

تحضير وتوصيف الزنك النانوي من عشبة القرنفل

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المقدمة:

تم تحضير و تخليق جسيمات أكسيد الزنك النانوية النباتية (ZnO NPs) و عشبة القرنفل بإضافة وزن 3 جرام من المادة العشبية (عود القرنفل) إلى محلول أكسيد الزنك (4.5 جرام من أكسيد الزنك في 200 مل من الماء الخالي من الأيونات) وتم تحريك المزيج على المحرك المغناطيسي (magnetic stirrer) لمدة 8 ساعات وبعدها، تم تفحيم المستخلص ومن ثم معادلة الحامضية وكانت ال قيمة الحامضية ال pH (6.97) وتم اختيار افضل وزن من الفحم و هو 0.05 وكانت قيمة ال 94% و كذلك أخذت القياسات الطيفية و التطبيقات و منها الاشعة ال UV و كان الطول الموجي لأوكسيد الزنك عند 372 نانومتر و عند امتصاصية 0.083 و كذلك تم أخذ طيف الاشعة تحت الحمراء و بينت فيها القمم و كانت عند ال قمة و الحزمة 1600 Cm^{-1} و تم قياس نسب العناصر المحضرة من أكسيد الزنك و عشبة القرنفل و كانت أعلى نسبة من Zn هي 53.87 L و الأوكسجين هي 22 K و الكربون هي 24.13 K و من خلال هذه ال اوزان تبين ان الافضل للكربون و نسبة هي 0.86 وبعدها تم تنقية المادة بأستعمال الفحم المنشط و كان أفضل زمن هو 30 min و كان أفضل تركيز هو 3×10^{-3} عند الامتصاصية 0.083 و كانت النسبة المئوية هي 95.5%

مشكلة البحث:

استعمال مواد كيميائية خطيرة قد تختلف بسمية بيئية و بشرية و الغرض هو تطوير و تنقية المواد المعقدة و الخطرة و الاوراق من المخلفات الى مواد صالحة للاستعمال.

أسئلة البحث:

هل يمكن مستخلص القرنفل ان يعمل كعامل مختزل لتكوين جسيمات النانوية؟ ما الظروف المثلى للتحضير؟ ما الروابط الكيميائية المسؤولة للكشف بوساطة التقنيات ، XRD ، UV-، (FTIR، VIS)، SEM، TEM؟

أهداف البحث:

استخلاص المركبات الفعالة من عشبة القرنفل و استعمالها في اختزال ايونات الزنك بطريقة صديقة للبيئة و تحديد الظروف المثلى من (Ph ، ودرجة الحرارة ، و زمن التفاعل ، و التركيز)

أهمية البحث:

تقديم بديل أخضر و آمن لتحضير الجسيمات النانوية و تقليل تكلفة و رفع كفاءة الانتاج و إنتاج الجسيمات النانوية و توفير بيانات علمية جديدة و تفتح آفاقاً علمية متطورة و دعم التنمية المستدامة

منهج البحث:

جمع و تحضير المادة النباتية : اخذ عشبة القرنفل و تجفيفها و طحنها للحصول على مسحوق ناعم و استخلاص المركبات الفعالة باستعمال الماء الساخن والايثانول و ترشيح المستخلص في عملية الاختزال

تحضير: تحضير المحلول من احد املاح الزنك و إضافة المستخلص النباتي وملاحظة تغير اللون يدل على تكون جسيمات نانوية و استعمال عدة مطيافيات حديثة و من ضمنها: uv-

TGA ، DLS ، SEM ، TEM ، XRD ، FTIR ، vis

و اختبار البايولوجية و مضاد للبكتريا و الفطريات للجسيمات النانوية
الكلمات المفتاحية: جسيمات الزنك النانوي ، التحضير الأخضر ، عشبة القرنفل ، التوصيف الطيفي ، الخصائص الفيزيائية و الكيميائية

Preparation and characterization of nano-zinc from clove gras

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Abstract:

Plant-based zinc oxide nanoparticles (ZnO NPs) were synthesized using clove stick (3 g) added to a zinc oxide solution (4.5 g in 200 mL ionized water) and stirred for 8 hours. The resulting extract was carbonized, and the pH value was adjusted to 6.97. The optimal charcoal weight was determined as 0.05 g, achieving 94% efficiency. Spectroscopic analyses were conducted, including UV–Vis absorption at 372 nm (absorbance 0.083) and FTIR, showing peaks at 1600 cm^{-1} . Elemental analysis revealed Zn at 53.87%, O at 22%, and C at 24.13%, with an optimal carbon ratio of 0.86. Activated charcoal purification was performed for 30 minutes at a concentration of $3 \times 10^{-3}\text{ M}$, achieving 95.5% adsorption efficiency. The activated ZnO NPs demonstrated antibacterial activity. Kinetic analysis based on the Lagergren model showed first-order constants $k_1 = 0.0772$ and $k_{-1} = 0.0093$ with $R^2 = 0.9863$. The pseudo-second-order model provided excellent correlation ($R^2 = 1$), with experimental and theoretical values closely matching (58.78 vs. 58.47).

Keywords: Zinc nanoparticles, Green synthesis, Clove extract, *Syzygium aromaticum*, Nanotechnology.

Introduction:

Over the years, the global pace of diagnosis and development of environmentally friendly and sustainable nanosynthesis approaches has increased. It is descended under the name of green nanosynthesis, and the main is added to the reduction and reduction of the use of harmful and dangerous chemicals in the synthesis and complete disposal of nanoparticles. Also, from the effectiveness of the technology, which distinguishes it, efficiency and suitability for the environment, and it has been of great interest, not at the scientific level, but at the biological and medical level, including the treatment of the Sultan for the accuracy and speed of particle transport, their low interaction with the blood, and the reduction of the risk, the nanosynthesis by plants has resulted in biocompatible nanoparticles, so that they can be used in applications. [1]. One of the most important applications that have been used is chemical and physical nanosynthesis methods, including infrared, ultraviolet spectrum and the surface area of the carnation plant, but it faces disadvantages that include the use of hazardous chemicals, the need to control pressure and temperature, high energy consumption, high cost, and time constraints, which negatively affect the environment. [2].

What is Cloves

Clove is known for its medicinal properties and aromatic sweet flavor, and the many benefits of cloves are due to the fact that the compounds in it have many health benefits, as they help support liver health, stabilize blood sugar levels, and are a rich source of antioxidants and bacteria.



Benefits of cloves as an antioxidant Benefits of cloves in protecting against cancer * Benefits of cloves as an antibacterial

❖ The practical part :

1- Preparation of the nanomaterial from (clove stick):

Each of the preparations, which is (Clove Stick) was washed and dried in order to clean them from dust and impurities, and then dried them by an oven with a temperature of 53 degrees Celsius for 20 hours,

and then they were ground by an electric grinder, as the modified sol-gel method was used, by adding 3 grams of herbal matter (Clove Stick) to the zinc oxide solution (4.5 grams of zinc oxide in 200 ml of ionized water) and the mixture was stirred on the magnetic motor. stirrer) for 8 hours, then put the mixture in the vibrating incubator for 6 hours at a temperature of 45 degrees Celsius and at a speed of 120 rpm as in Figure (14), it was observed that the solution changed from adoption to pale white, then the separation of the precipitate by centrifuge at a speed of 3000 d.m. and for 25 minutes, then the sediment was washed several times by deionized water, and then the precipitate was dried by an electric furnace at a temperature of 65 degrees Celsius, and then grinded Precipitation after drying by ceramic mortar to obtain fine powder

1.3 Preparation of activated charcoal

The preparation of activated charcoal is done in many ways and carefully followed, and this method is done by deshydration, carbonisation, and oxidation of organic substances organiques, and most of the materials used in the industrial and commercial manufacture of activated charcoal are environmental wastes.) 7 (

- preparation of raw material

(Clove stick, marmaki and jouyfa haltita) are **washed** several times with ionized water to get rid of impurities and make sure that they are free of dust, then put in a drying oven for 20 hours at a temperature of 55 °C to dry them.

After that, it was cut into small pieces and ground by an electric grinder to obtain the powder of the clove stick, the marmaki and the hollow haltita. .

