

Synthesis and characterization of Nickel Oxide with Nitrogen quantum Carbon dots as nanoadsorbent (NiO-NCQD) nanocomposite

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Abstract

This research deals with the manufacture of nickel oxide nanoparticles NiO, it has been characterized by Field emission scanning electron microscopy (FESEM), and Fourier Transform Infrared (FTIR), and their radii were range (10-20) nm and frequency (58.49%), where did not exceed the largest particles is (60 nm). Nitrogen carbon quantum dots (NCQD) were prepared based on a carbon-rich source and a nitrogen-rich source with a heat source. Nitrogen carbon quantum dots (NCQD) were prepared from L-glutamic acid as a carbon source and ethylene diamine (EDA) as a nitrogen-rich source depending on the source of heat: (reflex, autoclave, UV-digestion, oven, and microwave). The (NCQD) synthesis was characterized using spectral scanning (UV-VIS), then, the effect of its quenching ratio was studied using a sulfuric acid solution, it was found that the best thermal method is the autoclave then a UV-digester then oven and microwave. Nickel oxide-nitrogen quantum carbon dots (NiO-NCQD) are prepared from a mix between NiO nanoparticles with nitrogen carbon quantum dots as nano-adsorbent. (NiO-NCQD) has been characterized by Fourier Transform Infrared (FTIR) and energy-dispersive X-ray (EDX).

Keywords: Ethylenediamine; L-Glutamic Acid; Nano-adsorbent; Nickel Oxide Nanoparticles; Nitrogen Carbon Quantum Dots (NCQD); Thermal Methods.

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INTRODUCTION

Quantum carbon dots were discovered by chance for the first time in 2004 [1], Symbolize it by (CQDs, C-dots or CDs) [2]. General Tiny spherical carbon nanoparticles [3] Quantum carbon dots size less than 10nm [4-5]. It has several advantages, including: Classified from semiconducting [6], zero-dimensional OD [7] type of hybridization in it sp^2 [2], It is characterized as well dispersed in water or an organic solvent [3], carbon quantum dots can be classified into (CQDs), graphene quantum dots (GQDs), and polymeric dots (PDs).

Graphene quantum dots are qubits [8], two-dimensional (2D), graphene blocks of cross-

sectional size less than (100nm) [9-10], while Polymer Dots (PDs) consists of linear assembled polymers or polymer chains around or within a carbon core [11] whose ideal particle size is less than 100 nm [12], it has photoelectric properties such as high chemical stability, large conductivity, and optical absorption [13], as in Fig. (1) [9, 11, 14-16].

There is a relationship between the size of quantum carbon dots and the energy gap, as shown in the Fig. (2) [6].

Despite the importance of quantum dots in improving synthesis methods, but they suffered from a weak quantum yield. The quantum yield was improved and doubled by introducing a group of nitrogen or sulfur [17] or phosphorous [18].

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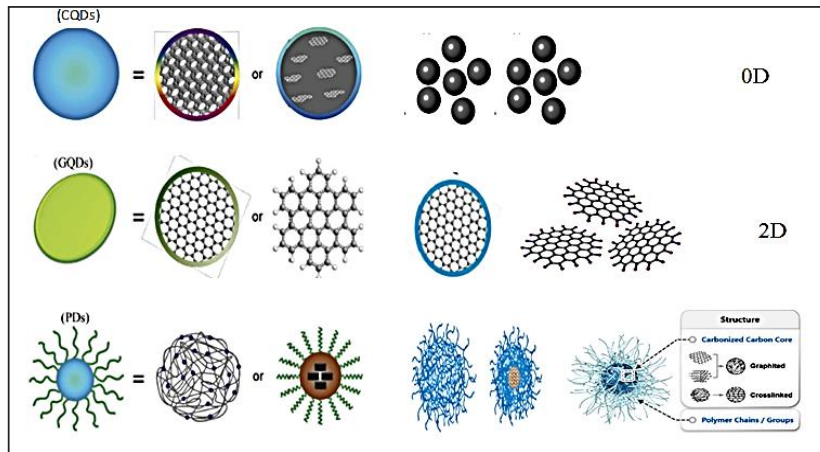


Fig. 1. Types of carbon dots (CDs).

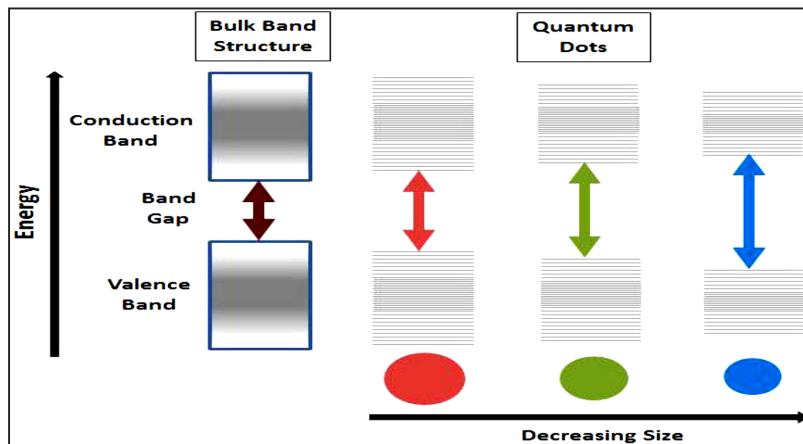


Fig. 2. Band gap energy levels due to quantum dots due to their size.

