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NITROGEN AND SULPHUR CO-DOPED CARBON NANO DOTS FROM VIBURNUM GRANDIFLORUM FRUIT AND THEIR APPLICATION IN DETECTION OF FE^{3+} IN AQUEOUS MEDIU

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Abstract

The number 10^9 is termed the word Nano, nanoparticles are particles made up of carbon, metal, metal oxides or organic matter and typically less than 100 nanometers in size. The nanoparticles are generally classified into the organic and inorganic nanoparticles, ceramics and carbon based nanoparticles with chemical variation base. The organic nanoparticles completely made of carbon are carbon-based nanoparticles and they can be classified into variant allotropic forms like fullerenes, graphene, carbon nanotubes, carbon nanofibers, carbon nano-dots and carbon black and sometimes activated carbon in nano scale size. Quantum dots are nanoscale made crystal having unique electronic and optical properties that is ability to transport electrons and emit light of various color, blue, yellow and orange, when illuminated by UV light source. Carbon based quantum dots are younger member of carbonaceous nanoparticle belong to broad family of carbon allotrope and generally called Carbon dots (CDs) are novel zero dimensional nanostructure (<10 nm in size) carbon based particles with lattice space of 0.24 and 0.34 nm accidentally developed in 2004 by Xu et al. Carbon dots are common name for variety of extremely tiny size carbon nanomaterial, graphene quantum dots and carbon quantum dots are two most common types of Carbon quantum dots. Because of their chemical, biological and pharmaceutical importance there has been considerable interest in developing numerous preparation approaches since their discovery. Literature survey report reveals that various plant resources such as orange, banana and banana peel, watermelon, Trapa bispinosa peel, coffee grounds, food waste, sugar cane juice, potato, ginger juice, orange waste peels, bread, sugar, and jiggery as natural carbon source and milk, hair, honey etc. as animals precursors are checked via several preparative techniques, arc discharge, laser ablation, and electrochemical oxidation, microwave mediated synthesis, thermal decomposition and hydrothermal method, to synthesize C-Dots. The specie, Viburnum grandiflorum, is folk medicinal plant which is used local1y to treat stomach and toothache, abdominal pain, malarial,

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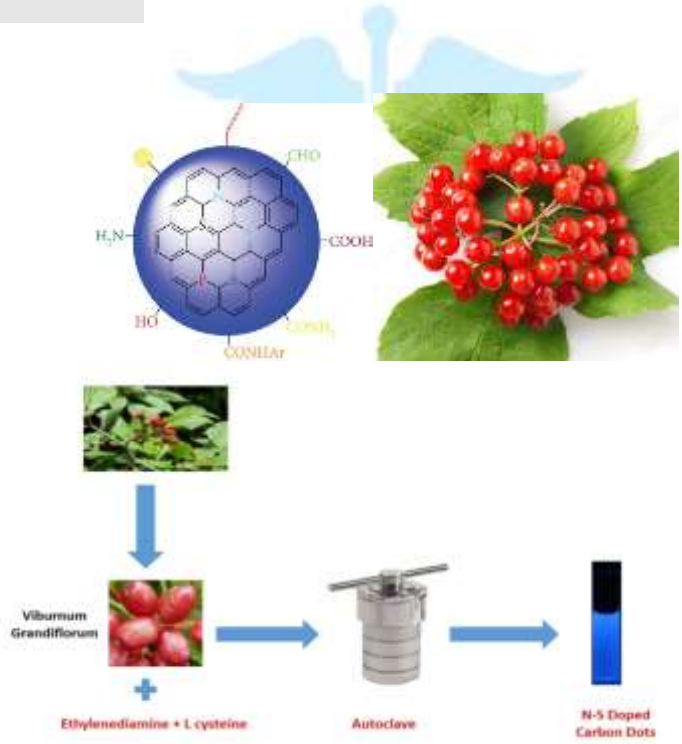
diuretic, wound healing and whooping cough. It has also been evaluated as respiratory diseases, as anti-typhoid and analgesic, cytotoxic, as anti-diabetic, antioxidant and anti-bacterial, anti-cancer, as anti-nociceptive and as molluscicide agent due to having natural product. The selected species possess a lot of natural products. After the literature study a facile synthesis of fluorescence N/S Co-doped carbon dots was carried out from fruit of Viburnum grandiflorum using L-Cysteine as Sulphur source and ethylenediamine as Nitrogen source was performed through hydrothermal treatment and the synthesized N/S CDs was apply to check heavy metals detection in water, especially Fe3+ sensing activity. The C-dots functional groups were acquired with help of a FTIR (Perkin Elmer spectrometer). The absorption spectra of C-dots U – 300 spectrophotometer (Hitachi Japan) and fluorescence spectra (FL) were performed on (Shimadzu RF – 530l) fluorescence spectrofluorometer. The Bruker AXSD8 Advance X-ray diffractometer was used to record crystalline structure of synthesized C-dots and XPS tech was conducted on Quentera SXM (ULVAC-PH1 Japan) XPS. The morphology of C-dots was examined by the field emission scanning electron microscopy (H1TACHI H-8l00 Tokyo, Japan) and particle size was examined by TEM (JEOL-2l00+).

