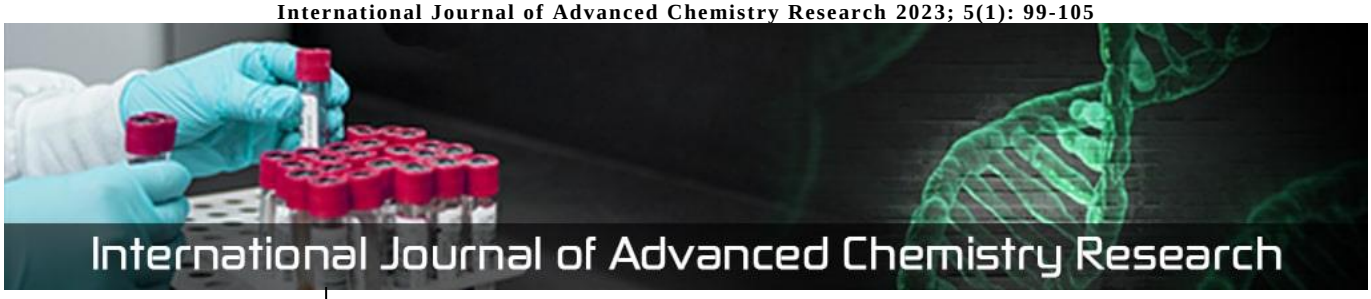

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Preparation and diagnosis of nano-activated carbon from falling sidr leaves

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Abstract
The research included the preparation of activated carbon, at the nano-scale from the leaves of the Sidr plant. the prepared carbon was identified using different techniques, such as infrared spectrum(FT-IR), X-ray diffraction, scanning electron microscope (SEM), dispersion energy X-Ray Detection (EDX) Besides surface area analysis (BET), as it showed the results of a spectrum (FT-IR), the prepared carbon does not contain any active group, that it is chemically inert and this is a good characteristic to be added to the prepared, activated carbon, as shown by the results of (XRD), the average crystal size of the prepared, activated carbon was found to be equal to (9.943)nm, which confirms that the prepared carbon contains nanoparticles, as shown by the results (SEM) and the (EDX) The sizes of the prepared carbon particles are approximately between (43.57-81.02 nm) and that the shape of the stomata (pores) on the surface bear the characteristics of nanomaterials with their properties, and that the highest percentage of carbon was (83.0%), and this result supports the efficiency of the prepared nanoscale activated carbon and its use in the adsorption process, in addition to that, activated carbon was obtained that has a surface area according to the theory(BET) How much (816.13) m^2/g, It is very suitable to be used as an adsorbent and to measure the biological activity against a type of bacteria that causes water pollution. Some physical properties of carbon have also been studied, such as the percentage of moisture, density, acidity function and percentage of ash content, as it has a moisture content of about (6%) and density(0.6)g/cm^3 while the pH was (7.2) and the percentage of ash content was around (2.6%).

Keywords: Activated carbon, nano, adsorption, sidr leaves

1. Introduction
The use of activated carbon in adsorption processes is of great industrial importance because it contains effective sites as it comes surface area Highly versatile adsorbents that enable it to have a high ability to remove pollutants organic and Inorganic water [1], and the need to find absorbent surfaces, made of natural materials arose as a result of the high cost of commercial, carbon and the difficulty, in obtaining it. Therefore, workers in this field began to search for alternatives. Material Good quality and high efficiency depending on what is available in their areas, of presence, using agricultural waste such as sawdust, peels of some fruits and palm fronds, jell as well as fallen leaves, Activated carbon has been obtained at a low cost and at the same time obtaining a clean environment, as these organic materials are subjected to a number of chemical and physical activation methods [3, 2].
Activated carbon is a tasteless carbon material with a black color and has porous properties. Its porosity varies according to the raw material and the method of its preparation. The name activated charcoal refers to a large family of charcoal materials that do not have a specific chemical composition. Its type can be known through its surface properties. and porosity, which is due to the crystalline imbalance between the rings that make up the coal that is being prepared Bimodalities several and using different materials [4], the technical properties of (Activated Carbon) thousands of years ago, and before (450) years BCY was Sand and activated charcoal will serve as a filtering agent for drinking water purification, as well as Use come medical, And in particular Treating poisoned patients With the passage of time, the uses of activated carbon have increased globally [5], In addition, that Adsorption is a simple technology with low economic costs compared to other treatment methods. For this reason, many researchers have resorted to developing different adsorbents.

