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Absorption and Desorption Remediation of Environmental Pollution to Remove Heavy Metal Ions From Waste Water Using Batch experimental System

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ABSTRACT

Graphite is the substance of allotropes of carbon forms with diamond, and amorphous carbon, Graphite considered one of methods that we can use it for adsorption of heavy metals ion from water, by a new sorbent material having functionalized for solid phase extraction (SPE) metals (Zinc and Cobalt) from aqueous solutions. Firstly we used Graphite substance by chemical process to convert it to Graphene and graphene oxide has been used in oxidation by hummer method process by using H_2SO_4 and $KMnO_4$ that considered a good oxidizing materials. Graphene oxide is a Composite material fabricated from Graphite. Micrometer thick films of graphene oxide paper are also named as graphite oxide membranes, secondly we silanized it with 3-(trimethoxysilyl) propylamine to get (GO-Si) product, thirdly react this product with functional gropus (Aldehyde) (3,5,Di-tert-butyle-2-hydroxy-benzenaldehyde) to get (Go-Si-Ald) that form graphene Schiff base material, that we used it to several process under different laboratories factors such as (pH, different concentrations of metals / Zinc and Cobalt, different stirring time, with different temperatures), The adsorption of metals (Zn and Co) were investigated successfully, this is one of development method for treatment of waste and drinking water for reuse. This method is benefit to solve environmental problem which is harmful to health. All these work made by several instrumental analysis such as ICP, FTIR analysis, EDX, Uv-visible, TGA, TEM, SEM, XRD Analysis.

1. Introduction

There are several types of water pollutants include pathogenic organisms, oxygen-demanding wastes, metals (Zn&Co), plant nutrients, synthetic organic chemicals, inorganic chemicals, microplastics, sediments, radioactive substances, oil, and heat, in this work using the Graphite substance that considered one types of Carbon element Graphite substance as hybrid material, that considered one of essential or excellent absorbance of heavy metal ions aqueous solutions, in this study I used two metal ions firstly, Zinc is relatively harmless compared to several other metal ions with similar chemical properties. Only high doses are toxic, so acute zinc poisoning is rare[1].

Zinc is a mineral. It is referred to as an "essential trace element" because it requires only very low levels of zinc for human health. Since the human body does not store excess zinc, it must be consumed regularly as part of the diet. Common food sources for zinc are red meat, poultry and fish. Zinc deficiency can lead to dwarfism, reduced taste sensitivity of food and the inability of testes and ovaries. It is also used to strengthen the immune system, improve the growth and health of infants and children with zinc deficiency and for the treatment of the common cold and recurrent ear infections, influenza, upper respiratory tract infections, prevention and treatment of lower respiratory tract infections, swine flu, bladder

infections, Tinnitus and severe head injuries. It is also used for malaria and other parasitic diseases [2].

Zinc is an essential element whose importance for health is increasingly recognized and whose lack of disease can play an important role. Zinc is one of the organism's most important trace elements, with three important biological functions such as catalyst, structure and regulatory ion. Zinc binding motifs are found in many proteins that are physiologically encoded by the human genome, and free zinc is regulated primarily at the level of individual cells. Zinc has a crucial influence on homeostasis, immune function, oxidative stress, apoptosis and aging. With zinc deficiency, significant disturbances are of great interest to public health [3][4][5].

Secondly, cobalt is widely distributed in low concentrations in the environment. People are exposed to low levels of cobalt by inhaling polluted air, drinking contaminated drinking water or eating food. The cobalt concentrations in the soil range from about (1 to 40) ppm and the amount of cobalt in the air is less than (2 ng / m 3). Mineral cobalt ions are widely used trace minerals in nature. Trace elements are essential in certain quantities for normal physiological function; They play a role in the prevention of malnutrition, the function of the immune system, the regulation of gene expression, the antioxidant defense and the prevention of chronic diseases. The only biological function known to cobalt is its role as a mineral component of vitamin B_{12}, also known as cyanocobalamin, while other cobalt compounds have been classified as environmentally and antibody toxic after overexposure [6][7][8].

