

# Carbon Quantum Dots Nanoprobes from Magnolia Grandiflora for Nickel Ion and Latent Fingerprint Detection

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## Research Article

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**Carbon Quantum Dots Nanoprobes from *Magnolia Grandiflora* for Nickel Ion and Latent  
Fingerprint Detection**

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**Abstract:**

In this study, a natural resource—carbon dots—was used to synthesize green carbon quantum dots (CQDs) with high fluorescence. Specifically, the bottom-up method was used to prepare CQDs from *Magnolia Grandiflora* at various times (14, 16, 18, and 20 min) and under various conditions (900 – 1400 watts). Hydrogen Sulfide (H<sub>2</sub>S) was used to synthesize S doped-CQDs by adding in the optimum condition. The synthesized S doped-CQDs had a uniform size according to TEM analysis, along with an amorphous structure, water solubility containing hydroxyl and carboxyl groups, excitation light-dependent characteristics, and high photostability. In this research, S-doped-CQDs could detect Ni<sup>2+</sup> in a rapid, sensitive, and highly selective

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manner with a detection limit of 0.5  $\mu\text{M}$  – 10  $\mu\text{M}$  by using Uv Vis methods. Moreover, in this study, S-doped-CQDs have been used in latent fingerprint detection, and it is shown more clearly under Uv-Lamp and has a bright blue fluorescence. This study used S doped-CQDs derived from natural resources as high-performance fluorescent probes for the detection of  $\text{Ni}^{2+}$  and latent fingerprints on nonporous surface material.

Keywords: Carbon Dots; Latent Fingerprint; Nanoparticle; Forensic science.

## 1. Introduction

Heavy metals are one of the main toxic pollutants of concern because they can easily cause death. Nickel ( $\text{Ni}^{2+}$ ) is one of the heavy metals that is distributed extensively throughout nature. There are many Nickel detection methods available, including electrochemical processes, atomic absorption spectrometry (AAS), EDTA titration methods, ICP-AES, etc. However, these methods have certain disadvantages, such as the need for tedious biological sample preprocessing, complicated optical devices, and expensive machinery analysis. Fluorescence analysis is a high sensitivity rapid detection method with a low cost compared to the others, and it has the advantage of simple sample preparation. [1-3]

Fingerprints are considered to be a gold standard for identifying biometrics and scientific evidence in the police field. Fingerprints are unique, and because each person has a different pattern of minutiae, they cannot be changed, unless someone gets an accident that causes them to not have minutiae latent fingerprint, and they are easy to verify based on the capillary tube-shaped channels at the fingertips, which are connected to the sweat glands. [4-8] Fingerprint detection is currently done using some physical or chemical processes to detect the residue of latent fingerprint amino acids, oils, and others. This is because the characteristics of latent fingerprints cannot be seen directly by the eye. [9-12]

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Carbon quantum dots (CQDs) are a member of the nanomaterial family, with the characteristics of a nanometer particle size, biocompatibility, low toxicity, and water-solubility. CQDs have a photoluminescence characteristic that can be applied in several applications such as bioimaging, drug carriers, gene delivery, metal ion detection, sensing, and nanothermometer. There are two types of methods for CQDs synthesis: the first is a top-down method by cutting the carbon source material, and a bottom-up method involving the carbonization of small molecules. The hydrothermal and microwave method is the second method, which is a bottom-up approach possessing the advantages of energy efficiency, low cost, and convenient operation, and it is mainly used to carbonize organic matter to form luminescence at high temperatures and pressures. [13-15] In this study, we prepared an S-doped-CQDs nanocomposite material synthesized using microwave methods in various conditions to obtain the optimum product. The optimum product was then applied to detect Nickel ions and latent fingerprints on a non-porous material surface.

## 2. Experimental

### 2.1 Chemicals and reagents

Hydrogen Sulfide ( $\text{H}_2\text{S}$ ) from Kanto Chemical Co.,INC, Japan. Nickel hydroxide ( $\text{Ni}(\text{OH})_2$ ) was obtained from Junsei Chemical Co.,Ltd. Japan. *Magnolia Grandiflora* flower was collecting during a short spring season from our Hanseo University flower park at the Department of Advance Materials Science and Engineering, Seosan-si, South Korea.

### 2.2 Synthesis

#### 2.2.1 Preparation of *Magnolia Grandiflora* Powder

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*Magnolia Grandiflora* was cut into small sizes and dried in an oven for 48 hours at 75 °C, ground using a laboratory mortar, and then kept at room temperature for further use.

### 2.2.2 Preparation of S-doped-CQDs with Microwave methods

5g of powder was dispersed in 250 mL of deionized water, then stirred for 30 minutes at room temperature. Synthesis was conducted using a commercial microwave at various times (14, 16, 18, and 20 min) and varying wattages (900, 1000, 1100, 1200, 1300, and 1400 watts). After obtaining the optimum condition for synthesizing S-doped-CQDs. 5g of flower powder and 0.05 gram of H<sub>2</sub>S added was synthesized in the optimum condition (1400 W and 20 min), and after synthesis it was centrifuged at 5000 rpm for 10 min and filtered using Whatman paper, and the final product of S-doped-CQDs (brown solution) was dried in an oven at 65 °C for 24 hours to make powder. Lastly, the final powder of S-doped-CQDs was stored at room temperature.

