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Synthesis of quantum carbon dots (CQDs) from hard walnut skin by hydrothermal method

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ABSTRACT: In this study, biocompatible carbon quantum dots (CQDs) were prepared in a single step by hydrothermal method from walnut skin as a carbon source. Particle size, surface chemistry and crystal structure of carbon quantum dots were investigated using FTIR, DLS and XRD analyses and the optical properties of the material were investigated using absorption and fluorescence analyses. The results showed that the quantum dots of synthesized carbon had a good particle size distribution and the average particle size was 5.7 ± 2.5. Also, the effect of temperature on initial carbonization was investigated, which with increasing the temperature of the pyrolysis furnace, the percentage of carbon also increased, which was 46% by weight at 150 °C and 83% by weight at 450 ° C. Due to the fact that, the difference in weight of carbon produced between temperatures of 350 and 450 degrees is very small, and considering the increase of 100 degrees, the temperature of 350 degrees was considered as a suitable temperature for the initial carbonization of walnut shell. The results of the absorption spectrum and fluorescence show that at the 238 wavelength an absorption peak of the sample is observed and the sample shows good fluorescence scattering at the wavelength of 402 nm. According to the results of the studied FT-IR spectrum, the quantum surface of carbon dots is covered by hydrophilic functional groups such as hydroxyl, carbonyl and amine groups, and based on this, the synthesized nanoparticles are expected to show high dispersion in water.

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1- Introduction

Carbon quantum dots with other names such as carbon nanoparticles and graphene quantum dots are another group of carbon nanomaterials. In terms of the chemical structure and physical properties, carbon quantum dots are very similar to graphene oxide, and their differences are due to the size difference between them, which graphite carbon quantum dots are under 10 nm in size. For thousands of years, graphite and diamond were the only two known materials of pure carbon atoms. In recent decades, a series of carbon nanostructures have been discovered. Carbon nanomaterials with a size between 1 nanometer and 1 micrometer are comparable to proteins (1 to 100 nanometers) and nucleic acid (2 to 3 nanometers). The optimal size of carbon nanoparticles has made them ideal nanocarriers and nanocapsules for drug and gene transfer to a specific target in the body [1]. These materials have superior physical and chemical properties due to their smaller size compared to other carbon nanoparticles. These materials are graphene nanoparticles with different degrees of oxidation. The chemical composition of these carbon quantum dots includes carbon, hydrogen, oxygen and nitrogen, and the percentage of these elements varies depending on the synthesis method and the type of precursor. The crystalline network consisting of carbon atoms has clearly confirmed their graphitic nature in the transmission electron microscope images in the reports of carbon quantum dots. Carbon quantum dots have the ability to emit light in a wide range of visible, ultraviolet and near-infrared, for this reason, they are very similar to semiconductor quantum dots [2]. Research on carbon quantum dots has just begun. The discovery of carbon quantum dots, like many other discoveries, was accidental. These quasi-spherical carbon quantum dots were obtained for the first time by chance during the purification of single-walled carbon nanotubes through electrophoresis in 2004. These fluorescent nanoparticles, which were later called carbon quantum dots, were separated from the remaining carbon impurities during the synthesis of carbon nanotubes by the electric arc method. Researches in the field of these nanoparticles expanded by synthesizing these materials with different methods and many advances were made in the field of understanding the physical chemistry process of the origin of the inherent fluorescence of these materials. It has been found that the inherent fluorescence power of these materials increases with the increase of their surface [3]. Many precursors for the production of carbon quantum dots such as: graphite [4], fullerene [5], single and multi-walled carbon

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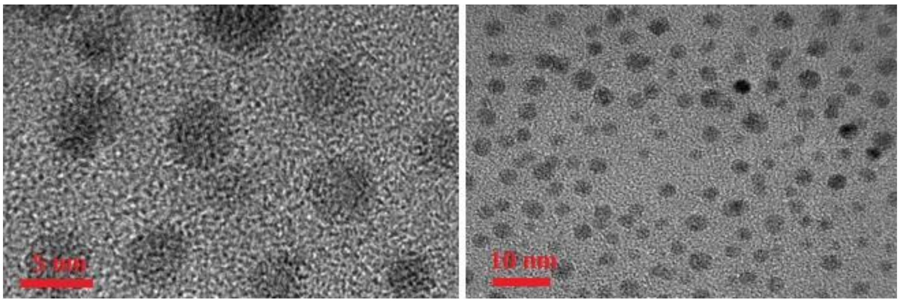