Widely used in nature and part of many human activities. Although cobalt plays a necessary biological role as a mineral in vitamin B_{12}, overexposure has been proven to have many adverse health effects. detection and measurement methods for assimilation and interpretation, reported health effects, and common concentrations. Less than (300) micrograms / liter (100 micrograms / liter have a safety factor of 3), blood and endocrine disruption are the primary health endpoints in healthy people, and chronic exposure at acceptable doses is not expected to be a risk to the patient Patient represents health. Cobalt in less than 1 mg, of which (0.36 mg) in lipids, (0.3 mg) in hair, (0.28 mg) in bone, (0.2 mg) in muscle and (0.11 mg) in the liver [9].

2. The aıms of thıs study

This study Investigate the effects of pH and ionic strength on Zn (II) and Co (II) sorption, This thesis demonstrated the broad applicability of this fascinating material in environmental pollution cleanup by aqueous samples under the various laboratory conditions such as pH, concentration, temperature, and time effect on their adsorption capacity and reusability, For improvement of hybrid materials, Preparing final product 3, 5-Di-tert-butyl-2-hydroxy-benzenaldehyde on GO, For protection of environmental pollution such as water pollution by Removal of heavy metal ions, For making comparative between Graphene oxide with aldehydes under different concentrations of metals that when react with Zinc and Cobalt metal ions, Know how much Graphene oxide can absorb heavy metal ions, The adsorption of metals (Zn and Co) were investigated successfully this is one of development method for treatment of waste and drinking water for reuse, In other hand this method is leading to significant environmental problem which is harmful to health by a new sorbent material having functionalized for solid phase extraction (SPE) and discovered its compliance based on the removal of some heavy metal ions. .

3. MATERIALS AND METHOD

3.1. Oxidation of Graphite (Synthesis of Graphene Oxide from Graphite)

3.1.1. Steps of Cold bath (ICE Bath):-

Starting with (5) gm of Graphite, then Adding (250) ml of H_2SO_4 in a beaker, ICE Bath + Magnetic stirrer. The rotating field may be created either by a rotating magnet or a set of stationary electromagnetic , placed beneath the vessel with the liquid) . because of its small size , a stirring bar is more easily cleaned and sterilized than other stirring devices, Addition of Graphite on H_2SO_4 in ICE Bath, for (30) minutes , addidng (8gm) of $KMNO_4$ as a Strong oxidizing Agent Adding on solution Mix on ICE Bath (1 hour) addition slowly then waiting for (2hr.) 10:35 to 12:35, all these steps shows in figure (1) below.

Fig. 1: Cold steps oxidation of Graphite to Graphene and Graphene Oxide

3.1.2. Steps of Warm Bath:

Getting Graphite solution out of ICE Bath and put in warm bath at 35C for (1) hour 12:50 to 1:50 pm, NOTES:-We have to check the temp. Of sample it shouldn't decrease less than 25°C, then Adding (100ml) of D.W at (40-60)°C for (50) minutes, Addition of D.W in (50) minutes, After addition the temperature of mix must be (50°C) (green –brown) shows in figures below (2) (3).

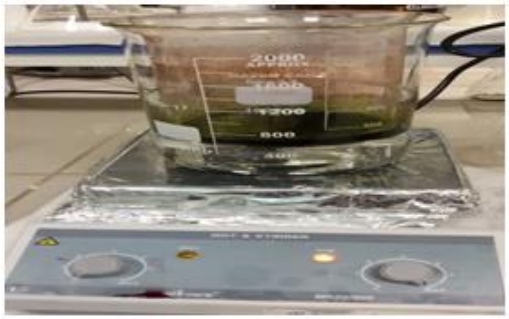


Fig. 2 and 3: Warm steps of oxidation of Graphite

For (1) hour the sample will be left on stirrer (35°C) water bath warm (chocolate color). Strring for 1 hour Temperature (40°C above 50°C), Increasing the temperature to (95°C-93°C) with bubbles waiting for 15 minutes, Addition of (30) ml D.W (600ml) little yellow, Addition of (20ml) H_2O_2 Dark brown - yellowish brown, Wait for (15) hours over night, Preparation of 5% (1000 ml) HCL for washing , Filtrations of sample by Buchner funnel, Washing sample by HCL 5%, The washing with pure water D.W shows in figure (4) below.