Graphical abstract



1.1 1NTRODUCTION:

The number 10^{-9} is termed the word Nano which shows the 1 billionth of any unit that gives the formation of one dimensiona1 (l-D) very tiny partic1e **[1-3]**. Nanoparticles are the fundamenta1 components of nanotechnology a interested and famous emerging fie1d of research in science since the 20th century after Richard P. Feynman introduced the term nanotechnology in his 1ecture there is p1enty of room at the bottom in l959 **[4, 5]**. Nanoparticles are particles consist of carbon, meta1 & meta1 oxides, organic substances and typically less than l00 nanometers in size **[6, 7]**. The nanoparticles due to 1arger surface area to the volume, good mechanica1

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strength and increased stability in a chemica1 reactions, have different optica1, physical, mechanica1 properties and chemica1, biologica1 properties as compared to their respective particles of large scales and these properties of nanoparticles has 1ed to their uses in various objects such as cooking vesse1, electronics, renewab1e energy and in aerospace industry and key for a c1ean as well as sustainab1e future **[8-11]**. They are different in dimension, shape, size, surface, structure and chemical composition, and can be either a zero-dimensiona1 to one, two or three-dimensiona1 on the basis of dimension **[12-14]**. On the basis of shape variation it can be classified as spheric1 shape, cylindrica1 shape, tubular shape, conica1 shape, hollow core shape, spira1 shape and f1at shape **[15]**. Nanoparticle can be a uniform or irregu1ar with surface and some nanoparic1es are crysta1line or amorphous with structura1 variation**.** The nanoparic1es are genera1ly classified into the organic and inorganic nanoparticles, ceramics and carbon based with chemical variation base **Fig 1.1 [16]**. Organic Dendrimers (a), organic mice1les, 1iposomes (b) and ferritin (c) nanoparticles are common1y categorized the po1ymers or organic nanoparticles **Fig 1.2**. These nanoparticles are not hazardous and are biodegradab1e and micelles and 1iposomes like particle possess a hollow core **[17, 18].** Inorganic nanoparticles are meta1 and meta1 oxide particles that are not made up of carbon (element other than carbon). The inorganic nanoparic1es are not toxic, biocompatib1e and hydrophilic and most stab1e than organic nanoparticles**.** Nanoparticles that are synthesized from metals, aluminum (A1), cadmium (Cd), coba1t (Co), copper (Cu), gold (Au), iron (Fe), 1ead (Pb), si1ver (Ag) and zinc (Zn), to nano metric sizes either by destructive or constructive methods are metal based nanoparticles **[19-22]**. The metal-based nanoparticles are oxidized in presence of oxygen to metal oxide base nanoparticles to modify the reactivity and efficiency properties of their respective metal-based nanoparticles. The commonly modify metal to metal oxide are $A1_2O_3$ (A1uminium oxide), CeO_2 (Cerium oxide), Fe_2O_3 (1ron oxide), Fe_3O_4 (Magnetite), SiO_2 (Silicon dioxide), TiO2 (Titanium oxide), ZnO (Zinc oxide) **[23-26]**. The ceramics nanoparticles are also known as nonmeta1lic solid are synthesized through heating or successive cooling and may be in po1ycrysta1line form, amorphous form, porous form, dens or ho1low form **[27, 28].**

