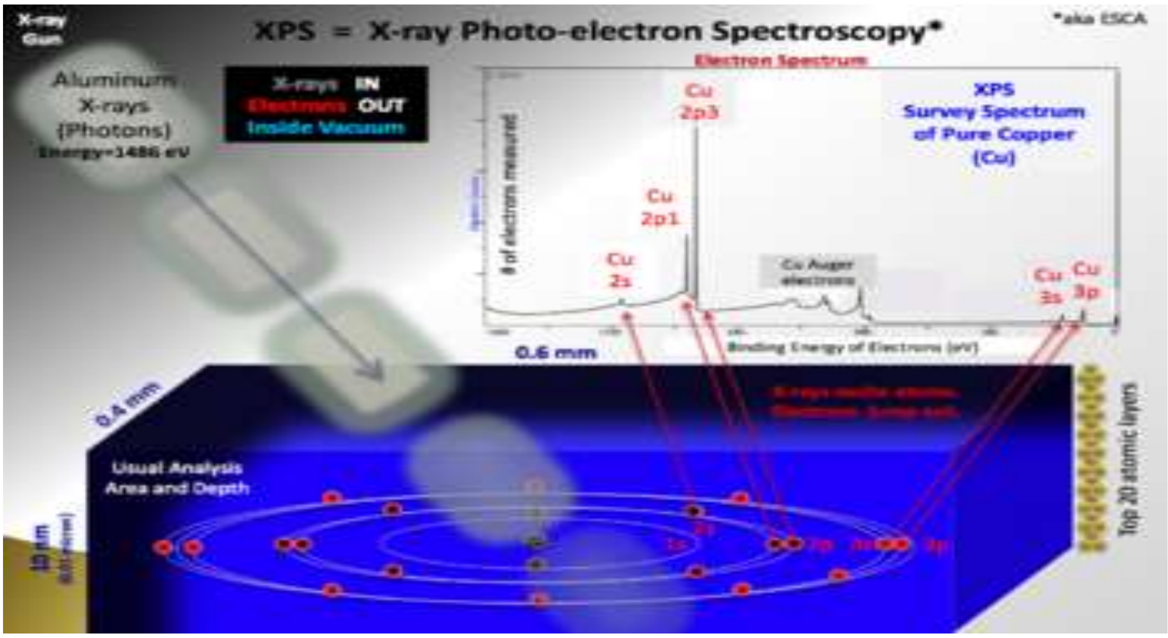
4. Materials and Methods

2.1 Materials

Corn starch powder, urea, glycerol, Potassium bromide, KBr (FTIR grade ≥ 99%) were purchased from sigma Aldrich. All chemical reagents included in this work have been of scientific grade and were used without any further treatment or chemical modification.

2.2 X-RAY PHOTOELECTRON SPECTROSCOPY

X-ray photoelectron spectroscopy (XPS), also known as electron spectroscopy for chemical analysis (ESCA), is **a technique for analyzing a material's surface chemistry**. XPS can measure elemental composition as well as the chemical and electronic state of the atoms within a material. XPS is the most widely used surface analysis technique because it can be applied to a broad range of materials and provides valuable quantitative and chemical state information from the surface of the material being studied. The average depth of analysis for an XPS measurement is approximately 5 nm. Spatial distribution information can be obtained by scanning the micro focused x-ray beam across the sample surface. Depth distribution information can be obtained by combining XPS measurements with ion milling (sputtering) to characterize thin film structures. The information XPS provides about surface layers or thin film structures is important for many industrial and research applications where surface or thin film composition plays a critical role in performance including: nanomaterials, photovoltaics, catalysis, corrosion, adhesion, electronic devices and packaging, magnetic media, display technology, surface treatments, and thin film coatings used for numerous applications. XPS is typically accomplished by exciting a samples surface with mono-energetic Al kα x-rays causing photoelectrons to be emitted from the sample surface. An electron energy analyzer is used to measure the energy of the emitted photoelectrons. From the binding energy and intensity of a photoelectron peak, the elemental identity, chemical state, and quantity of a detected element can be determined.