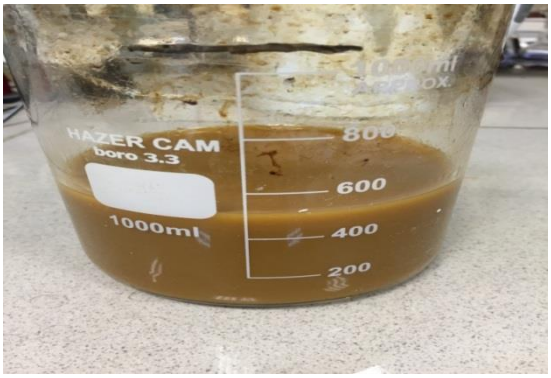


Fig. 4: Continously of warm steps and additional of D.W to solution

3.1.3. Steps of Filtration

I decantate the sample solution. addition of HCL (5%) (800ml) on the ppt then make stirring it with magnetic stirrer for couple minutes. then leaving the sample solution till all ppt (Digest) on the bottom of beaker . After that decantate it again, then Waiting for 25 days to drying grapheme we will get figure (5) below :-

Fig. 5: Filteration steps to find the powder of Graphene Oxide

Oxidizing of Graphite by hummer method, by using H_2SO_4, $KMNO_4$, H_2O_2 in 65°C for 15 hr to get Graphene oxide (GO) shows in Reaction **(A)** below

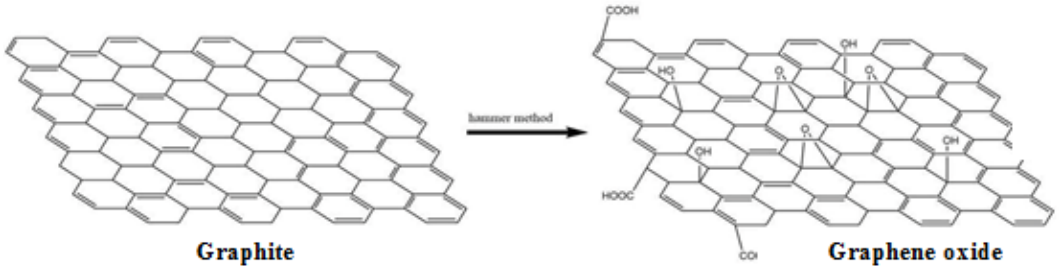


Fig. A: The reaction of formation of Graphene Oxide

3.2. Steps of Silanization (Salts) to Get Graphene Oxide - Silan Substance:-

Taking (6) gm of Graphene Oxide (GO) that i get it from first step and added it to (500) ml Dry Ethanol 96%, Under the flut then stirr for (2) days, With ballon for inertness (N_2) inert gas , I used N_2 gas because of I don't want the air follow through the instrument, Addition of (50) ml (3-triethoxsilyl–propylrimine) (Silanization) by injection closing the ballon from top of condenser then opening one arms of flask and inject (3-triethoxsilyl –propylrimine) into it , Leaving sample stirring for 2 days at (60°C-65°C), that shows in figure (6) below

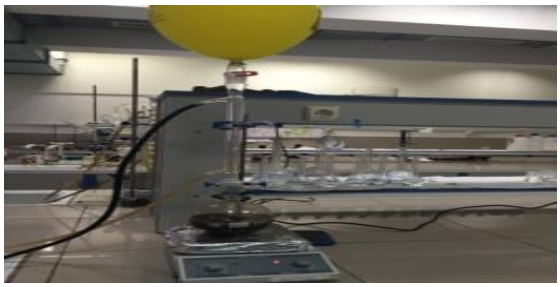


Fig. 6: Instrumental of Silanization steps of Graphene Oxide

3.2.1. Steps of Filtration:-

Filtration of sample that appear in figure (8) below, Addition (100 ml) of methanol (to dilute the unreacted Silanization molecules), Then washing by D.W, Drying at (35°C - 40 °C) in oven, Grinding that shows in figures (7) & (8) below