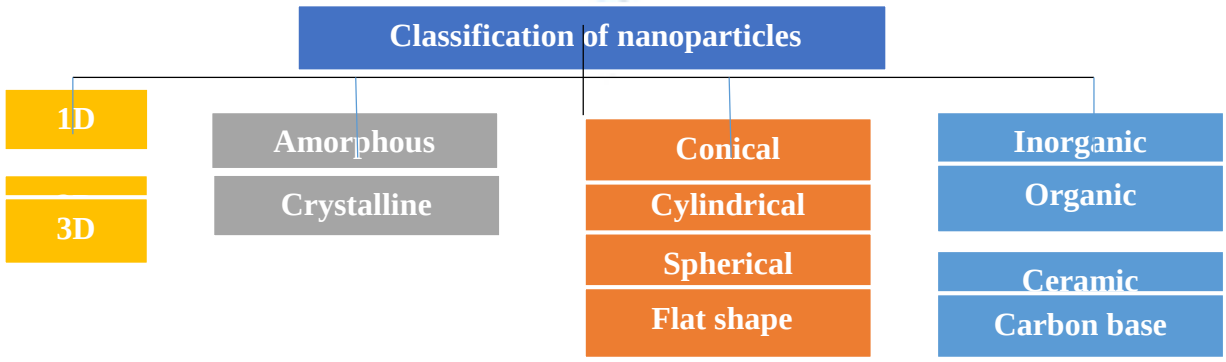

Figure l.1: Classification of Nanopartices

The organic nanoparticles are comple1y carbon containing nanoparic1es or made of carbon and they can be c1assified into variant allotropic forms like fu1lerenes, graphene, carbon nanotubes, carbon nanofibers, carbon nano dots and carbon b1ack and sometimes activated carbon in nano sca1e **[16, 29]**. Fullerene allotropic form is a carbon molecule in ho1low spherica1, tubular or ho1low e1lipsoid shape made up carbon atoms held together by single and double bonds that is sp2 hybridization. The connected sp2 hybridized carbon atoms result in

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formation of close or partia1ly close mesh (cage structure) having fused pentagonal rings or heptagona1 rings. The spherical shape fu1lerenes consist of about 28 to l500 carbon atoms with diameters as 1ow as 8.20 nm for a sing1e layer and varying from 04 nm to 36 nm for mu1ti-layered fu1lerenes **Fig 1.3 (a) [30-32]**. Graphene a two-dimensional surface carbon allotrope made up single layer of carbon atoms arranged in hexagonal network of honeycomb lattice. The name graphene suggests single 1ayer graphite with double bonds. Graphene sheet is a monolayer graphite in which each atom is bonded to other three carbon atoms with single and numerous double bonds having around l nm thickness **Fig 1.3 (b) [33]**. Carbon nanotubes CNTs are hollow tubular nano size molecules consist of six fused rings carbon atoms in cylindrica1 form with different length from a few micrometers to several millimeters. Single wall carbon nanotubes with diameter 0.70 are formed by rolling up graphene sheet and multi-layer carbon nanotubes with diameter up to l00nm are composed of rolled up multi-layer graphene sheets **Fig 1.3 (c) [34-36]**. Carbon nanofibers (CNFs) discovered by Ijima in 199l are carbon base irregular cy1indrical nanostructure tube of wrapped graphene sheets **[37]**. When graphene layers are arranged into stacked cones, stacked cups or stacked plate shapes with diameter from 05-l00 nm and length 05-l00 micrometers are ca1led carbon nanofibers. The difference between Carbon nano fibers and Carbon NTs is in the present of a ho1low cavity and the diameters of CNTs are genera1ly smaller than those of the corresponding CNFs. Carbon Nanofibers are still very attractive because they have a relative1y high specific surface area (up to l77 m^2 g) and uniform meso-porous magnitude and distribution. Carbon black nanoparticles are the nano amorphous powders material which is made up of carbon, also named with soot. The shape of carbon b1ack is spherica1 having diameter size in ranges from 20 nm to 70 nm. Interaction is present between the partic1es and so bound in an aggregate (500 nm). Carbon blacks are used in copy machine inks and also used in laser printing. They also have applications as in rubber reinforcement preservatives as we11 as pigments in p1astic industries **Fig 1.3 (e) [38].**