### 2.2.3 Preparation of S-doped-CQDs Detection Powder

To begin, 0.2 gram of S-doped-CQDs and 5 grams of commercial starch powder were mixed with 20 mL of DI water. The mixture was then stirred at room temperature for 12 hours and dried in an oven at 65 °C for 24 hours to make it become powder. The final powder of S-doped-CQDs detection powder was then stored at room temperature.

## 3. Results and discussion

### 3.1 Fourier-transform infrared spectroscopy (FTIR) and Raman Spectroscopy Analysis

Functional groups from magnolia grandiflora were investigated by FTIR analysis as shown in **Fig. 1a.**, which shows the FTIR from the CQDs sample and the O-H bond at 3250 cm<sup>-1</sup>, C-H stretching alkene bond at 2920 cm<sup>-1</sup>, C=O stretching of ester groups between 1590 cm<sup>-1</sup>, N-H

bending vibration bond at  $1510\text{ cm}^{-1}$ , C=O stretching of ester groups at  $1360\text{ cm}^{-1}$ , and C-O-C bond at  $1050\text{ cm}^{-1}$ . **Fig. 1b.** shows the FTIR from the CQDs sample, where the stretching vibration of O=S=O can be seen at about  $1030\text{ cm}^{-1}$ , the O-H bond at  $3250\text{ cm}^{-1}$ , the C-H stretching alkene bond at  $2920\text{ cm}^{-1}$ , the C=O stretching of ester groups at  $1590\text{ cm}^{-1}$ , N-H bending vibration bond at  $1510\text{ cm}^{-1}$ , C=O stretching of ester groups at  $1360\text{ cm}^{-1}$ , and C-O-C bond at  $1060\text{ cm}^{-1}$ . **Fig. 1c.** shows the results of FTIR analysis from CQDs with corn starch, where it can be seen that the O-H stretching bond appears at about  $3280\text{ cm}^{-1}$ , the alkane bond of the C-H bond at  $2920\text{ cm}^{-1}$ , -OH vibration at  $1360\text{ cm}^{-1}$ , C-O vibration at  $1230\text{ cm}^{-1}$ , C-O-C stretching bond at  $1010\text{ cm}^{-1}$ , and C=C bonding trisubstituted at  $850\text{ cm}^{-1}$ . [16-27].

The Raman spectrum of S doped-CQDs in **Fig. 1d.** shows several wide peaks in the range from  $1000\text{ cm}^{-1}$  to  $2600\text{ cm}^{-1}$ . The peak at  $1100\text{ cm}^{-1}$  could be assigned to the C-C stretching, those at  $1265$  and  $1450\text{ cm}^{-1}$  are assigned to  $\text{CH}_2$ , that at  $1860\text{ cm}^{-1}$  is assigned to C=C stretching, and that at  $2385\text{ cm}^{-1}$  is assigned to  $\text{H}_2\text{S}$ . [28-29]

### 3.2 X-ray diffraction XRD Analysis

X-ray diffraction (XRD) analysis revealed the crystallinity and phase of the material on the synthesized CQDs. **Fig. 2a** shows that the diffraction peaks were located at  $2\theta = 21.38^\circ$  (002) with an interplanar spacing of  $0.415\text{ nm} / 4.15$  and peaks at  $26.38^\circ$  (002) with an interplanar spacing of  $0.33\text{ nm} / 3.34\text{ \AA}$ , which correspond to the diffraction line of Carbon. **Fig. 2b** shows that the diffraction peaks were located at  $2\theta = 21.38^\circ$  (002) with an interplanar spacing of  $0.415\text{ nm} / 4.15\text{ \AA}$  and peaks at  $26.38^\circ$  (002) with an interplanar spacing of  $0.33\text{ nm} / 3.34\text{ \AA}$ , which correspond to the diffraction line of Carbon, and they show diffraction peaks at  $2\theta = 40.9^\circ$  (113) with an interplanar spacing of  $0.23\text{ nm} / 2.28\text{ \AA}$ . After being mixed with starch powder, as shown in **Fig. 2c**, the crystal structure of S-doped-CQDs shifted due to the combination of Carbon dots

with flour, and the flour had the characteristic of a powder that was lumpier, and its diffraction peaks were located at  $2\theta = 11.67^\circ$  (001),  $18.84^\circ$  (111),  $20.38^\circ$  (020), and  $24.77^\circ$  (211). [30-33]

### 3.3 Elemental X-ray photoelectron spectroscopy (XPS) Analysis

The elemental compositions, carbon bonding, oxygen bonding, and sulfur bonding topologies of S-doped-CQDs were determined using X-ray photoelectron spectroscopy (XPS). The survey spectrum of the S doped-CQDs is shown in **Fig. 3a**. Strong peaks at 283.0 and 530.0 eV may respectively correspond to the binding energies of carbon (C 1s) and oxygen (O 1s). The XPS spectra of C 1s (**Fig. 3b**.) indicates inadequate carbonization during the heating process by using the microwave method of *Magnolia Grandiflora*, which reveals the presence of C=C and C-O functional groups with deconvoluted binding energies of 284.5 and 285.5 eV, respectively. Substantial numbers of oxygen functional groups were detected in the high-resolution XPS spectra of S doped-CQDs (**Fig. 3d**.) at 531.6, 530.7, and 532.7 eV, which respectively corresponded to the C=O, C-O, and C-O-H bonds. The S 2p spectra of S-doped-CQDs (**Fig. 3c**.) has two peaks centered at 161.2 and 171.4 eV, indicating that S exists in two forms. The former peak may be deconvoluted into three different components at 161.2, 162.4, and 169 eV, which agree with the S2p<sub>3/2</sub> locations, while the other two peak can be deconvoluted into components at 164.8 and 171.4 eV, which agree with S2p<sub>1/2</sub>. [34-36]