- Coking

A handmade iron capsule was made and used as shown in Figure (25) in the carbonization process, where 80 g of powder (clove stick, marmaki and jouyfa haltita) was placed inside the capsule at a temperature of 450 °C for two and a quarter hours. .



Figure (25): An iron capsule for charcoal leaf powder, cloves, marmaki and al-jowaifa sweetened at a temperature 450 C°

2. Clove stick powder, marmaki and jouyfeh were extracted after charcoal and then cooled at room temperature.

Chemical activation

- 1- 40 g of charred myrtle leaf powder has been carefully weighed.
- 2- The charcoal was activated by adding (20) ml of NaOH solution with a concentration of 0.1M to the charred powder as the contents are mixed in a large baker homogeneously.
- 3- Placing the homogeneous mixture inside the iron capsule closed at both ends prepared by hand for two and a quarter hours at a temperature of 800 Co .
- 4- The resulting activated charcoal was cooled and washed with ionized water several times to get rid of impurities using a funnel.
- 5- Equivalence of activated coal with 20 ml of hydrochloric acid with a concentration of 0.1M to remove the remaining sodium ions.
- 6- Repeat the process of washing with deionized water several times to get rid of acid residue.
- 7- The pH-meter was used to verify the equation of the acidic function of the prepared activated coal.

8- Dry the prepared activated charcoal for 3.5 hours with a grade of 120 °C.

Adsorption isotherm

The temperature determinant knows the change in the adsorption capacity of a substance within the limits of the adsorption concentration of the isotherm, the standard methods consider the adsorption capacity of the adsorbent and adsorbents, and the morphological form of the adsorbent is formed by drawing the amount of adsorbent on the surface against the concentration of that substance at constant temperature. The scientist Giles

1)1(Isothermal Freundlich Equation

$$Q_e = k_f C_e^{1/n} \dots$$

$$\log Q_e = \log(k_f + (1/n \log(C_e \dots$$

1. Results

Several measurements were used to examine the prepared nanomaterial :

- 1- X-ray diffraction measurement XRD
- 2- X-ray fluorescence analysis XRF
- 3- X-ray scattering measurement EDX

X-ray fluorescence spectroscopy analysis

The use of this technique aims to determine the elemental composition of the nanomaterial prepared from clove grass and zinc oxide, according to the results, the most abundant elements in the prepared sample were Zn, Si and Ca with average values of 78.59%, 2.38% and 1.40% respectively, which shows the percentage of elements included in the prepared nanomaterial, as well as Figure (2) which shows the analysis of X-ray fluorescence spectroscopy.

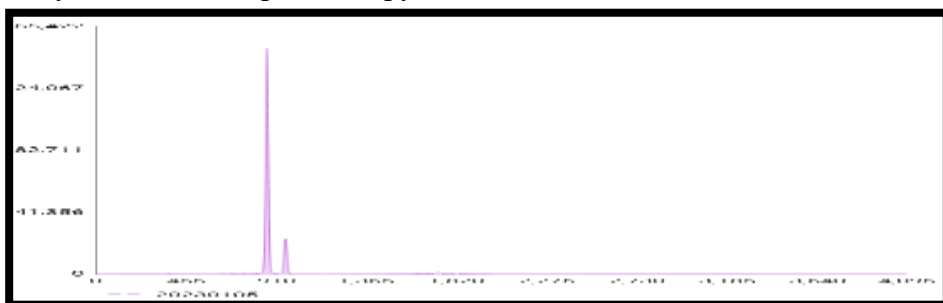
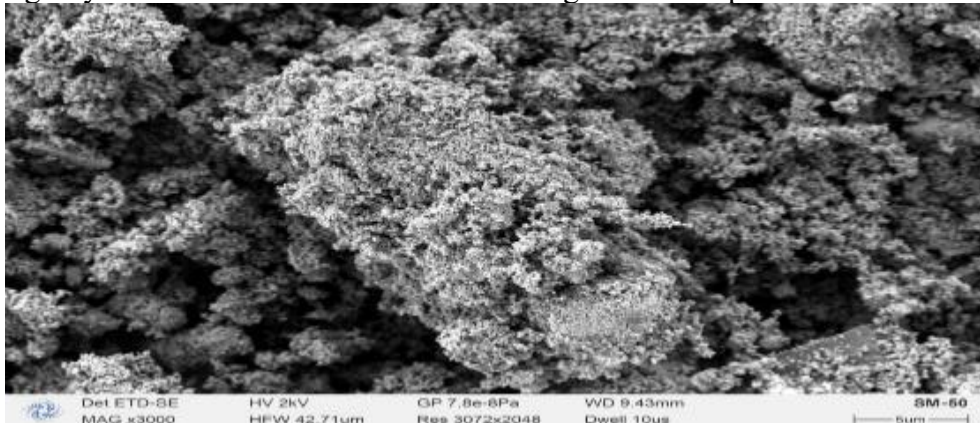