The most used functional groups in activating the surface of quantum dots with primary amines [18], Nitrogen carbon quantum dots are symbolize to as (NCQD), which are particles with a size of about 18nm. Electronic density and tuning of optical and electrical properties, they have high chemical stability, and increase the lifetime of the excited electrons [19].

Quantum carbon dots and nitrogen carbon quantum dots are used in such as: carbon quantum dots are used in: Enhancing the photocatalytic activity of TiO_2 nanoparticles [20], in biomedical applications[21], for selective irons detection and cellular imaging [22], and photoluminescence sensor for mercury (II) and lead (II) ions [23], in the manufacture of solar cells [24-25], the environmental and analytical fields such as determining bisphenol [26], mercury [27], copper, lead [28], iron [4], others metal ions. It

used in improve qualities lithium batteries [29], performance capacitors [30], filters for water purification [31], it used in reduced oxygen from carbon tubes [32], photo catalysis [33], the biological field [34], and in the pharmacological [35]. Colloidal quantitative drips bound to amino groups or boronic acid can influence the entry of herpes simplex virus type 1. They can also be used against one of the most common concerns and types of human infection. Today's pathogenic (Coronavirus HCoV-229E). Mechanistically, the entry of human MERS-CoV may be due to the inhibition induced by the interaction of boronic acid functions in colloidal quantum blobs with a protein through pseudo-lectin-based interactions [36]. Nickel oxide is used in many applications such as gas sensors H_2S and NH_3 [37], solar cells [38-39]. Nickel oxide and its derivatives are described by (X-Ray) and (FTIR), scanning electron microscopy

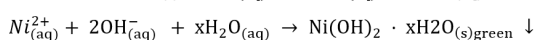
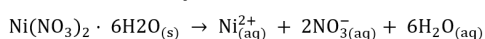
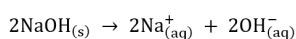
(SEM), and X-ray diffraction (XRD) [40-43].

This research is one of the important researches in improving the properties of nanoscale nickel oxide as a semiconductor by combining it from nitrogen carbon quantum dots as a nano-adsorbent, in order to use it in the manufacture of selective electrode that depend on modified carbon paste in order to determine an organic pollutant using electrochemical methods such as cyclic voltammetry. High purity materials were used in this research: L-glutamic acid 99% $C_5H_9NO_4$, sulfur acid 98%, ethylene diamine $C_2H_8N_2$ and symbolized by (EDA) (LOBA CHEMIE PVT.LTD), its purity is 98% and its, a nickel nitrate $Ni(NO_3)_2 \cdot 6H_2O_{(s)}$, sodium hydroxide, spectrophotometric, cells made of quartz and glass, autoclave made SYstec, thermal oven, UV-digester Produced by Metrohm Model 705, microwave, reflux.

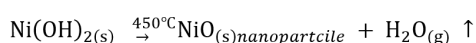
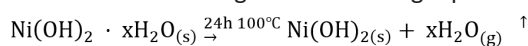
EXPERIMENTAL SECTION

Synthesis of nickel oxide Nanoparticles

A solution of (0.5M) nickel nitrate was prepared in a volume of 100ml and a solution of sodium hydroxide in a concentration of (1 M) in a volume of 100ml was prepared and put in burette Then, very slowly, sodium hydroxide is added, with constant stirring, at a temperature of (35 °C), and the titration is stopped when to get a pH between pH (7-8), then we get a green precipitate. The end point of the titration is known depending on the color change of the pH paper. The reaction mechanism can be extracted according to the following equations:



Then filtered by a Buchner funnel, then the product nickel hydroxide is dried for (24 h) at temperature (100°C) and then kept in (Dissector) and then completely transferred to an incinerator at a temperature of (450 °C) for (4 h), it turns black powder, the reaction mechanism can be summarized according to the following equations:



Then it is crushed vigorously in an agate mortar, and then kept until analysis by a scanner.

Synthesis of nitrogen carbon quantum dots (NCQD)

Synthesized based on the presence of a carbon source and a nitrogen source, then they were compared. Take (2 g) of L-glutamic acid $C_5H_9NO_4$ as a carbon source, then add 2ml of ethylene diamine (EDA) as a nitrogen source, then add (40 ml) of water through a distillation medium and mix with a magnetic stirrer. (15 min), then this solution using several methods a temperature, it is: autoclave (113 °C) and a pressure of (172.5 kPa) for six hours- an oven for 2.5 hours until dry, then add (40 ml) of double-distilled water, a UV-digester for an hour and two hours, a microwave in terms of time from (1 to 6) min, a reflux for six hours. The resulting samples were filtered using micro-filters (0.45 μ m), to ascertain the formation of nitrogen quantum carbon dots; the prepared samples were scanned based on their spectral survey.