2.3 Synthesis of Fluorescent Carbon Dots

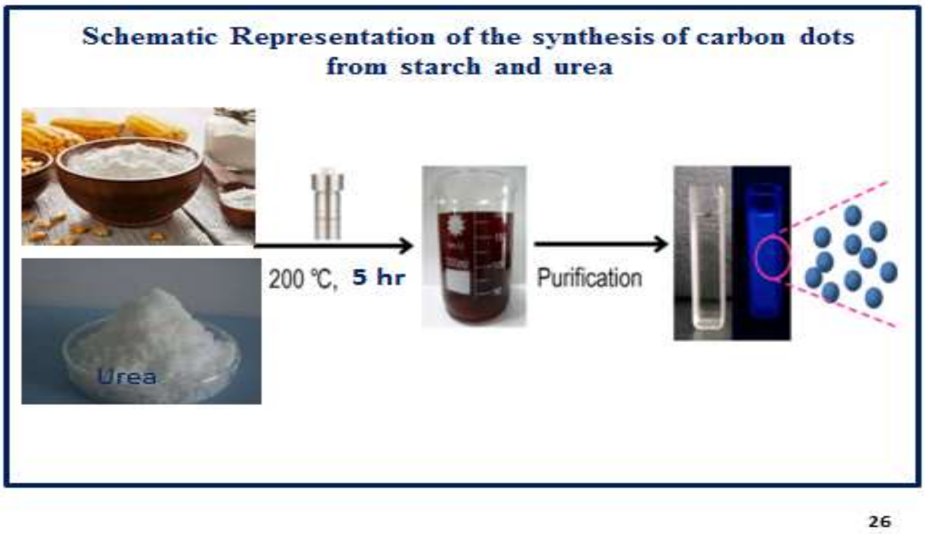


Figure 4. Schematic representation of the synthesis of carbon dots from starch and urea

2.5g starch powder and 2.5g urea were taken, added 100ml distilled water heated in a microwave oven for about 10 minutes. Repeat the heating until the solution turned to a brown color. Dissolve the brown residue in water and centrifuged. Acetone was added to the clear brown solution is then stirred by using a magnetic stirrer for 10 minutes. The solution is than placed in an open vessel for the evaporation of acetone. Removed the particles with bigger size by centrifugation.The solution is sonicated for 5 minutes.. The obtained solution was then placed in the UV chamber for observing the fluorescence of CD solution. UV light having wavelength 360nm is passed through the solution containing CD. The clear brown coloured solution is collected and kept 4°C(Figure.6).

5. Result and Discussion

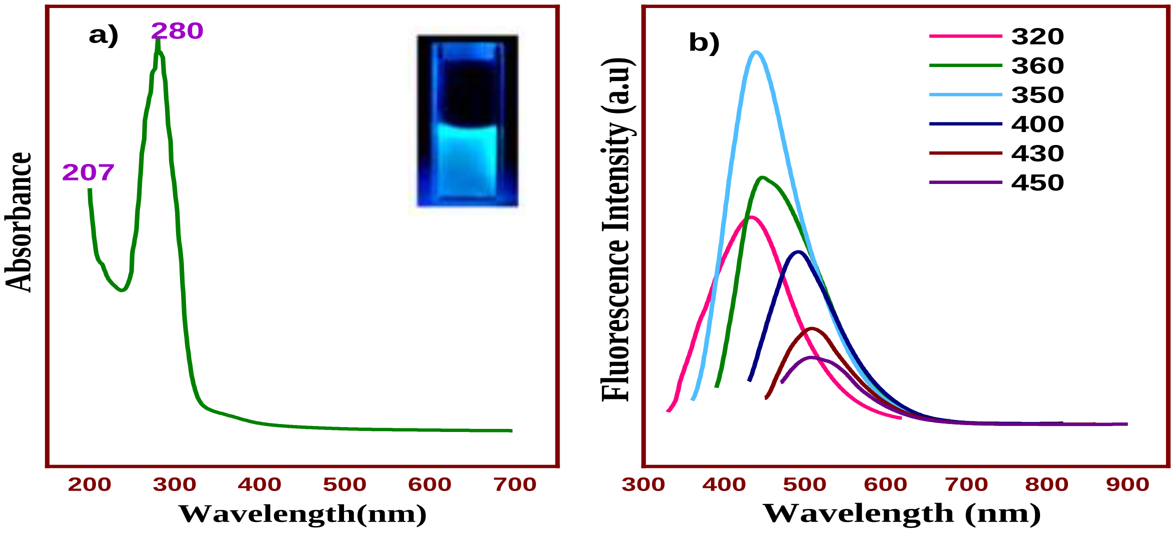


Figure 5. UV-Visible spectrum (a) and fluorescence emission spectrum in the region 320 to 450 nm (b) of CD: the inset shows the aqueous CD solution prepared from starch and urea, under UV-excitation at 365 nm in a UV-chamber

The solution exhibited light brown colour in daylight and bright blue colour by irradiating the sample in UV light having a wavelength 365 nm. A sharp peak at 280 nm observed in the UV-Visible spectrum **(Figure 5 (a)**) of CD solution was attributed to the n-π^* transition within C=O bonds, and the peak around 207 nm attributed to the presence of C-N bonds. The fluorscence emission spectra of the CD solution are shown in **Figure 5 (b).** The emission wavelengths of the CD shift toward longer wavelengths with the increase of excitation wavelength. The maximum fluorescence emission occurred at 440 nm, when the solution is excited at 350 nm. The emission peaks gradually shifted from 440 to 550nm with the increase in excitation wavelength from 320 to 450 nm[8]. Flourescence intensity increased with increase in excitation wavelength and then the intensity decreased[9].