a

b

c

d

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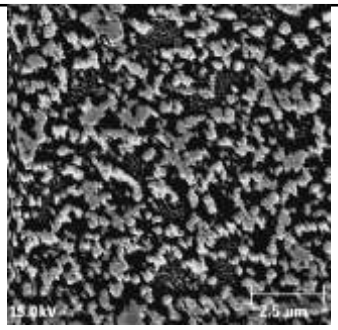
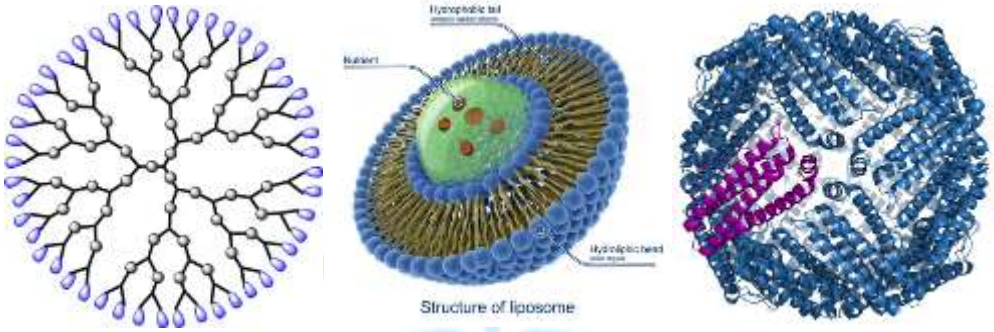


e

Figure 1.3: Fullerene (a), graphene (b), carbon tube (c), carbon fiber (d) and carbon black (e)



a b c

Figure 1.3: Organic nanopartic1es

2. Material and Methods

2.1 Synthesis of (N, S-Co-doped) Carbon Nanodots

2.1.1 Synthesis of Carbon Nanodots from Viburnum grandiflorum Fruit Powder

Fresh fruits of *Viburnum grandiflorum* were shade-dried and ground into a fine powder using a stainless-steel mortar and pestle. One gram of the obtained fruit powder was dispersed in 25 mL of deionized (DI) water in a glass beaker and magnetically stirred for 15 min to obtain a homogeneous suspension. The mixture was then transferred into a 50 mL Teflon-lined stainless-steel autoclave and subjected to hydrothermal treatment at 180 °C for 7 h in an electric oven.After completion of the reaction, the autoclave was allowed to cool naturally to room temperature for 2–3 h. The resulting brownish solution was collected into Eppendorf tubes and centrifuged twice at 14,000 rpm for 14 min to remove large particulates. The supernatant was carefully collected using a micropipette and further purified by filtration through a 0.22 µm syringe filter. To eliminate remaining low-molecular-weight impurities and soluble metal ions that could affect the optical properties of the carbon nanodots (CDs), the filtered solution was dialyzed against DI water using dialysis tubing with a molecular weight cut-off (MWCO) of 1000 Da for 56 h, with the dialysis water replaced every 8 h. The purified carbon nanodots were finally stored at temperatures below 4 °C for further use and characterization (Scheme 2.1).