RESULTS AND DISCUSSION

The results were as follows:

Synthesis of nickel oxide nanoparticles

Nickel oxide nanoparticles was analyzed using the (FESEM) scanner, and then the structure and the granular size of the NiO nanoparticles were traced using (FESEM) with an increasing magnification starting from (1 μ m) Fig. (3). The image was processed for dimensional measurement of the diameter of the nickel oxide nanoparticles using the (ImageJ) software package using the straight-line tool and the analysis/measurement function. Length measurements were calibrated using the scale bars in the image, and the analytical/determinant scale function in the image. For this analysis, only diameter measurements were performed, since they possess relatively fixed diameters. program measurements can be used and analyzed using Excel, showing that their radii were range (10-20) nm and frequency (58.49%), where did not exceed the largest particles is (60 nm), as Fig. (4).

Nickel oxide nanoparticles was analyzed using (FTIR), as Fig. (5).

From Fig (5), it shows the absorption bonds at (420 cm^{-1}), evidence of a vibration of the Ni-O bond, and it expanded to reach (522 cm^{-1}). that the closer the vibration is to the blue ray, an indication of the small size of the particles, which indicates the resulting oxide with nanoparticles dimensions and this is confirmed by (FESEM).



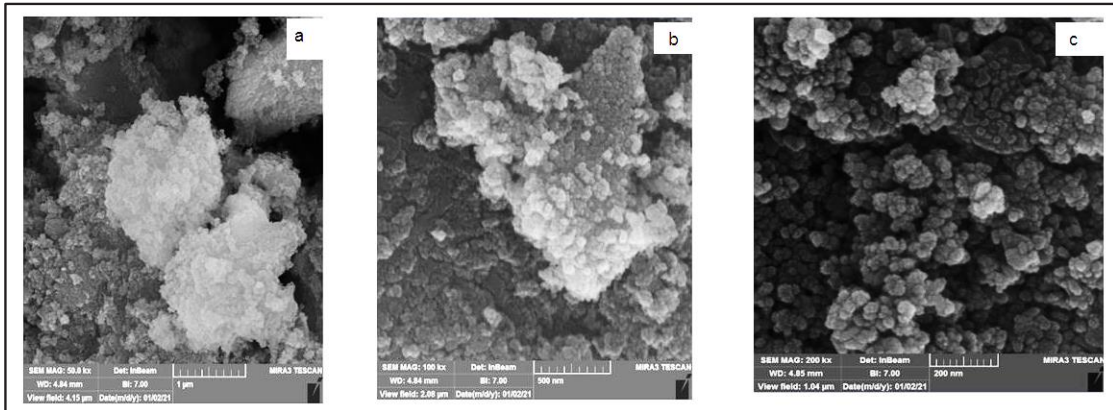


Fig. 3. FESEM for NiO nanoparticles a)1 μm, b)500 nm, c) 200 nm.

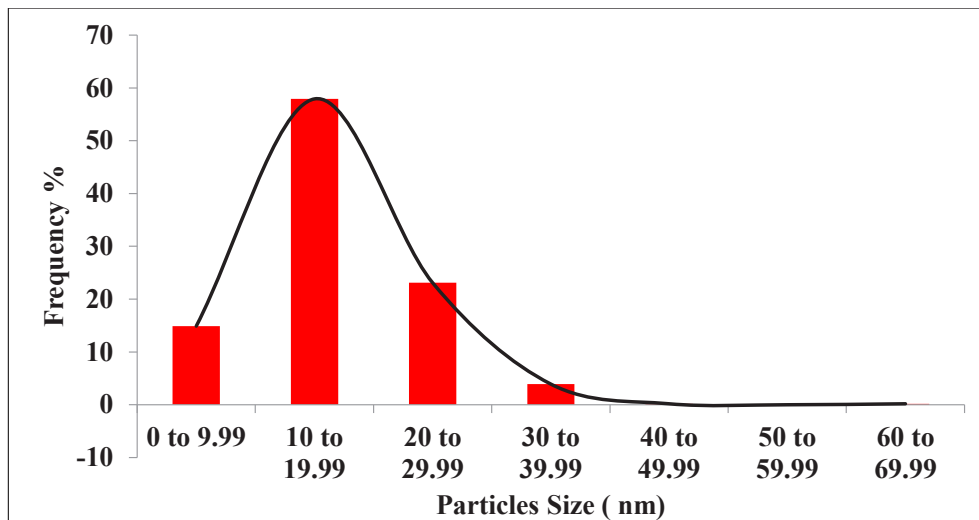


Fig. 4. Frequency percentage for NiO nanoparticles.