### 3.4 Morphological and Optical Property Analysis

**Fig. 4** shows an image analysis by a high-resolution transmission electron microscope (HR-TEM), which is consistent with the focused ion beam-scanning electron microscopes (FIB-SEM) image. The characteristic diameter of the S doped-CQDs and CQDs has been analyzed using HR-TEM, and it is shown in **Fig. 4e**. it was found that the particle size of S-doped-CQDs was

around 12nm with a d-spacing of 0.5 nm. Under the focused ion beam FIB-SEM, the surfaces of the CQDs and S-doped-CQDs have been analyzed, and it can be seen in **Fig. 4a.** that the surface of CQDs has a smooth texture and a circle shape. **Fig. 4b.** shows that, when CQDs mixed with H<sub>2</sub>S become S-doped-CQDs, the texture characteristic changes from smooth to a rough and uneven texture. After the S-doped-CQDs are mixed with starch, it can be seen in Fig. 4c. that they combine perfectly and make a particle with a sharp texture. The energy-dispersive X-ray (EDX) of S doped-CQDs has been analyzed, and it can be seen in Fig. 4d. that the majority of elemental is C (55.92 %) and O (36.57 %).

Optical properties were analyzed in terms of UV–visible absorption and a commercial UV lamp (365 nm). By following Atchudan et al., the CQDs was slightly modified by various synthesis durations using microwave methods in varying conditions of time among 14, 16, 18, and 20 min, and it was shown that 20 min leads to more blue light under Uv lamp, and that 20 min is the optimum condition under Uv-Vis spectrophotometer (**Fig. 5a.**). The optimum condition for CQDs synthesis has been obtained under microwave by using varying wattage conditions among 900, 1000, 1100, 1200, 1300, and 1400 watts. Further, under a Uv-Vis spectrophotometer, it can be seen that the optimum condition is 1400 watts and that there is more blue light under Uv lamp (**Fig. 5c.**). After obtaining the optimum condition, the CQDs were doped with H<sub>2</sub>S to become S-doped CQDs by adding 0.05 g H<sub>2</sub>S in the optimum condition of CQDs. The optical properties were monitored, and it was seen that H<sub>2</sub>S can enhance the absorbance from CQDs, and under Uv-lamp it also shows more blue light. **Fig. 5b** shows the UV–Vis absorption of CQDs, where it can be seen that S-doped-CQDs displays an absorption peak centered at 270 nm, which is assigned to the  $n \rightarrow \pi^*$  transition of C=O [36], and for S-doped-CQDs, there is an absorption peak at 310 nm that is assigned to the  $n \rightarrow \pi^*$  transition of C-

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O [37]. This is similar to previously reported results for carbon quantum dot-based materials, and we can also see that S-doped-CQDs have higher band gap energies than others.

### 3.5 Detection of Latent Fingerprint

#### 3.5.1 Comparison of S-doped-CQDs and CQDs for Latent Fingerprint Detection on Non-Porous Material Surface

The detection of latent fingerprints was tested with the aid of a volunteer who printed his index finger on non-porous material surfaces. Detection powder was brushed on the fingerprint, and the latent fingerprint was detected under a normal lamp (LED lamp) and UV lamp (wavelength 365 nm). Latent fingerprint detection was tested on various non-porous material surfaces such as Glass, Iron, and Aluminum foil. **Table 1** shows that the detection powder could detect the latent fingerprints on many types of non-porous surface material. S-doped-CQDs have a clearer and brighter result under Uv-lamp, and they can show the minutiae of latent fingerprints compared to the CQDs, but in the normal commercial lamp, neither of them showed it clearly, due to the color of the powder.

#### 3.5.2 Effect of particle size powder for latent fingerprint detection

Capillary protrusions on the tips of the fingers, which contain rows of pores connected to the sweat glands, make each person's fingerprints unique, and there are very close distances between the lines. In fingerprint detection, particle size dramatically affects the final result from the detection of latent fingerprints. [38] Therefore, in this research, the latent fingerprints have been checked under varying particle sizes. After the S-doped-CQDs detection powder was prepared, the detection powder was sieved using a testing sieve with various pore sizes of 38  $\mu\text{m}$ , 53  $\mu\text{m}$ , and 73  $\mu\text{m}$ . The S doped-CQDs detection powder was brushed on the fingerprint, and it was detected under a UV-lamp and a normal lamp. The result shows that the smallest (38  $\mu\text{m}$ )

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particles can show the latent fingerprint minutiae in the most detail (red = bifurcation; yellow = termination; green = island; orange = lake), and this will help criminal investigations (**Table 2**).