And from between adsorbents common and used is (activated carbon), and has the great development in the applications of the adsorption process prompted the need to provide new adsorbent materials to be used in the treatment of environmental pollution. On These materials should be efficient and have a low economic cost retrieve it then reuse it again, There are many studies in this field that have been prepared [6], activated carbon from coffee grounds (CW) for adsorption of Congo red dye from its aqueous solutions, I also use [7] kind of common waste (wheat husks) as an environmentally friendly and low-cost adsorbent for adsorption of Congo Red dye from its aqueous solution., has also been studied [8] adsorption of a nanomaterial prepared from fallen lemon leaves in the medical and industrial fields, the research included the preparation of activated carbon from, a cheap and environmentally friendly raw material. like that A study [9] included the preparation of nano-activated carbon as an adsorbent from a plant source, which is eucalyptus stems, and was diagnosed by several techniques, as well as studying the adsorption efficiency and the factors affecting the adsorption process. prepare and the diagnosis of a new appetizing substance from primary plant sources (walnut shells and date cores) as it was characterized by different techniques that showed the properties and topographical characteristics of the prepared Nano carbons, and in aim of this study It is the preparation of a new nano-adsorbent material from environmentally friendly, available and inexpensive plant sources, which are the leaves of the Sidr plant, as well as diagnosing it with several techniques and measuring some of the specifications the prepared, activated carbon, for purpose of using it in the adsorption process.

Table 1: Instruments used in the study.

No.	the device name	Model and origin
1	UV -Visible Spectrophotometer	SHIMAMDZU(UV-1900 PC)
2	Shaker with water bath	Julabo, SW23
3	pH- Meter	Jenway (3510 pH- meter)
4	Laboratory Oven	M420-elektro.mag
5	Furnace Electrical	Carbolite- England its max
6	X-ray diffraction	Philips-PW1730
7	Infrared Spectra (FT-IR)	1800FT-IR Shimadzu
8	Scanning Electron Microscope (SEM)	TESCCAN-MIRAIII
9	Energy Dispersive X-Ray Detection(EDX)	TESCCAN-MIRAIII
10	The Surface Area Analysis (BET)	BELSORP MINI II

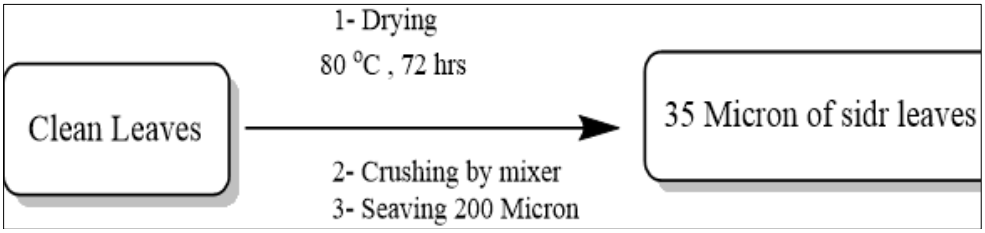
2.1 Preparation of Activated Carbon from Leaves of the Sider

2.1.1 Preparation of Raw Material

Firstly: The raw material (the leaves of the Sidr plant) were taken in their natural form trees (Kirkuk Governorate - Iraq). After that, these leaves are washed with tap-water to remove dust, then washed with water free of ions, after which they are washed with ethanol to get rid of all organic pollutants present on the surface of the leaves the plant.

2.1.2 Secondly: The clean Sidr leaves are placed in, an oven at temperature of (80 ºC) degrees Celsius for a period of (72) hours, then the leaves are crushed by a home blender and then grinded to obtain a very fine powder, after that the resulting powder is passed through molecular sieves with a size of (micron200) to get a fine powder from the leaves.