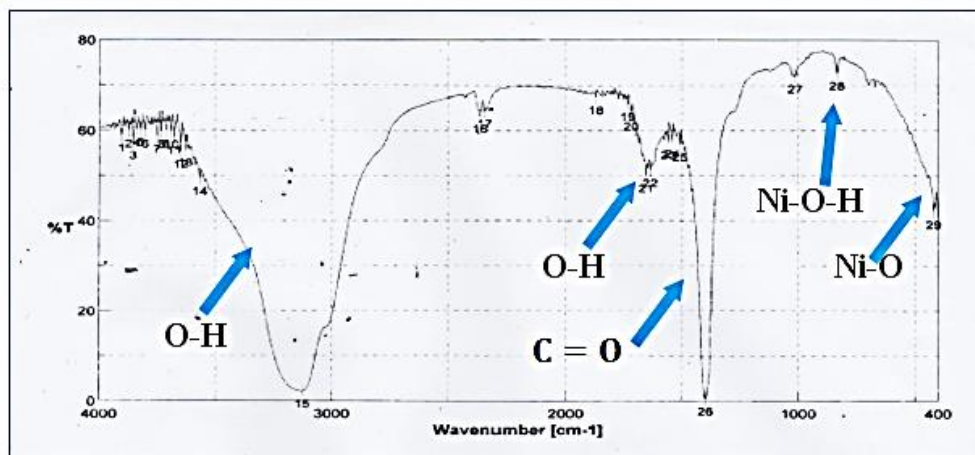


Fig. 5. FTIR for NiO nanoparticles.

The adsorption band is set at (834 cm^{-1}) for a Ni-OH stretching bond. The peak around ($3.127\text{-}3400\text{ cm}^{-1}$) on (FTIR) is due to the presence of the O-H bond. The absorbance at (1637 cm^{-1}) is attributed to the hydroxyl groups [44] Due to the influence of quantum size and nanoparticles spherical structures, the (FTIR) adsorption of Ni-O Nanoparticles is blue offset compared to the larger granular size. Besides the Ni-O vibration, that the broad absorption band centered near (3440 cm^{-1}) is attributed to the stretching vibrations in the O-H band and the weak band near (1635 cm^{-1}) is due to the H-O-H bending vibrations pattern due to water absorption in air when preparing (FTIR) sample discs in open air despite the sample drying in an oven. Also, it shows the effect of water in the structure. Meanwhile, it indicates the presence of hydroxyl in the starting material, and the broad absorption is around (767 cm^{-1}) back vibration and stretching C=O. While the serrated absorption ranges in the region from ($1000\text{-}1500\text{ cm}^{-1}$) is due to the symmetric and asymmetric vibration and stretching of C=O and C-O, indicating that the ultrafine forces tend to the strong physical absorption of H_2O and CO_2 [45]. The previous diagram shows the confirmed formation of NiO nanoparticles.

Synthesis of nitrogen carbon quantum dots (NCQDs)

Nitrogen carbon quantum dots were prepared based on a L-glutamic acid $\text{C}_5\text{H}_9\text{NO}_4$ as a carbon source and ethylene diamine (EDA) a nitrogen-rich source, and then subjected to several thermal methods, all samples were analysis by (UV-VIS). It was found that (pH = 9.18). The quenching ratio was studied based on adding a solution of (0.1M) sulfur acid with different volume.

It is noted from Fig. (6a, 6b), that the thermal effect by reflex distillation for six hours, in addition to without heat, that no peaks were obtained and thus the formation of (NCQD), and if it is likely not to be deleted The H_2O molecule between the carbon source and the nitrogen source, where it was observed that there was no change in color for the product studied in this way, Therefore, nitrogen carbon quantum dots cannot be prepared without heat and cannot be prepared using reflux. The effect of autoclaving on the preparation (NCQD) was also studied, as in the Fig. (6c), that the effect of the thermal autoclave at a temperature of ($113\text{ }^\circ\text{C}$) and for a

period of six hours, the appearance of clear peaks at wavelength of (280 nm), where the quantum carbon dots show a clear and strong absorption of ultraviolet. Therefore, this thermal method can be used to prepare nitrogen carbon quantum dots (NCQD). The effect of microwaves on (NCQD) was studied, as in the Fig. (6d, 6e), it is a study of the effect of microwaves on the sample in terms of time, noting the gradual change of color from transparent to yellow to black, according to the time taken in the effect. It is find that the best time is at 5min to form (NCQD) for two reasons, the first of which is absorption is maximum ultraviolet at a wavelength of ($\approx 300\text{ nm}$), as the nitrogen carbon quantum dots, and the second is the yellow color of the sample solution. Therefore, a microwave can be used in five minutes to prepare (NCQD). The effect of UV-digestion on the preparation (NCQD) was studied, as in the Fig. (6f, 6g), the thermal effect through digestion based on ultraviolet as showed in Fig. (6f, 6g) according to the effect of time for (1-2) h, and it was found that the best time is one hour for the appearance of maximum absorption within the ultraviolet field ($\approx 323\text{ nm}$). Therefore, one-hour UV-digestion can be used to prepare nitrogen carbon quantum dots (NCQD). The effect of oven on the preparation (NCQD) was studied, as in the Fig. (6h), for a period of two and a half hours until the sample was completely dry, then 40ml of double-distilled water was added and filtered by 0.45μ filter, where the maximum absorption peak appeared at ($\approx 300\text{ nm}$). Regarding the $\pi\text{-}\pi^*$ transition of the C=C bond in the short wavelength region, both (CQDs) and (GQDs) have photon-harvesting potential.