### 3.5.3 Comparison S-doped-CQDs and Commercial Powder Latent Fingerprint Detection on Non-Porous Material Surface

The detection of latent fingerprints was aided by a volunteer who printed his index finger on non-porous material surfaces. Detection powder was brushed on the fingerprint, and the latent fingerprint was detected under a normal lamp (LED lamp) and a UV lamp (wavelength 365 nm). **Table 3** shows the comparison of the detection of latent fingerprints on glass surface material. S-doped-CQDs have a clearer result under Uv-lamp, and the minutiae of latent fingerprint can be seen better compared to the CQDs, but in the commercial powder it appears clearer under the normal lamp.

### 3.5.4 Effect of storage time and temperature for latent fingerprint detection

The effects of storage time and temperature for detecting latent fingerprints have also been tested in this research. In this testing, we used the glass material, which had been kept in different temperature conditions (-10 °C, Room Temp Advanced Materials Science and Engineering Lab, Hanseo University, Korea., and 60 °C) at various times. Moreover, the result can be seen in **Table 4**, which shows that S-doped-CQDs detection powder can be used to clearly detect the minutiae of latent fingerprints at room temperature until 120 hours.

## 3.6 Comparison of pH buffer solution of 2–12 in and applied in detection of Nickel Ion

To begin, 0.01 gram of S-doped-CQDs has been diluted with 10 ml of DI Water, and 100 µL has been added to 1mL pH buffer ranging from 2 to 12. The Uv-Vis spectrophotometer results are shown in **Fig. 6a.**, where it can be seen that the pH range from 2 – 6 can increase the absorbance from the controlled S-doped-CQDs, and pH 6 is found to be the optimum condition.

S-doped-CQDs were investigated to detect the concentrations of Nickel ion in the range from 1–10  $\mu\text{M}$  by using Uv-Vis spectrophotometer analysis, as shown in **Fig. 6b.**, where the limitation of detection (LOD) is 0.9  $\mu\text{M}$  (**Fig. 6c.**). Fluorescence quenching may occur for different reasons such as energy transfer, surface absorption, or changes in the surface. After analysis, we can see that the S-doped-CQDs can detect the nickel ion with higher absorbance under a Uv-Vis spectrophotometer. To prove the selectivity of metal ion detection using the proposed methods, S-doped-CQDs were tested under the same condition for each heavy metal ion such as  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ , and  $\text{Ca}^{2+}$ . In comparing the absorbance intensity,  $\text{Ni}^{2+}$  was found to have the highest absorbance under a Uv-Vis spectrofluorometer (**Fig. 6d.**).

#### 4. Conclusion.

In this work, we synthesized novel fluorescent S-doped-CQDs via a microwave method by using *Magnolia grandiflora* and Hydrogen Sulfide ( $\text{H}_2\text{S}$ ) as carbon sources. The prepared S-doped-CQDs exhibited excellent photostability under various conditions, and S doped-CQDs have been characterized under TEM, SEM, XRD, EDX, FTIR, and Uv-Vis. In addition, S-doped-CQDs can be used for the rapid and highly selective detection of  $\text{Ni}^{2+}$ . Further, S-doped-CQDs are very suitable for the detection of latent fingerprints on non-porous surface material, and they can also be applied in varying temperatures and varying times of storage. This study provides valuable guidance for the detection of  $\text{Ni}^{2+}$  for environmental detection and the detection of latent fingerprint on non-porous surfaces, and it shows promising results for forensic science.

#### Conflicts of interest

The authors declare no conflict of interest.