2. Experimental part



2.1.3 Carbonization

1. The carbonization process was carried out by taking (50 gm) of leaf powder sidr plant, after dried and burn it in a bowl of stainless steel, which is coated with a layer of nickel, and with a temperature(400 ºC) for two hours.
2. Leaf powder is extracted Sidr after being carbonized and then cooledaat room temperature.

2.1.4 Activation Chemical

1. Taking Weight (10) gm of leaf powder Sidr charred non-stimulant carefully.
2. Perform the activation process by adding (10) ml of (NaOH) solution with concentration (0.1M) to the powder resulting from the carbonization process, as the content is completely mixed in the beaker.
3. Put the homogeneous mixture inside Nickel plated stainless steel bowlfor two hours at a temperature (800 ºC).
4. Cool the resulting activated charcoal and wash it with deionized, water several times to get rid of, itremains Impurities and (pH) equation.
5. Activated charcoal equation (10) ml of hydrochloric acidat a concentration of (0.1M) to remove the remaining ions the base.
6. Repeat process the wash with water Free of ions several times to purify it of residue Effect sour acid.
7. Use (pH-Meter) to verify the neutrality of the acidity of the prepared activated charcoal.
8. Dry activated charcoal record for a duration (4) hours (120 ºC) degree.
9. Grinding the activated charcoal resulting from the previous step by means of a mechanical mill to obtain fine particles, then it was sifted by molecular sieves with a size of (35micron) It was kept inside a sealed package and in a desiccator, isolated from air and moisture [8].

2.2 Study of the physical and chemical properties

2.2.1 Determination of moisture

1. Taking weight (0.5) gm of activated charcoal prepared in a weighted ceramic lidMpreviously, and placed in a drying oven at a temperature (110) ºC for three hours.

2. The model is placed in a desiccator and allowed to cool to room temperature, after which it is weighed and through the difference is calculated percentage. The adsorbent water representing the contentfor moisture.

2.2.2 Determination of Ash
1. Taking weight (1) gm from prepared activated charcoal, and put in an earthenware jar, previously weighed, then it is done Placing the lid in the burning furnace at the time of burning (1000 ºC) for three hours.
2. Place the eyelid in the dryer and leave to cool to room temperature. Then weighed with a balance sensitive, According to the weight of the residual, ashes the percentage, of ash was calculated.

2.2.3 Determination of Density
1. A quantity of prepared, activated charcoal was placed in volumetric flask (5ml) Turning on the activated charcoal Volume the vial up to the mark.
2. Coal minutes made pain active one level, at the edge of the mark by lightly, tapping the sides of, the vial or shaking, as the prepared charcoal occupies the volume, of the cannula used [8].
3. Is done afterward weigh the prepared charcoal contained in the volumetric vial using a sensitive balance, and through the difference before and after the addition of activated charcoal was calculated Density of relation the following:

Density (g/cm3) = mass/volume (1)

2.2.4 Determination the pH
1. Added (1gm) of activated carbon to (10) ml of the distilled water, Then shake the solution for, a while (30 min) by a vibrator, at a temperature (25ºC).
2. The solution was filtered, and the pH of the filtrate was measured, by an instrument the (pH-Meter).

2.2.5 Adsorption of Methylene blue dye
1. take (0.1) gm of the prepared carbon and put it in a conical flask, to which a known amount of (100) ml of methylene blue dye is added with a concentration, of (20ppm) The dye solution was prepared, by dissolving it (2 mg) of the dye in, (100) ml of distilled water.
2. the sample is placed, in the electric shaking device for a period of, (60 min) at the laboratory temperature.
3. When the color disappears, the activated carbon is concentrated, the solution is separated by filtration, and the absorbance is measured at a wavelength of (665) nm It is the wavelength at which methylene blue absorbs [11].

3. Results and Discussion
The preparation of activated carbon dates back to distant times, and many researchers have devised many methods of preparation based on various sources, as this research included, the preparation of, the adsorbent (activated carbon) from cheap, available and environmentally friendly raw materials, which are the leaves of the Sidr plant.

3.1 Characteristic evaluation of the prepared activated carbon
3.1.1 Determination of moisture ratio
After calculating The ratio percentage of humidity in the carbon The activator was found to be equal (6.0%) and thus be within the allowable limit of moisture content, for activated charcoal global which is less than (10%) [12].