In general, quantum dots usually show strong adsorption in the (UV) with range ($230\text{-}320\text{ nm}$), and can extend into the visible region, some adsorption shoulders may appear in the range ($270\text{-}390\text{ nm}$) This phenomenon is related to the $\pi\text{-}\pi^*$ transition of the C=C bond or $n\text{-}\pi^*$ bond returns C=O. The quenching ratio of the nitrogen carbon quantum dots prepared by the previous methods was studied from the effect of adding different volumes of (0.1M) sulfur acid as shown in the Fig. (9). To show the difference between them, the area was calculated manually, which is the first time it is used, in terms of wavelength and absorbance, and the graphs were as in Figs. (7-8).

To determine the best thermal method for preparing nitrogen carbon quantum dots, it can be summarized as in the Table (1).



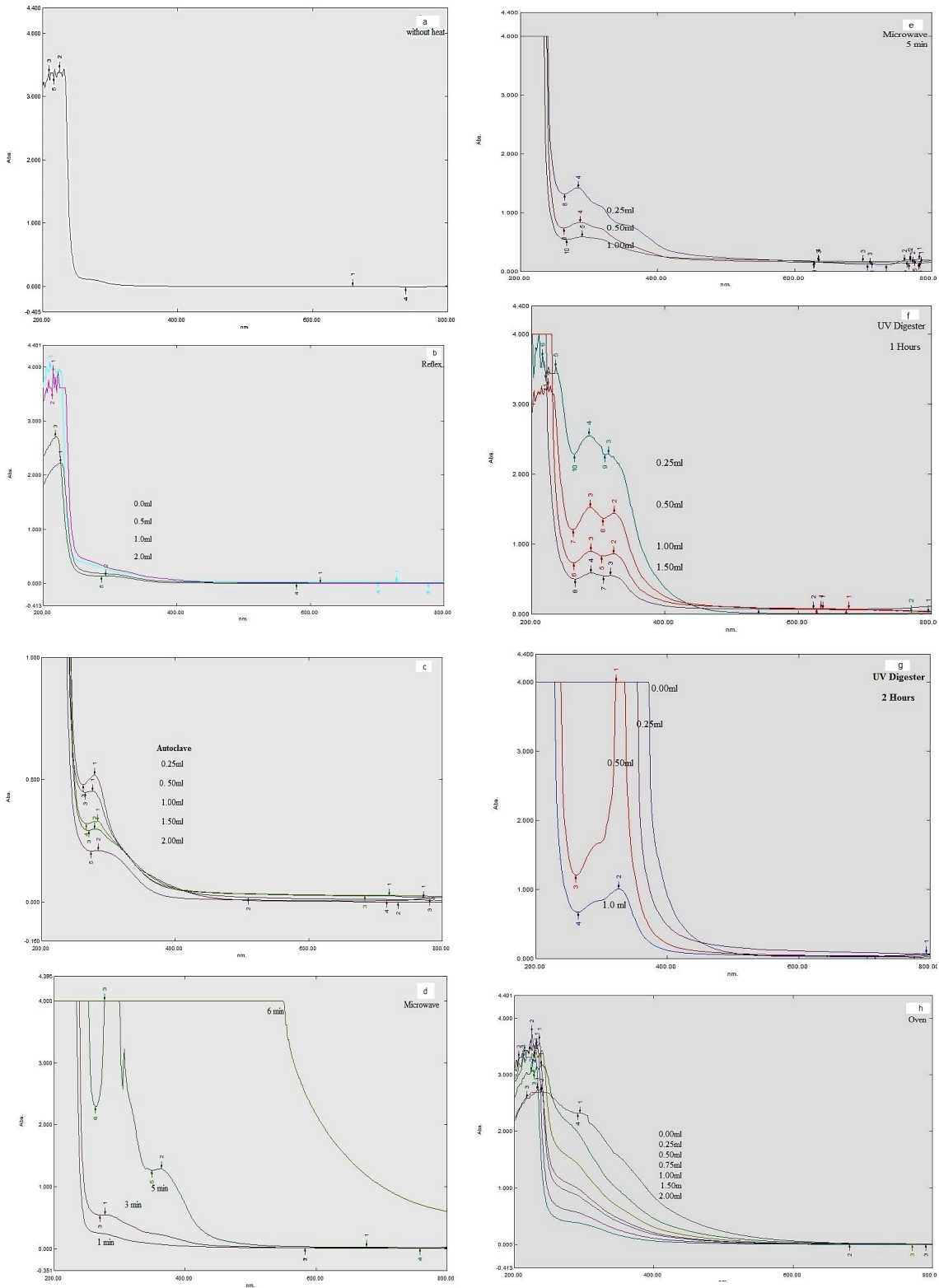


Fig. 6. UV-Vis for NCQD based on a) no heat b) reflux c) autoclave d) microwave in terms of time e) Microwave at 5min f) UV digestion for 1h g) UV digestion for 2h h) oven.