Figure 2 showing the X-ray fluorescence spectroscopy analysis of zinc oxide nanoparticles

X-ray dispersion measurement (SEM):

The image shows a very magnified view of a surface made up of clustered nanoparticles, taken with a scanning electron microscope at magnification up to 20,000 times. The surface appears very rough and zigzagged, and shows dense masses of small particles that bind together randomly to form larger clusters that resemble spongy or porous structures. Dark areas represent cavities or spaces between clumps, while light areas represent particles that reflect electrons more strongly towards individual particles or to the dimensions of small clusters within the field. The existence of a difference in measurements indicates that the material is irregular in size and that the particles are not identical but are distributed in different sizes within the nanofield. The overall appearance suggests that the particles grew heterogeneously and accumulated rapidly, resulting in a large, highly porous surface. This type of structure is common in nanooxides prepared by precipitation, combustion, or rapid drying, where the particles adhere together before they take on a full crystal form. The surface as seen in the image gives the material distinctive properties such as large surface areas and a high ability to adsorb or react, because all these zigzags and voids increase the amount of exposed surface. The image also shows that the particles are strongly bonded within a three-dimensional lattice, giving rigidity to the cluster structure but remaining nano and open



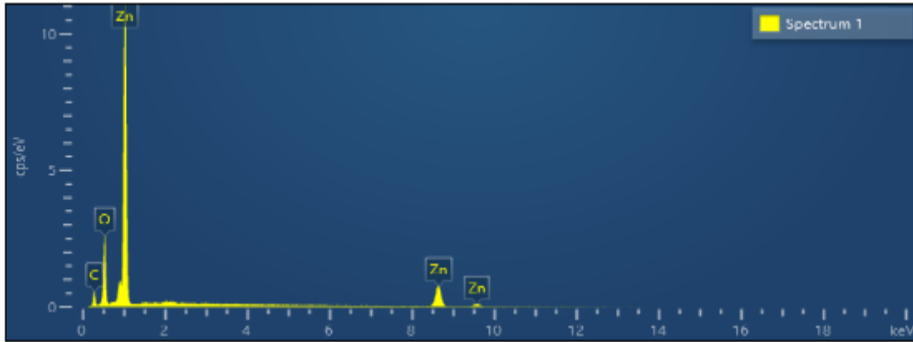


Figure 4: X-ray dispersion spectrum EDX for prepared nanomaterial

Measurement of the surface area of prepared activated charcoal (BET)

The image shows a fine surface taken with a scanning electron microscope at magnification 50 times, and the scene looks like a porous structure surrounded by cracks and ripples resulting from the interaction of the material. The overall appearance suggests that the sample is of an excellent and homogeneous nature, with regular masses of different sizes distributed among them and deep and overlapping spaces between them, suggesting that the material will be effective for purification, preparation and purification. The exposed surface bears sharp bumps and relatively smooth areas along with parts filled with tiny, structure-like holes caused by chemical reactions.