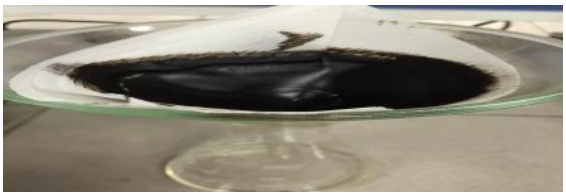


Fig. 7: Filtration of Silanization step of Graphene oxide

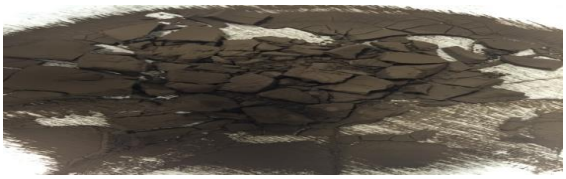


Fig. 8: Solution of Graphene Oxide Silanization after drying

Then i silylated the Graphene oxide (GO) with 3 (trimethoxysilyl) propylamine for salinization Graphene oxide under flux for 2 days to get product shows Reaction **(B)** below

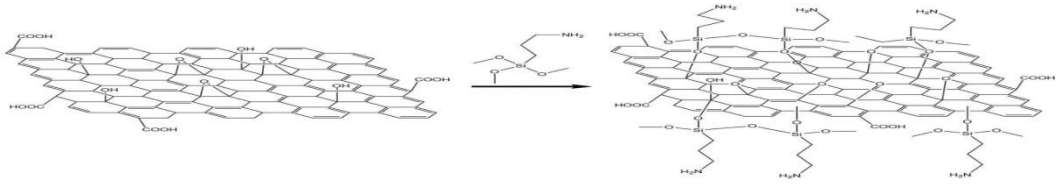


Fig. B: Shows the Graphene Oxide Reactions with Silanization

3.3. Steps of Additional of functional Groups (Aldehydes) to Graphene Oxide:-

Taking (1) gm of Aldehyde (3,5,Di-tert-butyle-2-hydroxy-benzenaldehyde) (that form legend with sample) and added to 30 ml absolute ethanol in round flask, Stir until the sample dissolve, Weigh (1) gm of Graphene oxide-Silanization (GO-SI) and added to the flask containing (legend + ethanol), Set up the flask with condenser, Raise temp. Of the sample between (60°C -65°C) for (30) min. and leave the sample for (30) hours under reflux that appear in figure (9) below.

Fig. 9: instrumental of additional aldehyde (functional group)

3.3.1. Steps of Filtration:-

After 30 hours then Filtration shows in figure (10) below

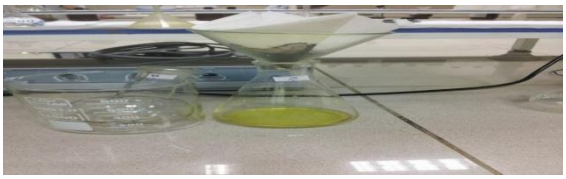


Fig. 10: Filteration process of sample of aldehyde

Leaving the filtration sample to drying for 2 days to get it as powder, the product that produced Graphene oxide-silanization from second step will react with aldehydes (3,5-Di-tert-butyl-2-hydroxy-benzenaldehyde) to get the GO-schiff base material final product of this work that shows as reaction (C) below :-

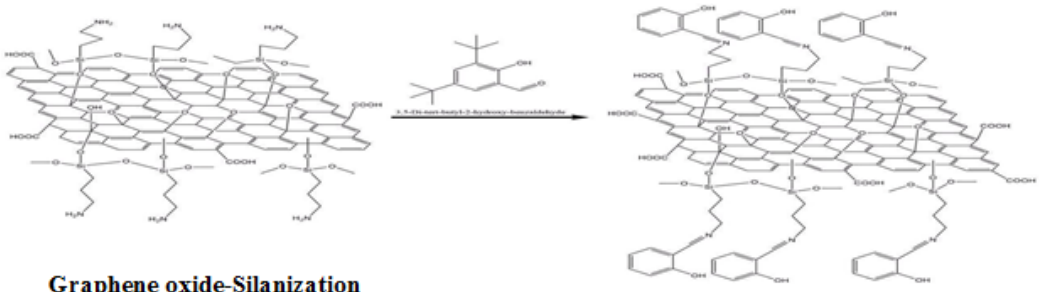


Fig. C: shows the Reactions of the Graphene oxide with Silanization and Aldehyde

3.4. Preparations:

3.4.1.Preparation (Acid & Base)

A)Preparation of HcL (I Used this law below for Preparation Acids):-

$N = \frac{D*1000}{Eq.wt}$, $N = \frac{1.14*\frac{32}{100}*1000}{36.46}$, N=10.005, N1*v1=N2*V2, 10.005 *V1=0.1 *100 , V1=1 ml. shows in figure (11) below.