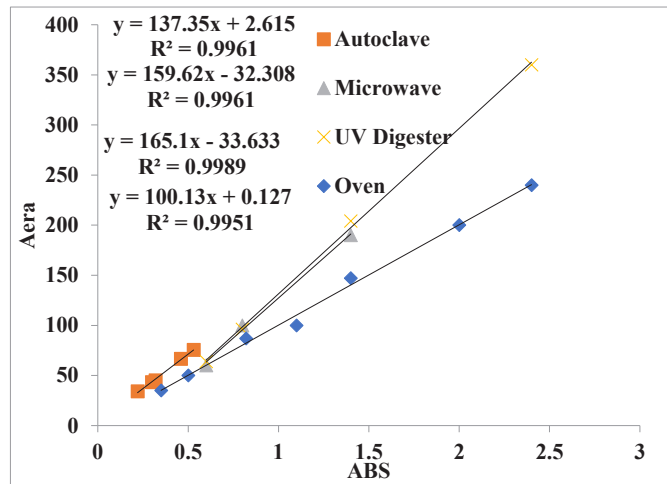


Fig. 7. Curves for the in terms of maximum absorbance vs area for JNCQD), prepared by thermal methods (Autoclave, UV-Digester at 1h, Oven, Microwave at 5min).

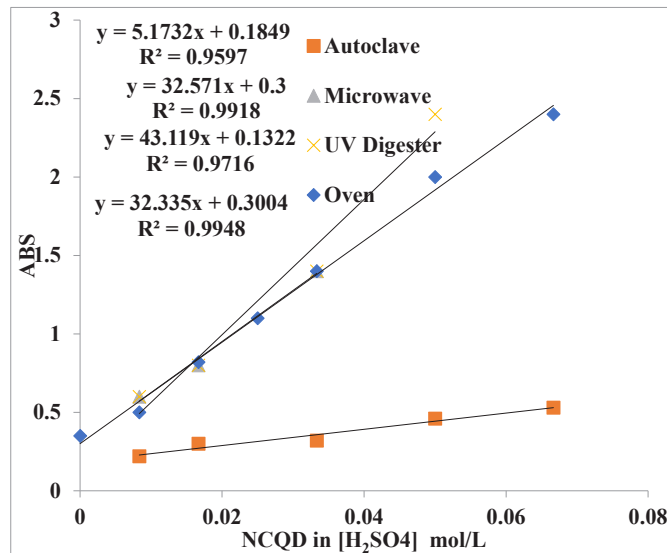


Fig. 8. Curves for the effect of adding sulfuric acid on JNCQD) prepared by thermal methods (Autoclave, UV-Digester at 1h, Oven, Microwave at 5min).

Table 1. Comparison of thermal methods used to prepare nitrogen carbon quantum dots from L-glutamic acid with (EDA) using spectrophotometer.

Thermal method	Conditions	Wavelength (λ_{max})	Correlation coefficient R^2 *	Correlation coefficient R^{2**}
Without heat	-	no peak	-	-
reflex	180°C , 6 h	no peak	-	-
Autoclave	172.5kPa, 6 h, 113°C	280nm	0.9961	0.9597
UV-digestion	1h	≈ 323nm	0.9989	0.9716
microwave	600W for 5min	≈ 300nm	0.9910	0.9918
oven	180 C - 2.5 h	≈ 300nm	0.9951	0.9948

*Area in terms of absorbance for JNCQD) ** effect of adding sulfuric acid on JNCQD)