3.1.2 Determination of Density
The density was measured carbon The activator prepared in this study using equation no (1) so is found that has density good equal (0.6)g/cm^3, and she is good at compare it a prepared types [13, 14] has been shown It is highly efficient for adsorption.

3.1.3 Determination of Ash ratio
according to the weight of, the leftover ash prepared carbon, and found equal (0.08) gm, from which the percentage was calculated and found to be equal to (%2.6) as this percentage is within, the near allowed in international and commercial, activated charcoal specifications the ash content usually ranges from(1-5%) of the original coal, weight under investigation [14].

3.1.4 Determination of pH
Use a measuring device (pH-Meter) to trace the pH of candid activated carbon record when washing it after activation it has been found that value of the pH final reach (7.2) This shows that coal the activator The record is equal, It is one of the good properties of the prepared activated charcoal, This indicates that the prepared charcoal does not contain acidic or basic groups This is a good feature added to the advantages of prepared activated charcoal [9].

3.2 Topography Characterization
For the purpose of topographic diagnosis (Topography) Surface activated carbon for all samples were measured using infrared spectroscopy (FT-IR) and X-ray diffraction technique (XRD) and a scanning electron microscope (SEM) as well as the analysis of the surface area (BET) and as follows:

3.2.1 FT-IR spectroscopy
Infrared spectrum (FT-IR) for carbon the activator record, this is to see, if activated charcoal contains, effective aggregates (Effective groups) or not, and represents the appearance (1) Collapse infrared, for charcoal the activator purer.

Evidenced by the shape of the infrared spectrum (FT-IR) the prepared, activated carbon does not contain, any active group effective groups, It may affect the adsorption process of the materials under study, and thus we conclude that the prepared activated carbon is (chemical inert)This is a good characteristic, added to the prepared, activated carbon.