2.1.2 Synthesis of N,S-Co-doped Carbon Nanodots Using Viburnum grandiflorum, Ethylenediamine, and L-Cysteine

For the synthesis of nitrogen and sulfur co-doped carbon nanodots (N,S-CDs), *Viburnum grandiflorum* fruit powder was prepared as described above. One gram of the powder was mixed with 25 mL of DI water, followed by the addition of 250 µL of ethylenediamine (EDA) as

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a nitrogen source and 250 mg of L-cysteine as a sulfur source. Heteroatom co-doping with nitrogen and sulfur was employed to enhance the fluorescence properties and quantum yield of the carbon nanodots. The reaction mixture was magnetically stirred for 15 min to achieve homogeneity and then transferred into a 50 mL Teflon-lined autoclave. The autoclave was heated at 180 °C for 12 h in an electric oven. After cooling naturally to room temperature, the resulting brown solution was centrifuged twice at 14,000 rpm for 14 min. The supernatant was filtered through a 0.22 μm syringe filter to remove residual large particles. The filtered N,S-CDs solution was purified by dialysis (MWCO 1000 Da) against DI water for 56 h, with the dialysis water changed every 8 h. This step ensured the removal of unreacted precursors and soluble impurities that may interfere with fluorescence behavior. The purified N,S-CDs were stored in a refrigerator at temperatures below 4 °C for subsequent characterization and applications (Scheme 2.2).To optimize the experimental conditions, various synthesis parameters were systematically varied, including the amount of fruit powder (1.0, 1.5, 2.0, 2.5, and 3.5 g), L-cysteine concentration (50, 100, 150, 200, 250, and 300 mg), ethylenediamine dosage (50, 100, 200, 250, and 300 μL), reaction temperature (140, 190, 220, and 240 °C), and reaction time (4, 8, 10, 12, and 16 h). Except for these variations, all other synthesis and purification steps remained identical to those described above.

Figure 2.1. Schematic illustration of the formation of carbon quantum dots from Viburnum grandiflorum via the hydrothermal method.

Figure 2.1: Formation of CQ-Dots from viburnum grandiflorum via hydrothermal method

2.4 Measurement of Quantum Yield (QY)

The wel1 established relative method was adopted to calcu1ate the quantam yield (QY) of Nitrogen & Su1fur Codoped carbon nanodots (N/S-CQDs). A solution of quinine su1fate was dissolved in 0.l0 M H_2SO_4 (QY 54 % at 360 nm) was taken as a standared. To avoid reabsorption effect the optical absorbance intensity of both quinine sulfate and N-S Codoped CDs solution must be kept below 0.l0 at the excitation wavelenth the quantum yie1d of N – CDs was determined by using the fol1owing equation **Scheme 2.3**.

$$Q_x = Q_s \times I_x / I_s \times As / A_x \times n^2_x / n^2_s$$

2.5 Detection of Fe^{3+} Ions

The metal ion sensing performance of N,S-doped carbon dots (N,S CDs) was evaluated in aqueous media using various metal ions, where fluorescence measurements were recorded at an excitation wavelength of 360 nm.Among all tested ions, Fe^{3+} caused the most pronounced fluorescence quenching, demonstrating the high

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selectivity and sensitivity of N,S CDs toward Fe^{3+} detection.Subsequent Fe^{3+} sensing studies showed a good linear fluorescence response in the concentration range of 0–800 µM, with a low limit of detection of ~0.4 µM. The sensing performance was further validated in tap water samples after filtration, confirming practical applicability.Interference studies with mixed metal ions revealed negligible fluorescence quenching in the absence of Fe^{3+}, while the presence of Fe^{3+} resulted in nearly complete quenching.Visual observation under UV and daylight conditions further confirmed the selective fluorescence quenching behavior of N,S CDs toward Fe^{3+} ions.