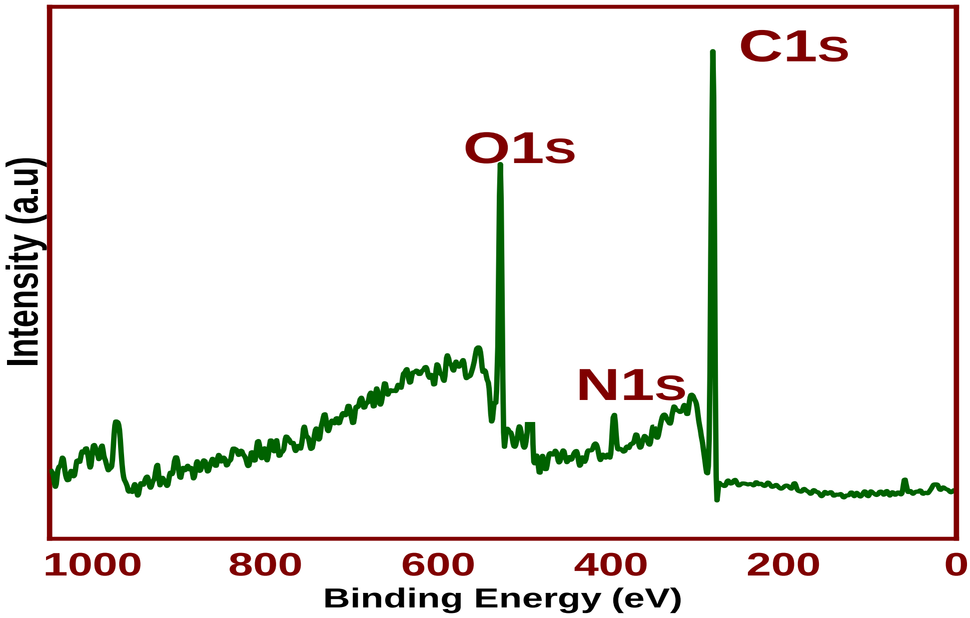


Figure 6. (a) XPS full spectrum of Carbon Dots

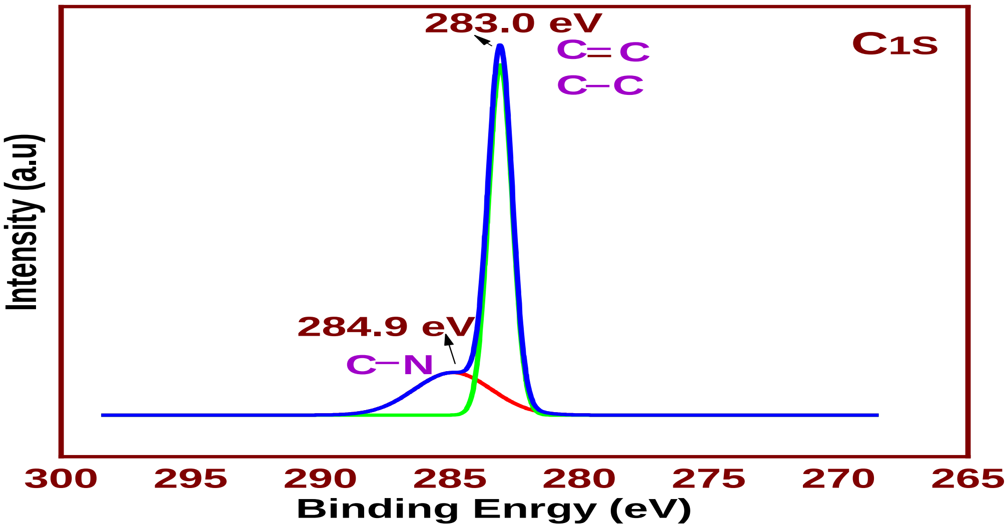


Figure 7. High-resolution spectra of C1s.