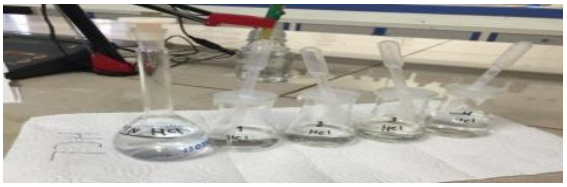


Fig. 11: Preparation of Acid HCL

B)Preparation of NaOH I Used this law below for preparation basics):-

$N = \frac{WT*1000}{Eq.wt*v}$, $0.1 = \frac{WT*1000}{40*100\ ml}$, WT = 0.4 gm of NaoH, shows in figure (15) ..

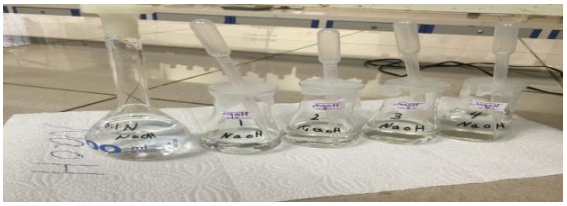


Fig. 12: Preparation of NaOH

3.4.2.Preparation of metals (Zn^{+2} & Co^{+2}):-

Taking $(CH3COOH)_2$ $Zn*2H_2O$ to preparation Zinc metal

Mwt.=219.49 g/mole , A.wt $(Zn)^{+2}$=65.41 , $ppm = \frac{\text{mg solute}}{\text{liquid solute}}$, ppm=1000 ,V=500 ml

X=1.67 gm of Zn , appear in figure (13) below:-

Fig. 13: Preparation of Zn metal

A) Taking $CoCl_2.6H_2O$ to preparation Cobalt metal

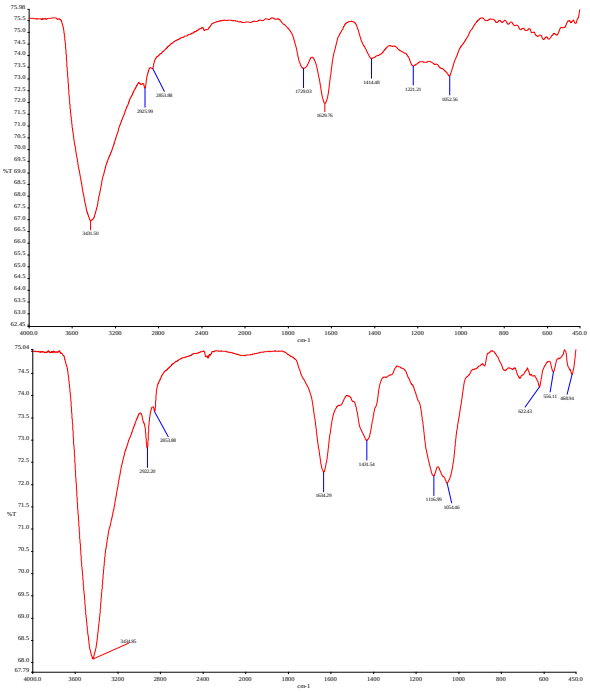
Mwt.= 237.93 g/mole , A.wt $(Co)^{+2}$= 58.93 , $ppm = \frac{\text{mg solute}}{\text{liquid solute}}$, ppm=1000 ,V=500 ml