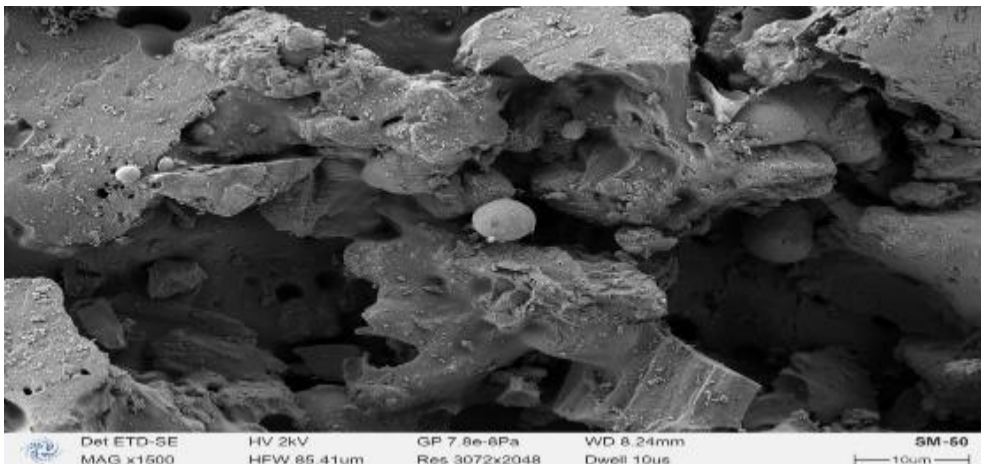


Figure (5) Surface area prepared for coal

X-ray diffraction (XRD)

Zinc oxide nanoparticles were diagnosed using X-ray diffraction (XRD) to determine the crystal shape of the prepared nanoparticles through Miller's coefficients, where the purity of the prepared nanomaterial is identified as well as the size of the nanoparticles using an equation (**Debye Scherer equation**):

$$D = \frac{K \lambda}{\beta \cos \theta} \dots$$

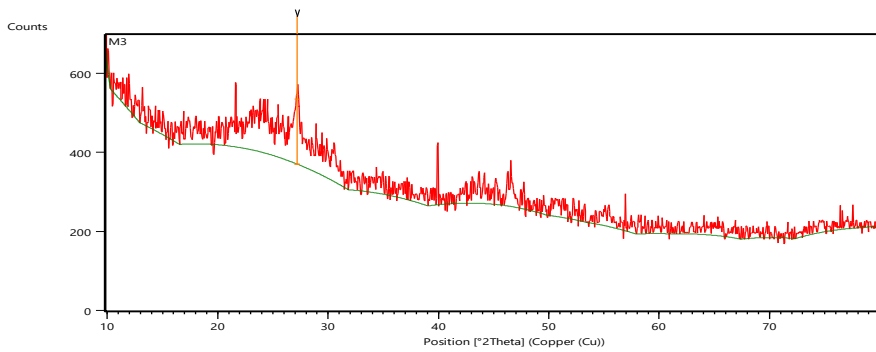


Figure (7) shows zinc oxide

2- X-ray diffraction:

Activated charcoal prepared from clove grass was diagnosed after adding zinc oxide nanoparticles and how the purification was good using an X-ray diffraction device (XRD) in order to know the crystal shape of the prepared nanoparticles through Miller coefficients, where the purity of the prepared nanomaterial is identified as well as according to the size of the nanoparticles using (**Debye Scherer equation**):

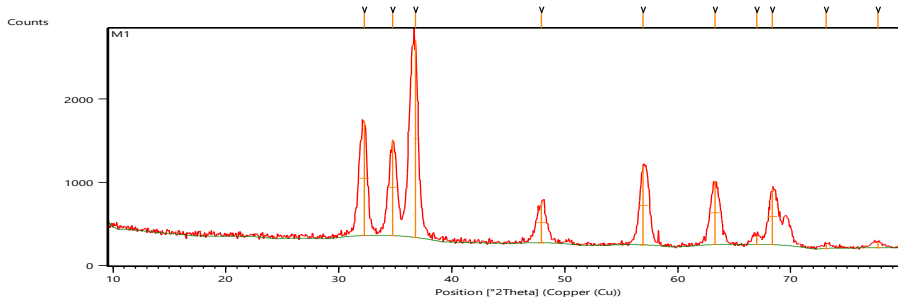


Figure 8 shows the charcoal extract with zinc oxide nanoparticles
Transmission Electron Microscopy (TEM)