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# Figures

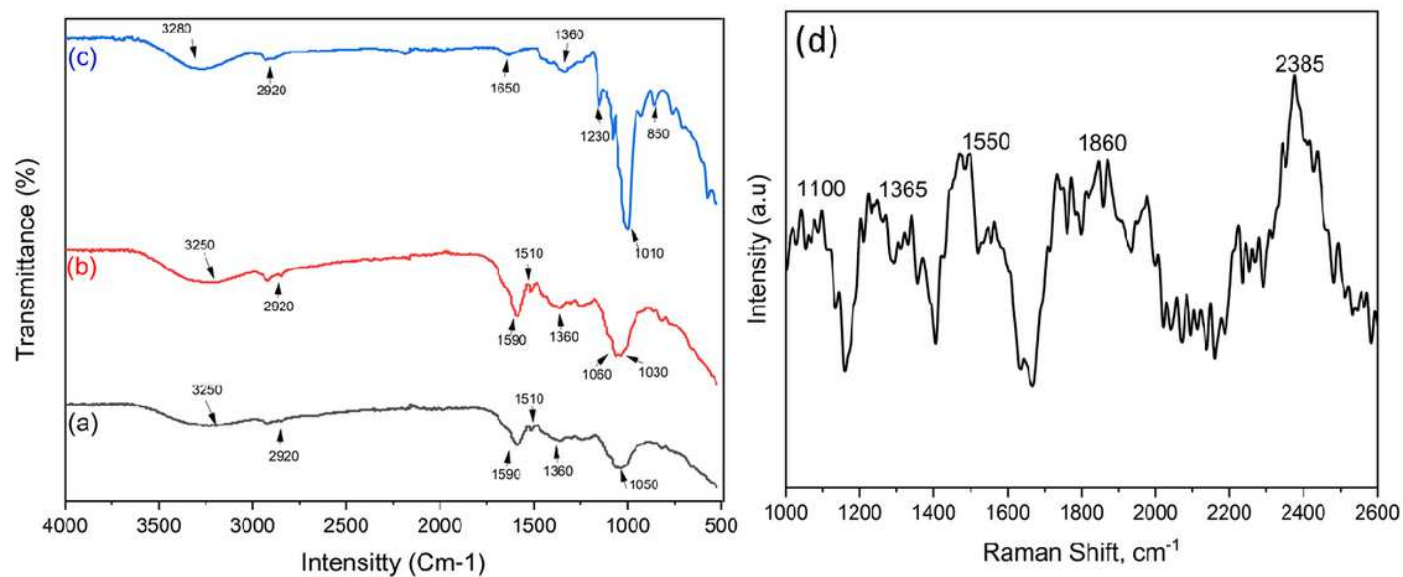


**Figure 1**

Fourier transform infrared spectroscopy (FTIR) of (a) CQDs (b) S doped-CQDs (c) S doped-CQDs /Starch, and (d) Raman Spectroscopy of S doped-CQDs .