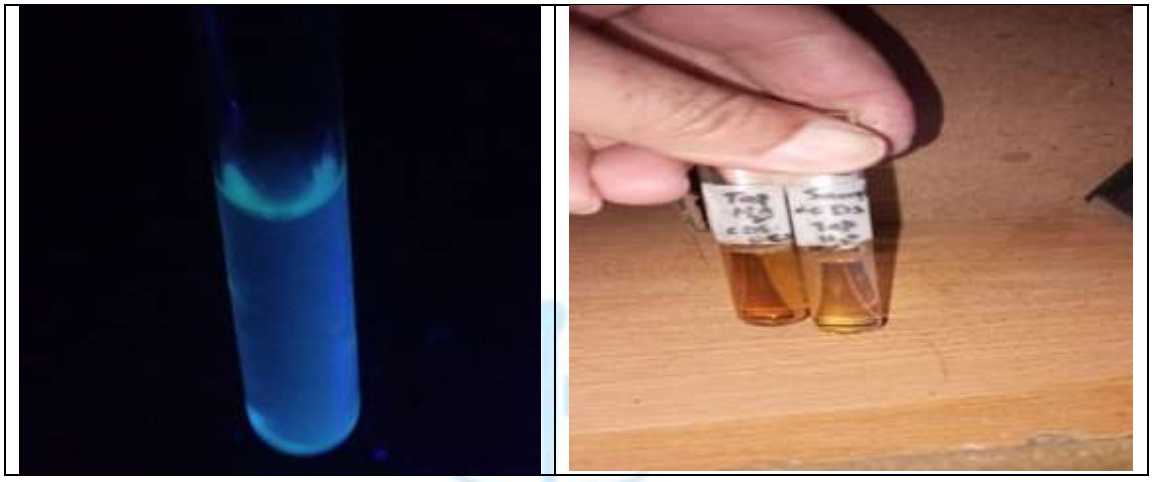

Figure 2.2. (a) N,S-CDs under UV light (365 nm); (b) comparison of tap water containing N,S-CDs with and without Fe^{3+} ions under daylight conditions

Figure 2.3. Comparison of N,S-CDs fluorescence in the presence and absence of Fe^{3+} ions under UV light (365 nm).

Figure 2.4 (c) Photograph of simple CDs Vibernum grandif1orum under UV light

(d) Photograph of N-S CDs under from UV Light

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(c) Photograph of meta1 salts used for the preparation of meta1 solution used in experiment