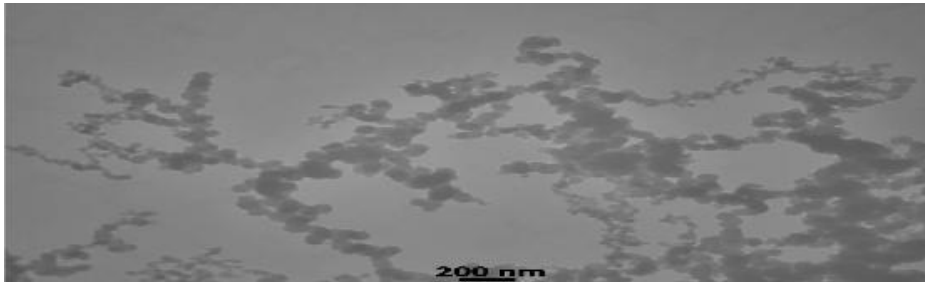


Figure 9: Photonic Activated Coal (TEM) Microscope prepared at
80nm magnification

Determine (λ_{max}) and construct a calibration curve for ZNO:

The maximum wavelength (λ_{max}) of the zinc oxide nanoparticles under study was determined as a specific concentration was prepared for a number of concentrations at which the highest absorption was achieved at the maximum wavelength (372 nm) using the Visible-Ultraviolet (UV-Vis) spectroscopy device within a range of wavelengths (190-800 nm).

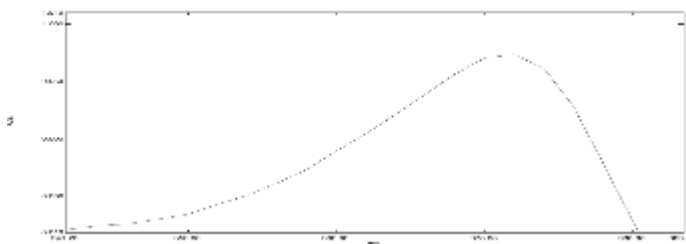


Figure 10 Maximum Wavelength of Zinc Oxide Nanoparticles

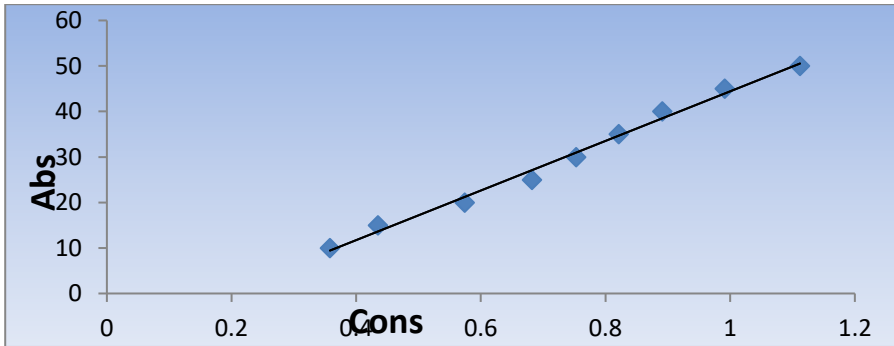


Figure (11): Showing the calibration curve of (ZNO)