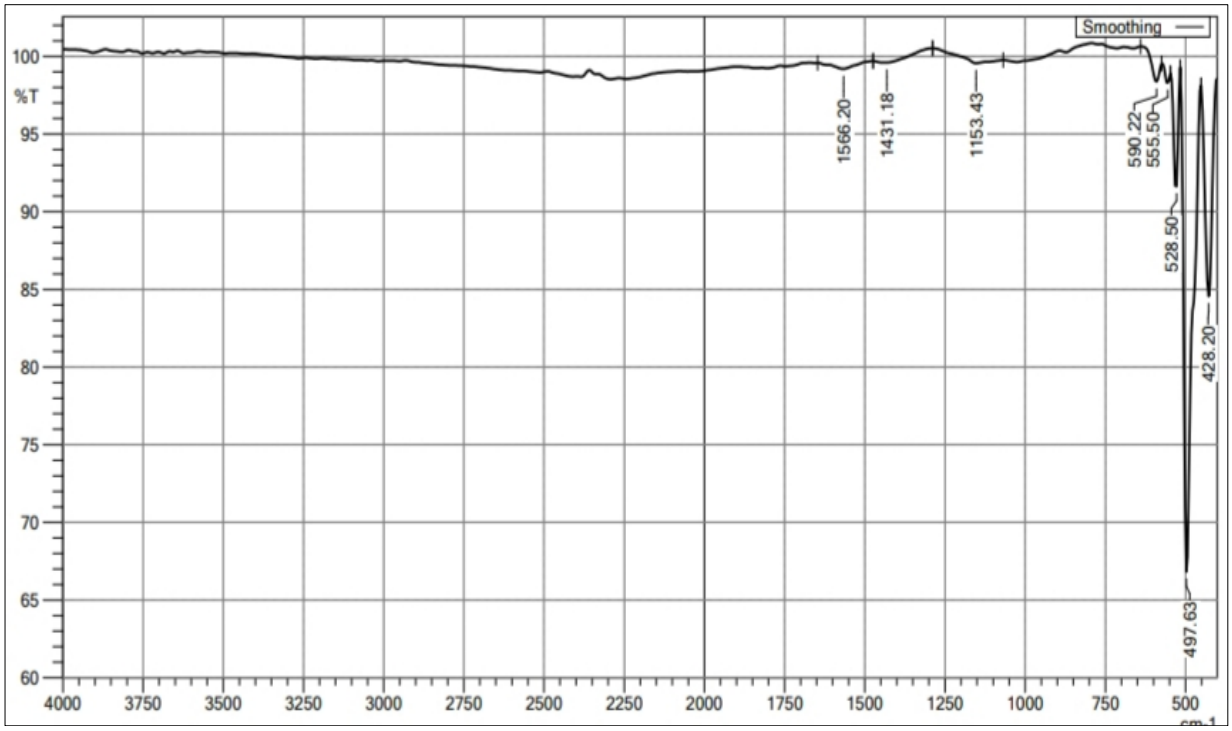


Fig 1: (FT-IR) for the prepared, activated carbon.

3.2.2 X-ray diffraction (XRD)

Surface diagnosed the subject of mezze is under study using an X-ray diffraction machine (XRD) to find out the crystal shape of the prepared nanoparticles through Miller's coefficients as it is recognized The purity of the prepared coal as well as the calculation of, the size of the nanoparticles using, the Debye Scherer Equation [16]:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

As if:

D = particle size in nanometers.

K = Debye constant and equal (0.94)

λ = the wavelength of the x-ray and is within limits (1.5406) angstrom for the element copper, and it is converted into a unit of nanometers, as each (1 angstrom = 10^{-1} nm), so the wavelength is equal to (0.15406) nm.

β= total width at the largest half of the apex (Full Width at Half Maximum) (FWHM), measured in degrees (Deg) and converted to (radian) units, as the FWHM value is multiplied by the amount.$\frac{\pi}{180}$

θ= cosine of the diffraction angle in units (Degree).

Figure (2): shows a diffraction spectrum X-ray (XRD) for the prepared activated carbon.

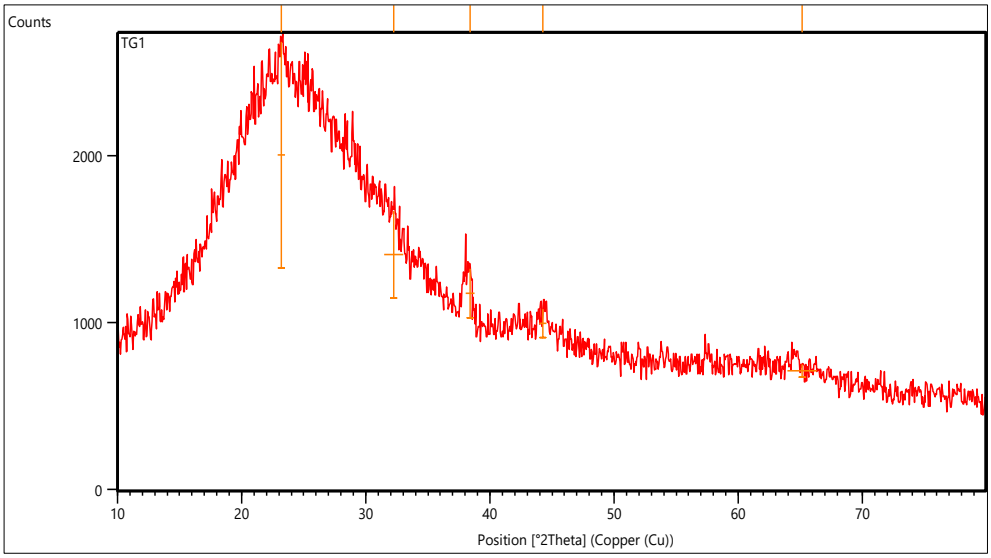


Fig 2: X-ray diffraction spectra (XRD) for the prepared activated carbon.

and figure (2) shows the data values obtained from the (X-ray) diffraction spectra of the five strongest beams, which were used to calculate the average crystal size of, the prepared activated carbon, which was found to, be equal to(9.943)nm, which confirms that the prepared charcoal contains nanoparticles.

Table 2: X-ray diffraction values (XRD) and the values of the sizes of the nanoparticles.