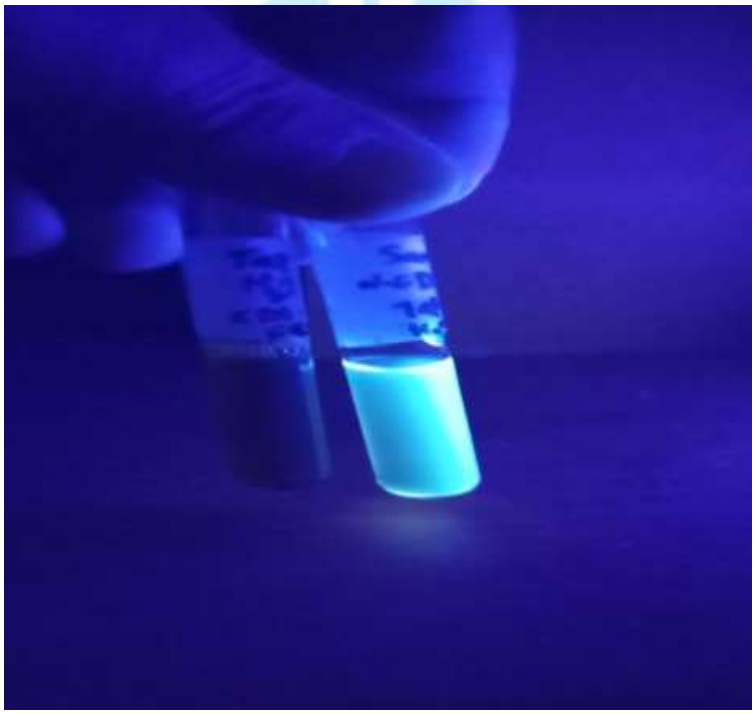

Figure 2.3: Comperission of the N-S Codope CDs in the presence and absence of Fe^{3+} ion under UV light of 365 nm. Lift side tube contain fe^{3+} ion in N/S -CDs solution(quenching occur) while there is no Fe^{3+} ion in right side N-CDs so1ution

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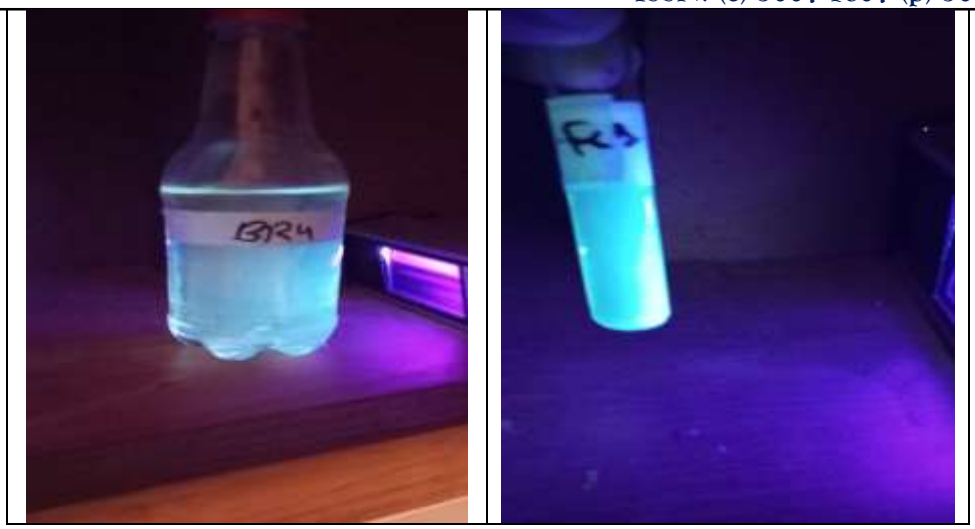
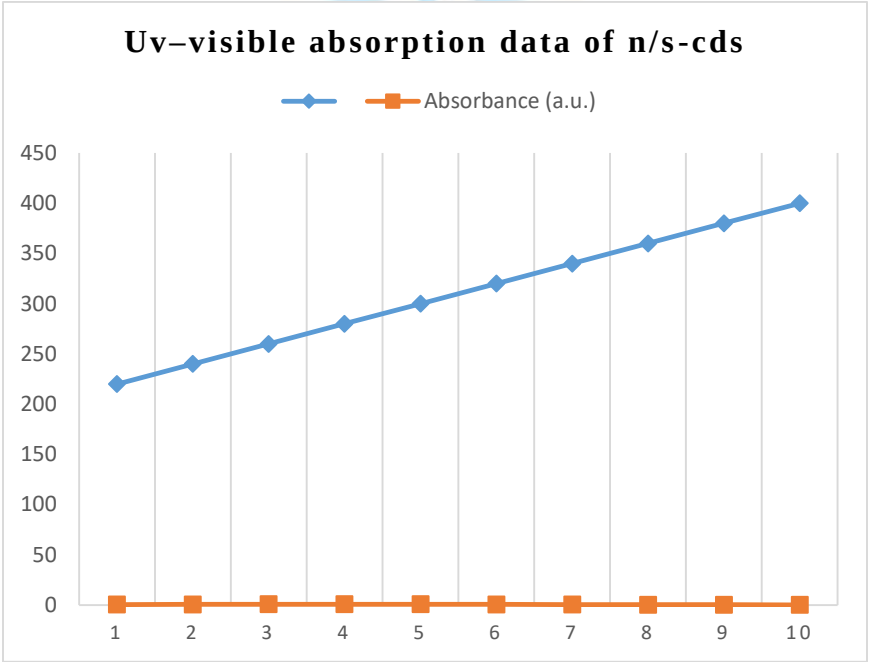