Balance Time Setting

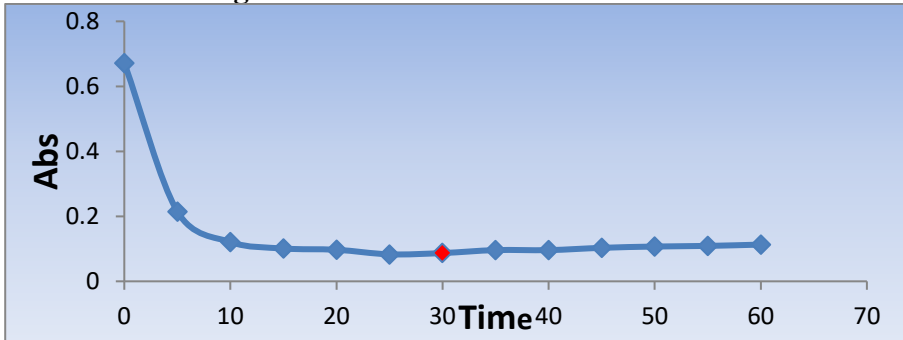


Figure 12: Absorption values change over time for ZNO

Effect of the Adsorbent weight

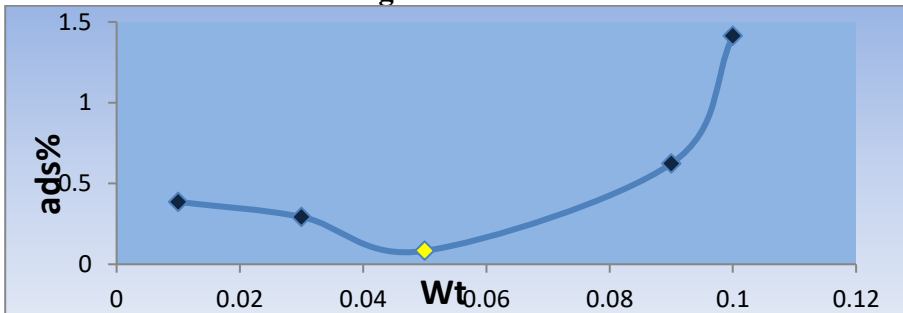


Figure 13: Adsorption efficiency of ZNO activated coal

Larger crane's Equation for False First Rank

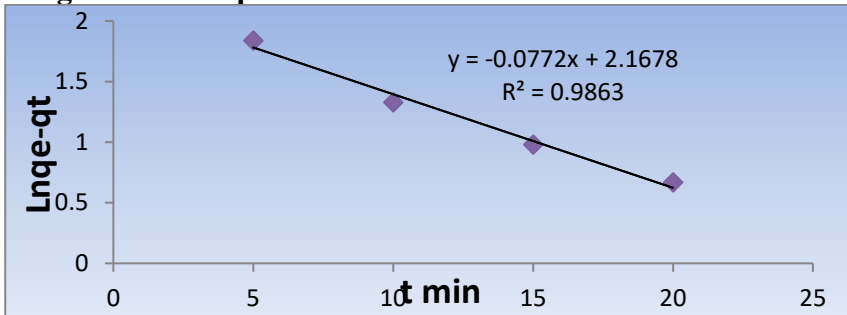


Figure 16 shows the false first-order variables of the ZnO adsorption system.

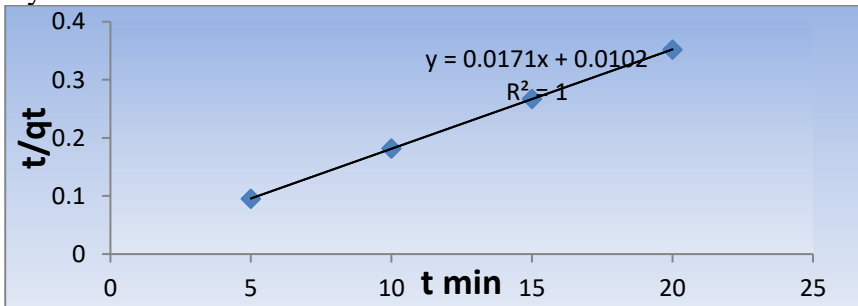


Figure (17) shows the pseudo-second order variables of the ZnO adsorption system

Conclusions

The study proved that the material prepared by the sol-gel method is an active nanomaterial and its surface area is large, and the results were within the nanoscale and contain zinc oxide ZnO, and the results of the surface area characterization (BET) showed that the prepared activated charcoal has a high surface area, and the diagnosis of the active aggregates showed that it contains nanoparticles as well as the shape and topography of the surface of the nano-activated charcoal. High density and neutral acid function of 6.97 (which is similar to the specifications of commercial activated charcoal, for prepared activated charcoal it is chemically inert because it does not contain active aggregates, as well as the best weight (0.03 gm) and the ideal and best temperature (30 °C) and the extent of homogeneity of its surfaces and containing a large number of pores and the shape of the nanoparticles were diagnosed by TEM and SEM techniques..

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