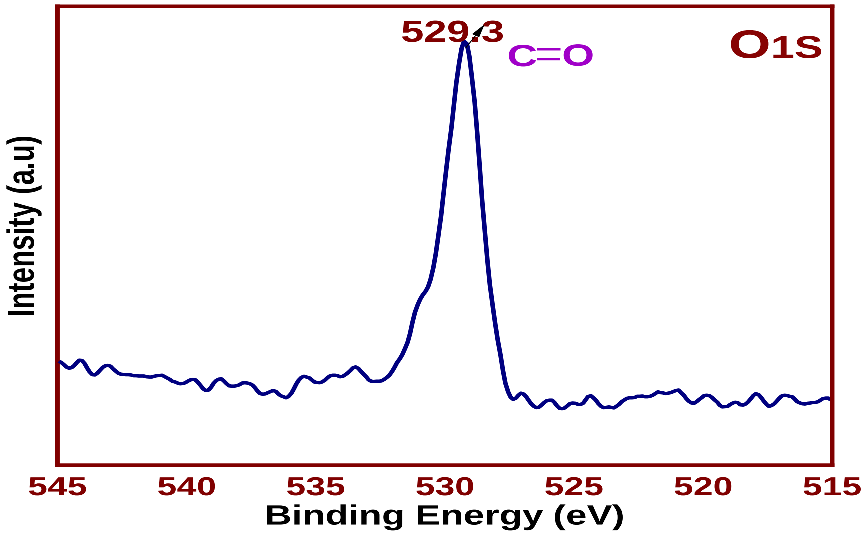


Figure 8. High-resolution spectra O1s

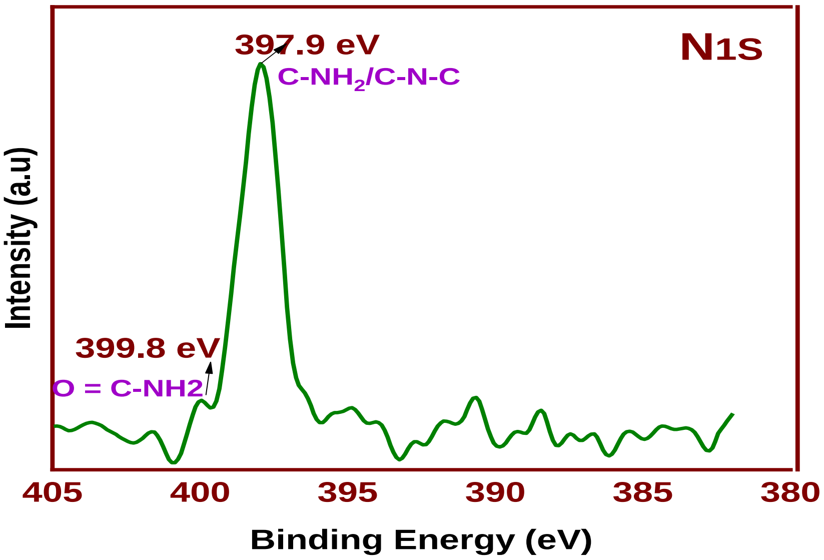


Figure 9. High-resolution spectra N1s

The surface functional groups and element analysis of carbon dots were analysed by XPS. As shownin **Figure 6** N-CDs displayed three predominant strong peaks at 283.0 eV, 397.9 eV, and 529.3 eV, which are ascribed to C1s, N1s and O1s peak, respectively. The elemental ratio of C:N:O is 73.25:: 5.65: 21.10. The C1S spectrum (**Figure 7)** of carbon dot particles could be deconvoluted in to 2 peaks (283.0 and 284.9 eV and attributed to the presence of C=C/C-C and C-N groups. The deconvoluted peaks of O1S spectrum From the high resolution O1s spectrum shown in **Figure 8**, the peak at 529.3 eV ascribed to C=O group. The NIs deconvoluted peaks (**Figure 9)** at 397.9 and 399.8 eV asribed to the presence of $C\text{-}NH_2/C\text{-}N\text{-}C$ and $O = C\text{-}NH_2$ groups.

6. Conclusion

The hydrothermal technique was used to synthesize fluorescent CD from starch and urea as a precursor. The excitation dependent emission properties with a pronounced red shift were observed in fluorescence studies. Structural analysis revealed that the prepared nitrogen functionalised carbon dot contains spherical particle having size 5nm. XPS analysis revealed that the elemental ratio of C:N:O is 73.25: 5.65: 21.10. The CD solution exhibited light brown colour in daylight and bright blue colour by irradiating the sample in UV light having a wavelength 365 nm. XPS Spectrum of CD displayed three predominant strong peaks at 283.0 eV, 397.9 eV, and 529.3eV, which are ascribed to C1s, N1s and O1s peak, respectively. In this work we developed facile and green method for the synthesis of fluorescent carbon dots(CD) by using the starch powder as the precursor via a hydrothermal method.

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