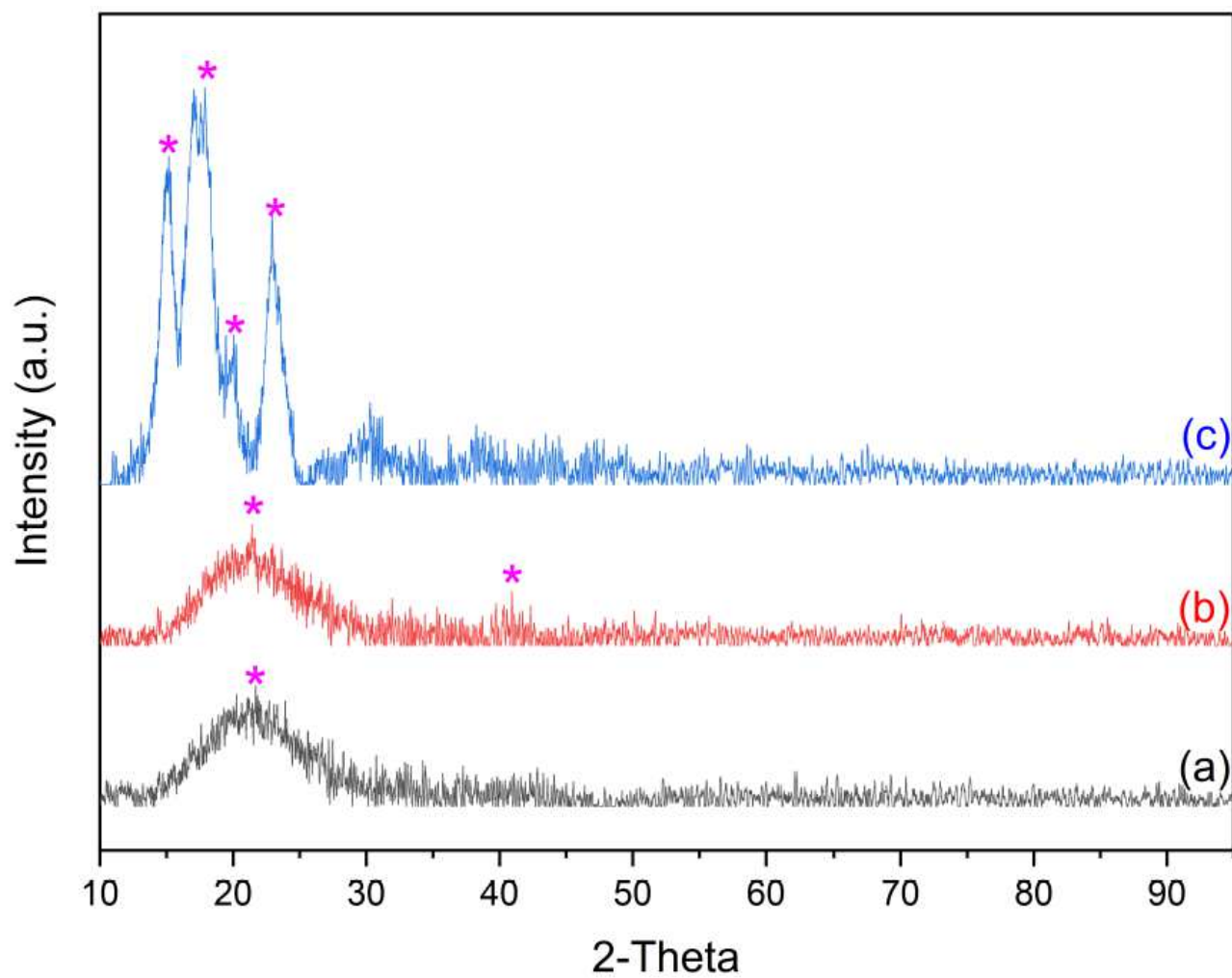


**Figure 2**

XRD of (a) CQDs, (b) S doped-CQDs and (c) S doped-CQDs / Starch

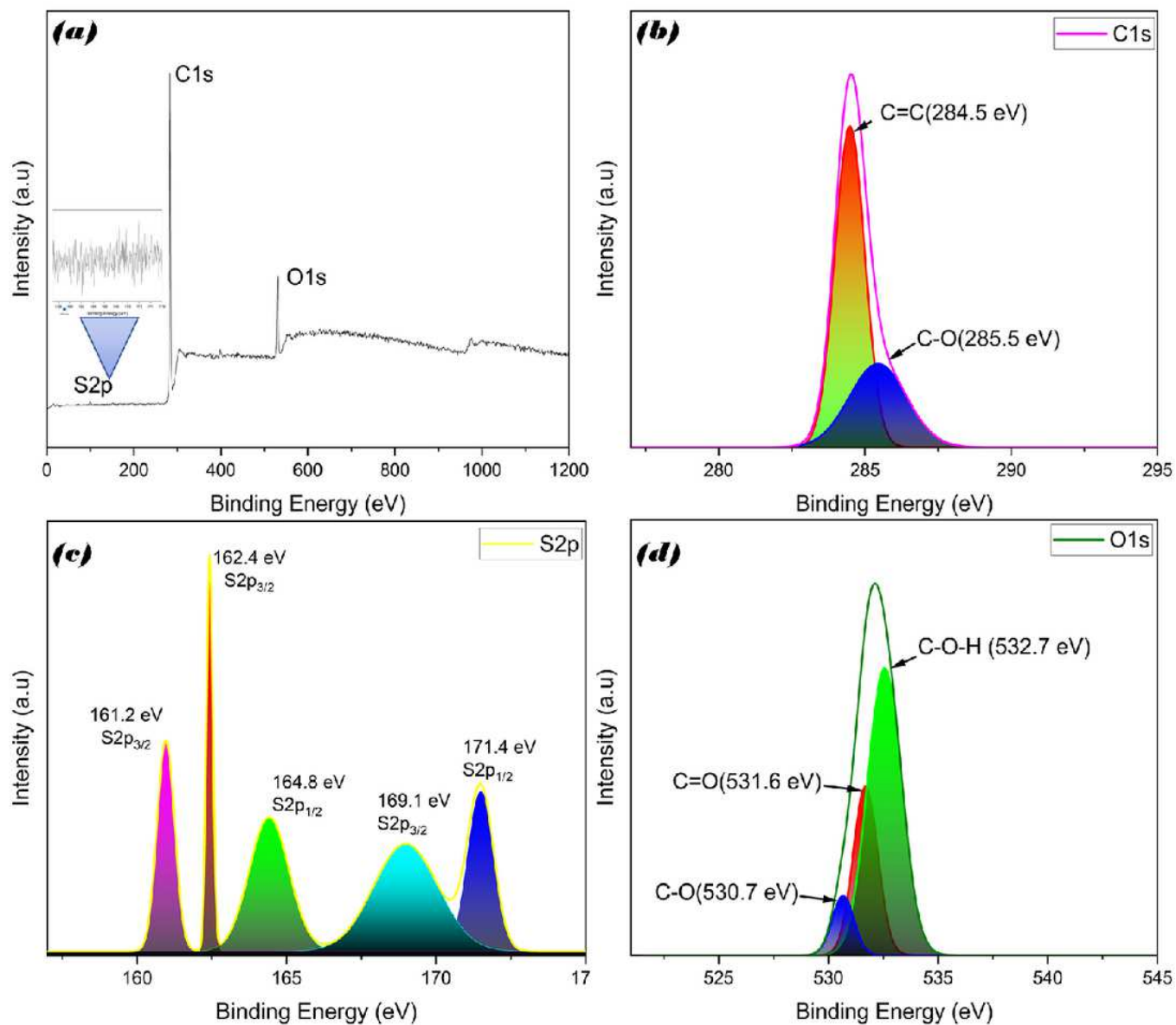


**Figure 3**

XPS of (a) S doped-CQDs (b) C1s (c) S2p (d) O1s