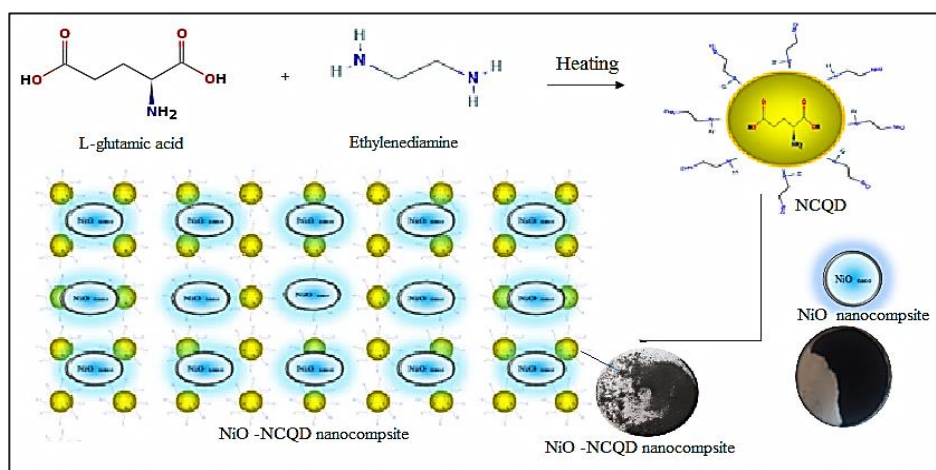


Fig. 9. Scheme of the NiO-NCQD nanocomposite.

The thermal method was chosen by autoclave as a method for manufacturing to (NCQD) for several reasons. The first reason: it has a shorter maximum wavelength where (NCQD) tend to absorb invisible rays at short wavelengths. The second reason: the thermal method by autoclave maintains the sample size. The third reason: A correlation coefficient greater than (0.9) The remaining thermal methods can be used, and they can also be arranged as follows. Autoclave, UV-digestion for (1 h), oven and microwave at (5 min).

Synthesis and characterization for (NiO-NCQD) nanocomposite

The method of preparation in this stage is based on the previous stages in the first stage in the first stage NiO nano (black powder) and the second stage Nitrogen quantum carbon dot (NCQD) (yellow solution) as nano-adsorbent, where (0.6 g) of NiO nanoparticles are added with (30 ml) of (NCQD) after filtering it with a micro-filter (syringe with filter) and subjected to ultrasonic waves for (1.5 h), then washed three times with distilled water and dried in an oven at (60 °C) for 12h, we get (NiO-NCQD) nanocomposite as in Fig. (9).

It is noticed from the sensory observation that there is a difference between the NiO nanocomposites where its color is black, while (NiO-NCQD) is greenish-black, (NiO-NCQD) is analysis by (FTIR) infrared, as shown in Figs. (10-11).

From the previous two Figs., it was shown the emergence of new spectral bands (peaks) indicating the presence of mono- and di-amine groups, the appearance of peaks for mono-

amino groups due to the dehydration of the water molecule between ethylene diamine and L-glutamic acid, where the presence of peaks at (420 cm^{-1}) indicates the presence of Vibration of the Ni-O bond was stretched up to (522 cm^{-1}), overlap wide peaks at (3344 cm^{-1}) and ($\approx 2998\text{ cm}^{-1}$), indicative of the stretching vibrations of OH and NH respectively. The peak at (1730 cm^{-1}) is the characteristic of the vibrational expansion of carbonyl C=O, the peak at (1409 cm^{-1}) is indicative of the bending vibrations NH, and the peak at (1234 cm^{-1}) is the result of Expansion vibrations of both C-N and CO, the FTIR results indicate surface operation of colloidal quantum dots with nitrogen, which mainly confirms the surface loading of NiO nanocomposite with (NCQDs). The EDX spectrum of the (NiO-NCQD) nanocomposite was studied as in Fig. (12).

It can be concluded from the (EDX) spectrum of (NiO-NCQD) nanocomposite that an energy band appeared at ($\approx 7.705\text{ Kev}$), which is a characteristic peak of nickel and excited at the K layer, the weight percentage of nickel was (79.09%), and the atomic percentage was (50.58%) and a band appeared at (0.525 Kev), which is a characteristic peak of oxygen, and the weight percentage of oxygen is (19.89%), and the atomic percentage of oxygen is (46.67%), and the percentage of impurities is (1.02%) of NCQD. Two electrodes were made of modified carbon paste, the first electrode was modified with nanoscale nickel oxide (12%) with graphite and paraffin oil to obtain a pasty texture, and the second electrode was modified with nanoscale nickel oxide (12%) with nitrogen carbon quantum dots (1.02%), graphite and paraffin oil

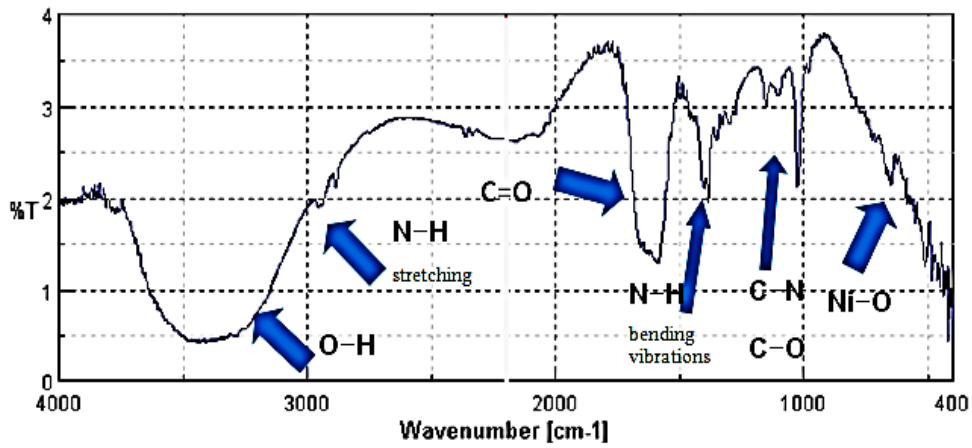


Fig. 10. FT-IR of (NiO-NCQD) nanocomposite.