No. of peak	Pos. [°2Th.]	FWHM Left [°2Th.]	Rel. Int. [%]	Cry. Size [nm]	over. Size [nm]
1	23.2138	0.5904	100.00	13.7366	9.9437
2	32.2351	1.5744	38.42	5.2523	
3	38.3871	0.6888	21.87	12.2123	
4	44.2513	0.5904	12.53	14.5254	
5	65.1527	2.3616	5.71	3.9919	

3.2.3 Scanning electron microscope (SEM)

It is a type of electron microscope, through which the surface of, the sample is photographed using, a stream of electrons, It gives complete information about terrain, the surface and composition of the prepared adsorbent material, The scanning electron microscope plays a major role in understanding the nature of adsorbed matter [17]. The sizes of the prepared coal particles appeared approximately between (43.57-81.02 nm), and as shown in the following figure (3):

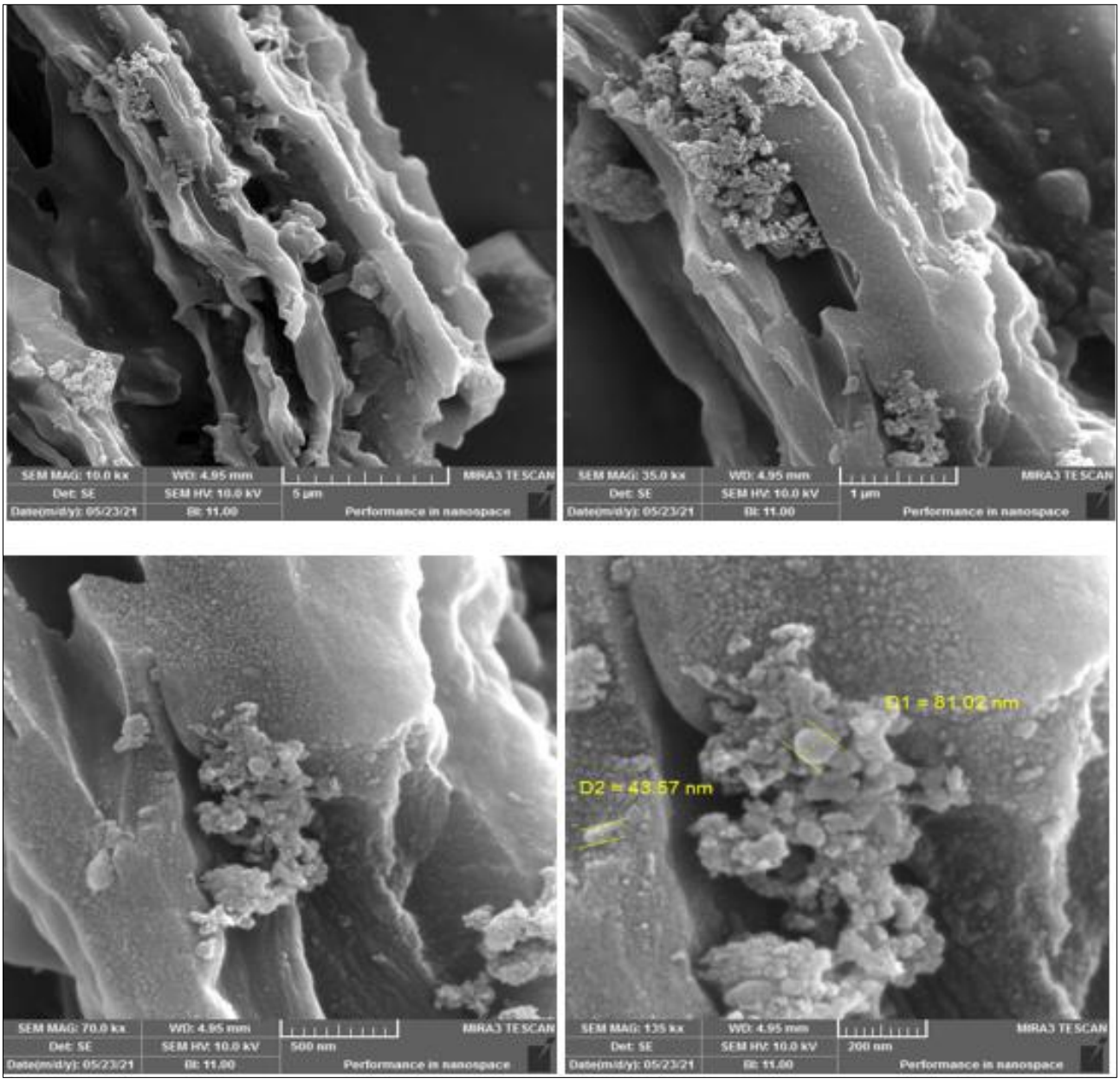


Fig 3: (SEM) measurement activated carbon.