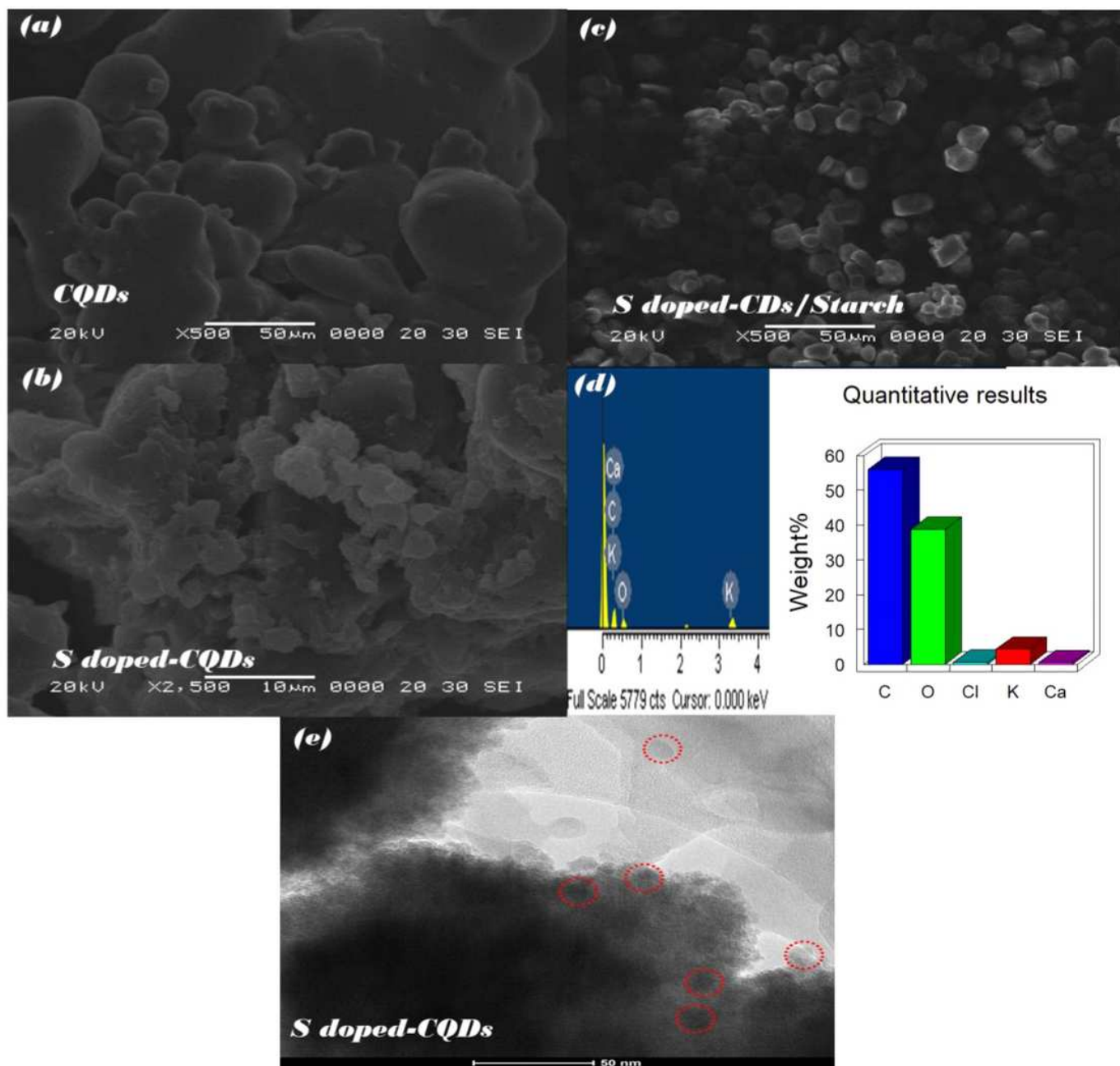


**Figure 4**

FIB-SEM of (a) CQDs (b) S doped-CQDs (c) S doped-CQDs/Starch and EDX of (d) S doped-CQDs, and HR-TEM of (e) S doped-CQDs