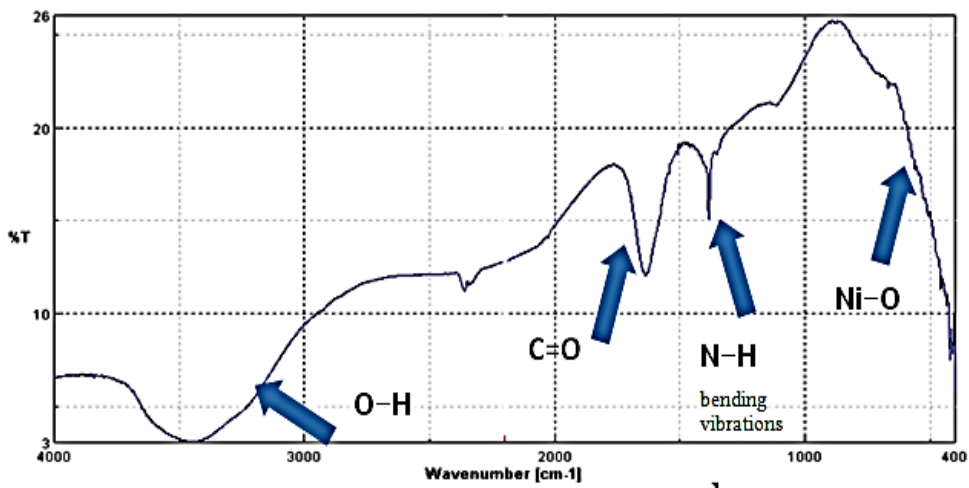


Fig. 11. FT-IR of (NiO-NCQD) nanocomposite after washing three times by distilled water.

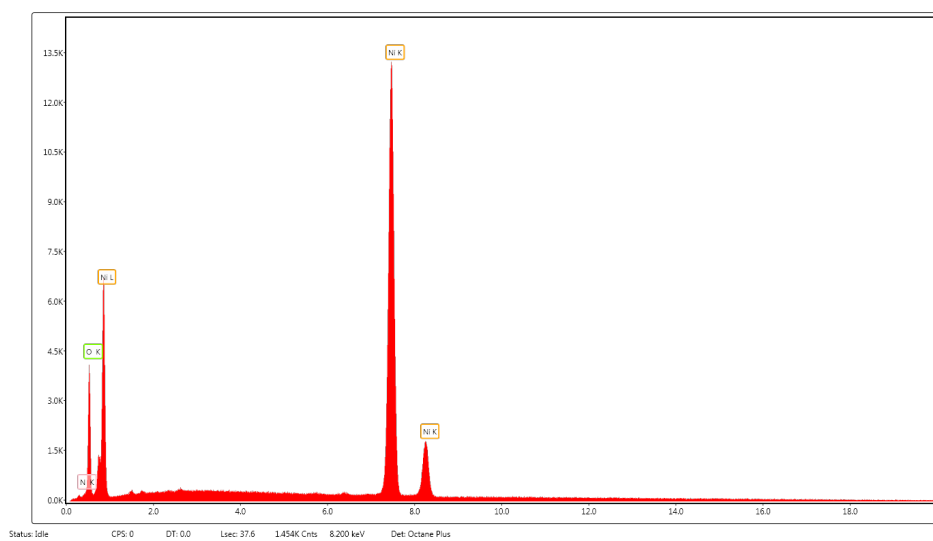


Fig. 12. EDX for (NiO-NCQD) nanocomposite.

to obtain a pasty texture. The resulting current on the surface of the second electrode is greater than that of the first electrode, which indicates an improvement in the properties of nickel oxide. Currently it is used in the manufacture of selective electrodes by electrochemical methods

CONCLUSIONS

Nickel oxide nanoparticles were synthesized; its radii range from (10-20) nm. Nitrogen quantum carbon dots were prepared from L-glutamic acid with (EDA) under the conditions of a heat source, the best method for preparing carbon quantum dots is by autoclaving. (NiO-NCQD) nanocomposite is prepared from mix between NiO nanoparticles with nitrogen carbon quantum dots as nano-adsorbent (NiO-NCQD). (NiO-NCQD) it has been characterized by Fourier Transform Infrared (FTIR) and energy-dispersive X-ray (EDX).

CONFLICTS OF INTEREST

The authors do not have any conflicts of interest.

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