3.2.4 X-Ray energy dispersion spectroscopy (EDX)

It is analytical technique, used to Element analysis to know, the (Chemical Composition) For the materials under study, this technique was used to find out the constituent elements of the prepared, activated carbon, as the Measuring the presence of charcoal nanoparticles, in very large quantities, as we note the presence of to carbonprepared as shown in (Fig.4), and the (tab.3) gives the percentages of the sample elements (TG_1) to check the chemical composition and purity of the prepared sample The results showed that the highest percentage of carbon was (83.0%), and this result supports the efficiency of the prepared nano-activated charcoal and its use in the adsorption process.

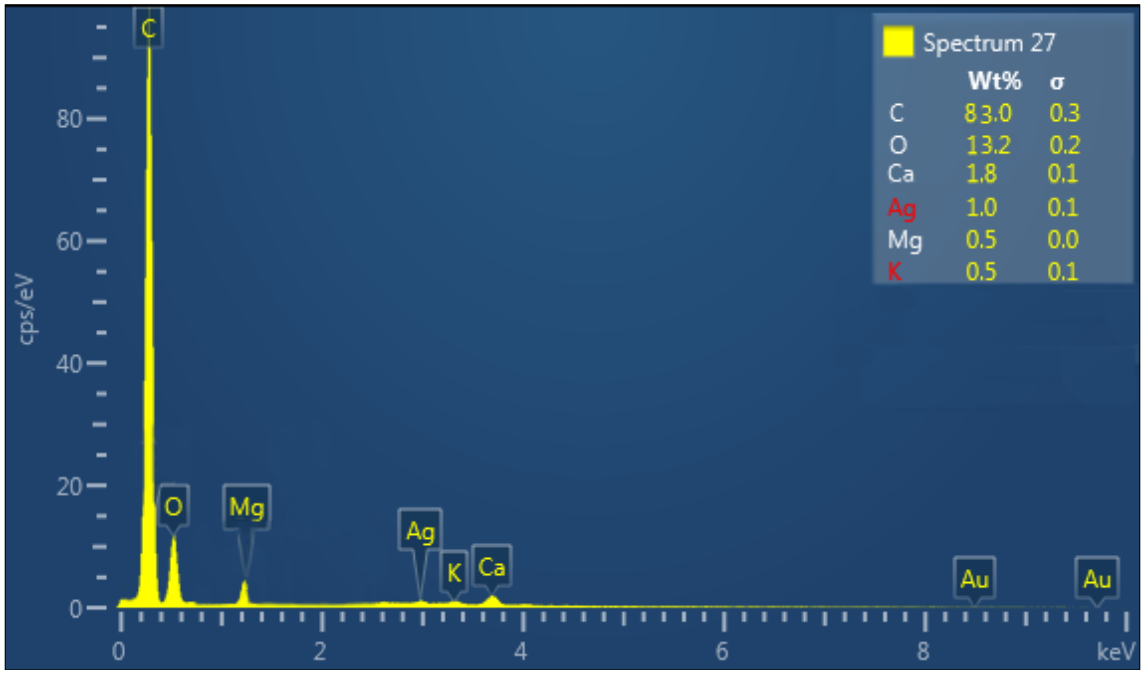


Fig 4: X-ray energy dispersive spectrum, (EDX) activated carbon (TG_1).

Table 3: Weight and atomic ratios of the constituent elements of the activated carbon (TG_1).