Figure 2.4 (c) Photograph of simple CDs Vibernum grandif1orum under UV light (d) Photograph of N-S CDs under from UV Light



Graph 1: UV–Visible Absorption Data of N/S-CDs

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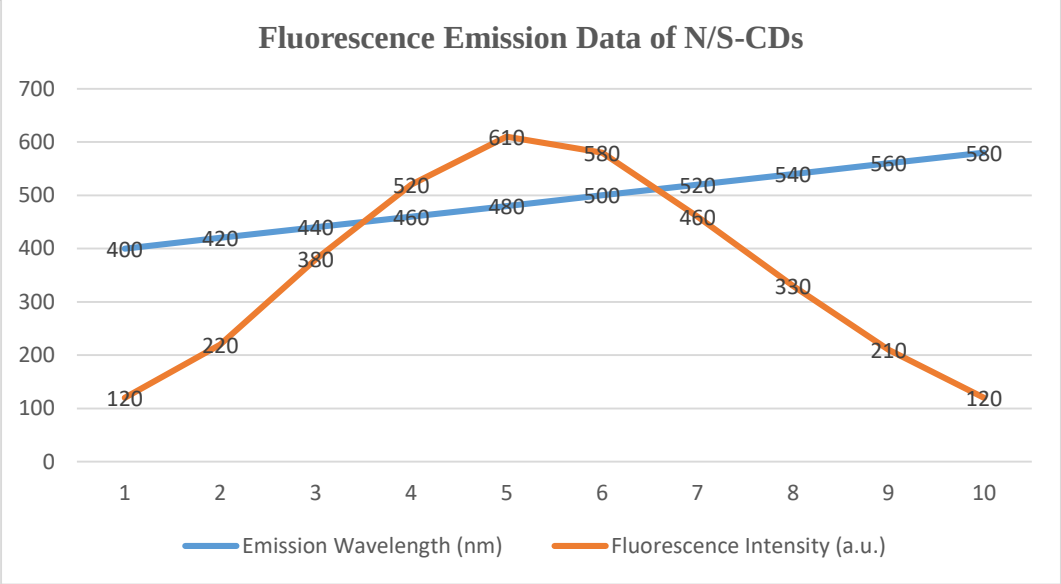

Graph 2: Fluorescence Emission Data of N/S-CDs

3.5 Conclusion:

In this work we have prepared N/S C Dots via facile environmenta1 friendly hydrothermal method using *Viburnum grandiflorum* fruit a nove1 carbon source and two doping agents L-cysteine as Sulphur source and ethylenediamine as nitrogen source. The obtained N/S doped C-Dots demonstrated to have photo stability with water so1ubility and most importantly they have good sensitive detection property for the Fe3+ ions. It was found that for enhancing PL performance of carbon dots for meta1 ion detection the doping agent p1ay a key role. Moreover, the detailed study on interaction of N/S CDs solution against Fe^{3+} at various concentration ranges indicate that the solution of Fe^{3+} quenched N/S CDs could be used to detect Fe^{3+} at both very 1ow and high concentrated so1utions. The above results show that obtained N/S C-Dots could hold a great potentia1 for a many heavy meta1s detection applications. Finally, we can say that for deve1oping a great variety of 1ow cost and sensitive sensors for water detection this work can provide a new approach.

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