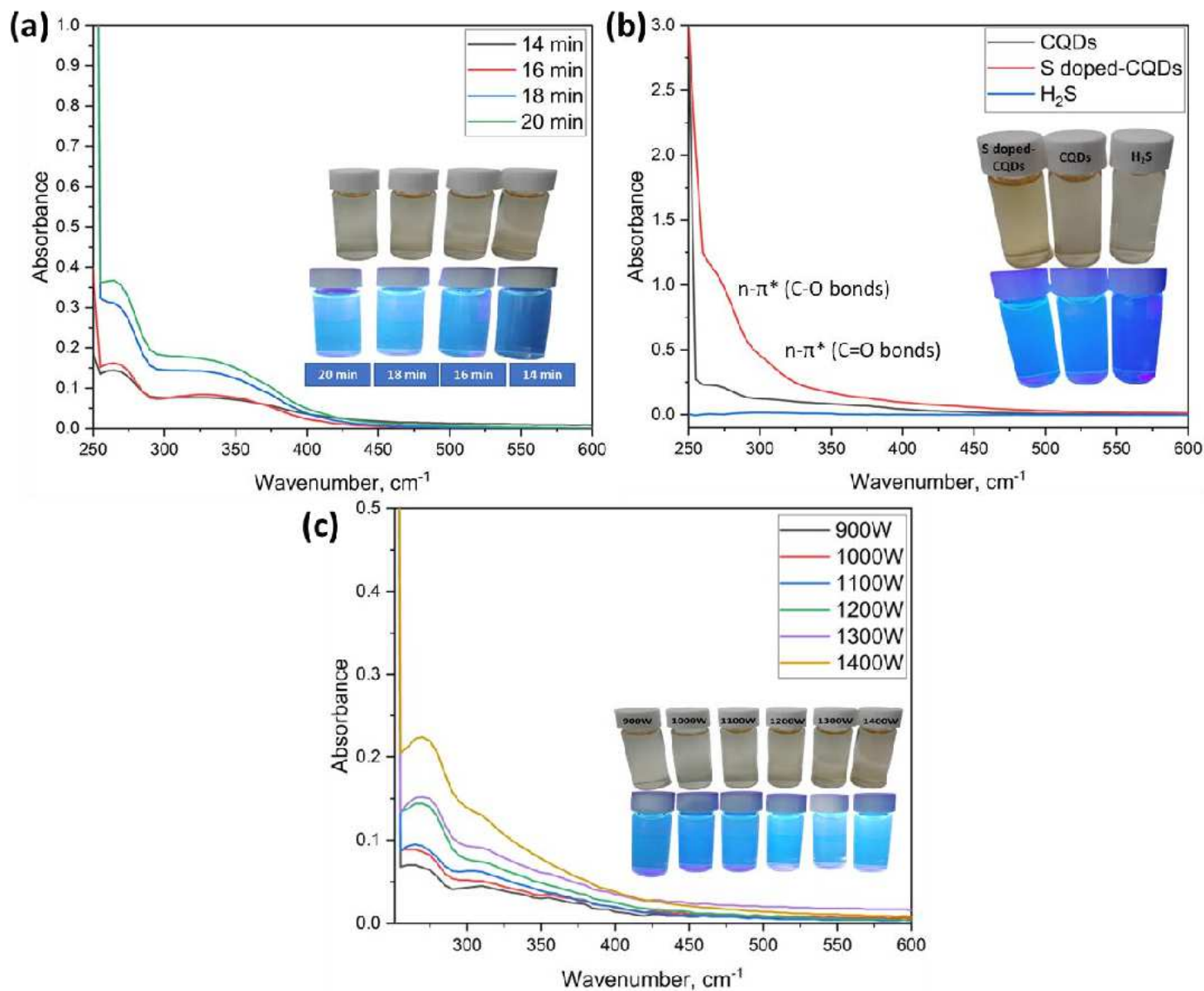


**Figure 5**

synthesis S doped-CQDs on (a) Comparisons synthesis in vary time (b) the effect of  $\text{H}_2\text{S}$  for enhance the absorbance under Uv-Vis (c) Comparison synthesis in vary watt

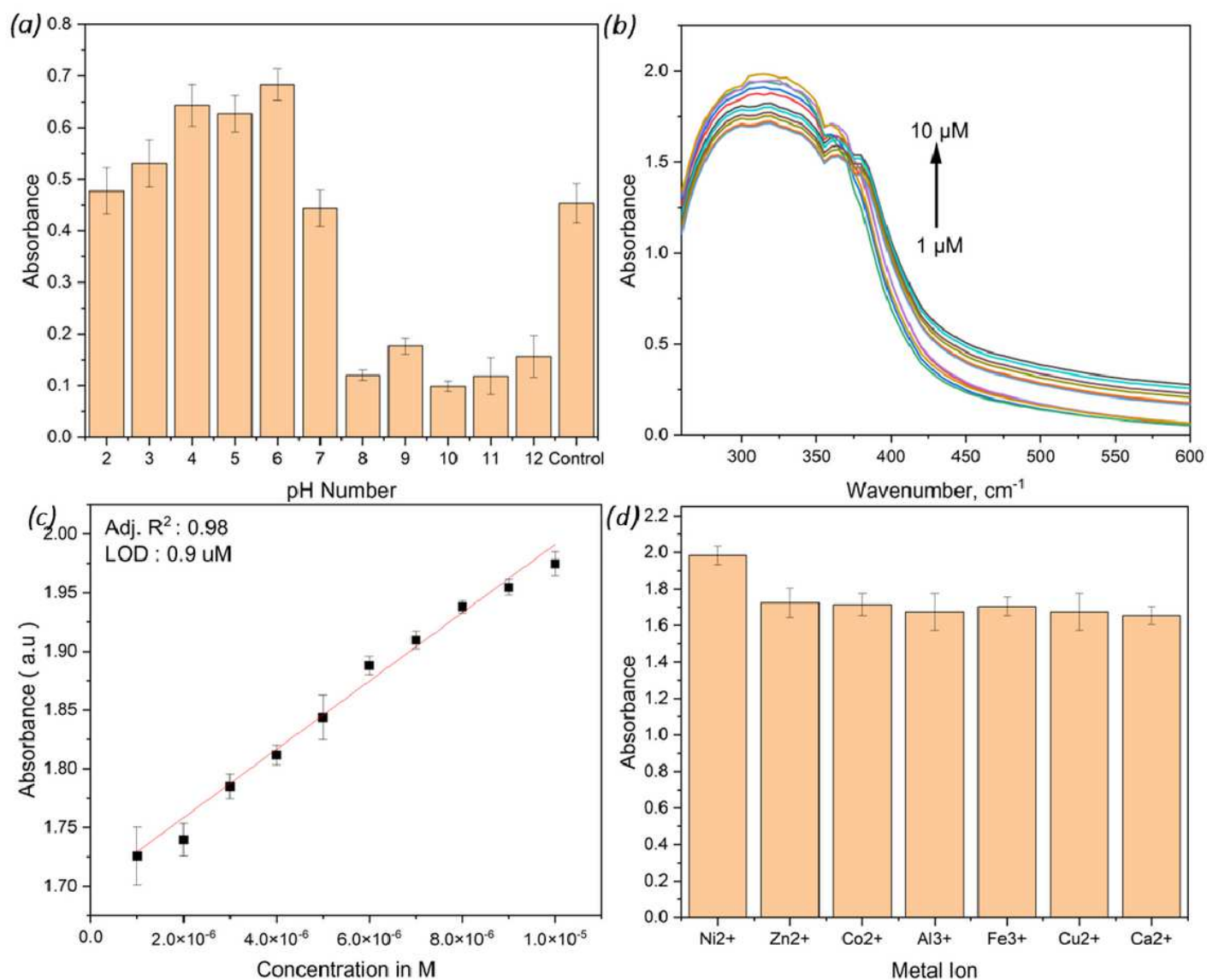


**Figure 6**

(a) The effect pH range 2-12 on S doped-CQDs (Error bars represent standard deviations of three independent measurements) (b) Detection Nickel ion under Uv-Vis Spectrophotometer. (c) Limit of detection Nickel. (d) absorbance in various metal ion.

## Supplementary Files

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