Element	weight %	Atomic %
C	83.0	87.29
O	13.2	11.42
Ca	1.8	0.59
Mg	0.5	0.27
Ag	1.0	0.13
K	0.5	0.15
Totals	100	100

3.2.5 Determination of Surface Area (BET)

Use a surface area measuring device (Prep 060 and Gemini BET Machine), in order to measure the surface area and specific size of the nanocharcoal prepared from the leaves of the Sidr plant at different temperatures, and the measurements were made and the required functions or factors were extracted using several methods, which are [18]: (BET method, Langmuir method, t-method, BHJ method)

The aim of this study is to know the surface area of the charcoal prepared by activating it with plant materials, and after selecting the experimental activation of the system, activated charcoal possessing surface area has been obtained according to theory (BET) its amount (816.13) m^2/g and as in Table: (4), which is very suitable for use as an adsorbent, as well as calculating, the surface area of the prepared carbon, according to the theory (Langmuir), and found that it is equal to(812.63) m^2/g.

Table 4: the results of the analysis (BET) for the prepared activated carbon (TG_1).

BET plot		
V_m	164.53	[cm^3(STP) g^{-1}]
as, BET	816.13	[m^2 g^{-1}]
C	17867	
Total pore volume(p/p0=0.990)	0.4949	[cm^3 g^{-1}]
Mean pore diameter	2.9864	[nm]
Langmuir plot		
V_m	163.73	[cm^3(STP) g^{-1}]
as, Lang	812.63	[m^2 g^{-1}]
B	29,874	
tplot		
Plot data	Adsorption branch	
a_1	926.77	[m^2 g^{-1}]
v_1	0	[cm^3 g^{-1}]
BJH plot		
Plot data	Adsorption branch	
V_p	0.2876	[cm^3 g^{-1}]
rp, peak(Area)	1.21	[nm]
ap	196.14	[m^2 g^{-1}]

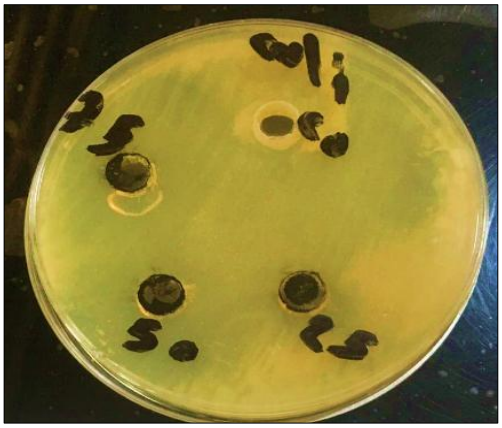
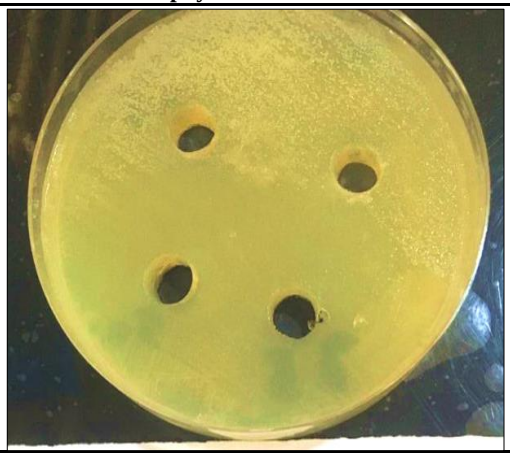
3.2.6 Biological effectiveness

The antibacterial activity was studied using two types of bacteria:

It is a positive bacteria (Staphylococcus aurous) (2)-Negative bacteria (*E. coli*).

These types of bacteria were selected; Because of its importance in medicine and because it causes some diseases [18], the results showed that the inhibitory effect increased with the increase in the concentration used, and the inhibitory ability of the compounds also depended on the

type of compound. activated carbon(TG_1) as well as on the type of bacteria isolated, in the case of using the compound TG1 showed moderate sensitivity against (E.coli) bacteria compared to the control laboratory, while the results showed that the inhibitory effect against bacteria(Staph. aurous),It increased with the increase in the used concentration, as the compound showed medium sensitivity compared to the control coefficient, the average diameter of inhibition was in the case of using the concentration of (50) mg/ml of the compound at (27) mm, while the inhibitory ability increased to reach (33) ml in the case of using the concentration of (75) mg/ml.

	E. coli	**Staphylococcus aurous**
TG1		
Cont.	0	0
25%	13	0
50%	17	27
75%	18	33

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