Fig. 1. Transmission electron microscope image of synthesized carbon quantum dots.

nanotubes [6], graphene [7], carbon fiber [8], candle soot [9], glycerol [10], glucose [11], aromatic organic compounds [12].

2- Methodology

2- 1- Materials

Walnut skin was collected from local gardens, phosphoric acid (H_2PO_4), sodium chloride (NaCl), sulfuric acid (H_2SO_4), nitric acid (HNO_3) and sodium hydroxide (NaOH) were purchased from Merck, Germany were used.

2- 2- Method

The skins of the walnuts were cut into small pieces to increase their effective surface. Then the skins were thoroughly washed with distilled water and heated in an oven at 70 °C for 24 hours until they were completely dry. After drying, the samples were removed from the oven, then 10 grams of walnut skin were mixed in 500 ml of an aqueous solution containing 1 M phosphoric acid and 0.8 M sodium chloride and left for 2 hours at room temperature, then the shells They were removed from the acid solution and heated in an oven at 70 degrees for 24 hours until they were completely dry. After drying, the sample was placed in the presence of air for initial oxidation in the pyrolysis furnace at different temperatures (degrees Celsius) with a temperature gradient of 5 degrees per minute, and after the temperature reached the desired temperature, nitrogen entered the furnace and at the same speed 5 degrees per minute, the temperature of the furnace reached 1000 degrees Celsius until the carbonization process began, and the sample remained at the same temperature for 25 minutes. took For this purpose, temperatures of 150, 350, 250 and 450 degrees were investigated. Then the furnace was allowed to cool down to room temperature. After carbonization, 1.8 grams of the carbonized sample was placed in 240 ml of nitric acid and sulfuric acid in a ratio of 3:1 in an oil bath for 12 hours at 100 degrees and 12 hours at 140 degrees. The color of the solution at this stage first changed to light brown and then to very dark brown. After this step, the pH of the solution was adjusted to 7 with 1M sodium hydroxide (NaOH). After the carbonization of walnut shells and acid treatments, carbon dots were formed. Finally, the obtained solution was filtered using a 0.22 micrometer filter. The filtered solution was centrifuged several times using a centrifuge at 30,000 rpm. It should be noted that each time the supernatant was used for the next step of centrifugation. To preserve the chemical structure of the synthesized substance, it was kept in a refrigerator.

3- Results and Discussion

3- 1- Results of the effect of temperature on primary carbonization

3- 2- XRD analysis results

3- 3- FTIR analysis results

3- 4- Size distribution analysis results

3- 5- Investigating the optical properties of synthesized carbon dot.

3- 6- Electron Microscopy (TEM) Results

The size and crystal structure of nanoparticles were accurately determined using the TEM method. Synthesized nanoparticles have a spherical structure and their size is below 10 nanometers (Figure 1).

4- Conclusion

It is important to choose the right raw material to achieve the desired properties. In this research, walnut skin was used as a precursor for the synthesis of quantum dots. These materials are easily separated after synthesis due to their insolubility in water and do not disturb the synthesized product. Also, due to its low toxicity compared to chemical precursors, its availability and cheapness, it is a suitable precursor for the production of carbon quantum dots. Increasing the temperature of the pyrolysis furnace has an effect on the production of carbon dots, and the temperature Optimum for this synthesis is 350 degrees Celsius. These synthesized dots have good stability and also the synthesized particles are hydrophilic and disperse well in water, there are hydroxyl, carbonyl and amine functional groups on its surface. The synthesized material has good optical properties and also the particle size distribution is very suitable and the dimensions of the synthesized quantum dots are less than 6 nm. According to these results and the analyzes carried out, the synthesis of quantum dots from walnut skin has been successful.

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