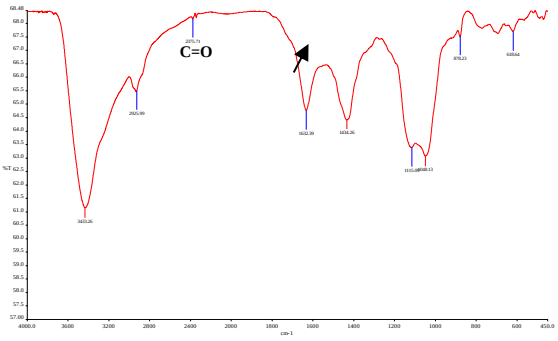
X=2.01 gm of Co , appear in figure (14) below:-

Fig. 14: Preparation of Co Metal

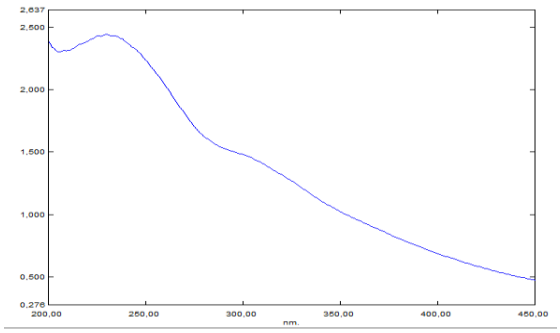
4. Results and Discussion

4.1.Instrumental & Device Analysis Fourier Transforms Infrared spectroscopy GO+ SILAN+ ALDEHYDE Images

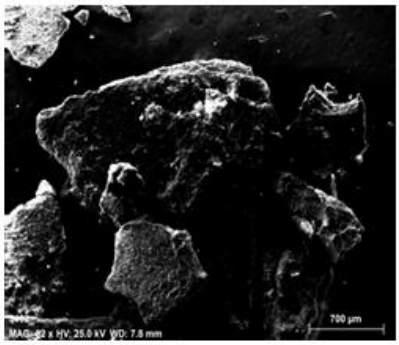


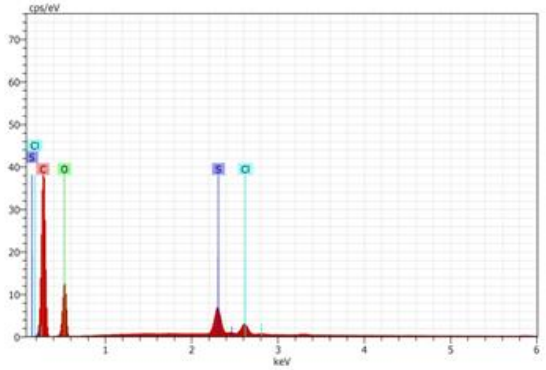


UV-VIS Analysis for Graphene Oxide



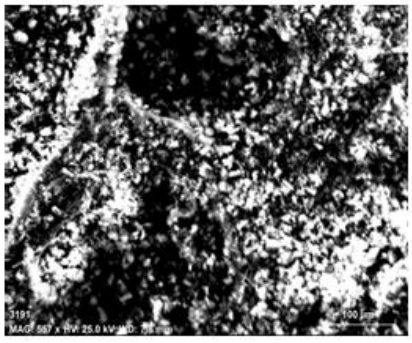
Energy-Dispersive X-ray spectroscopy (EDX) analysis EDX Analysis For Graphene oxide

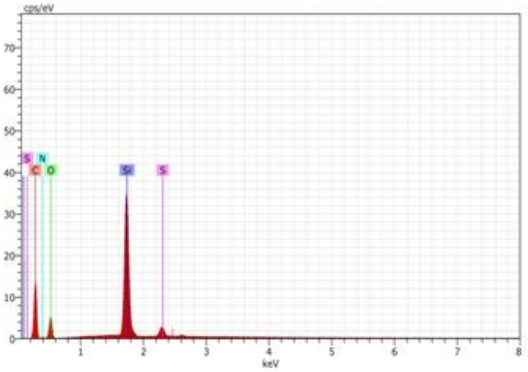




Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [wt.%]
Carbon	K-series	63.24	63.24	70.35	7.1
Oxygen	K-series	34.55	34.55	28.68	4.1
Sulfur	K-series	1.68	1.68	0.70	0.1
Chlorine	K-series	0.73	0.73	0.28	0.1
----------		Total:			
		100.00	100.00		
		100.00			

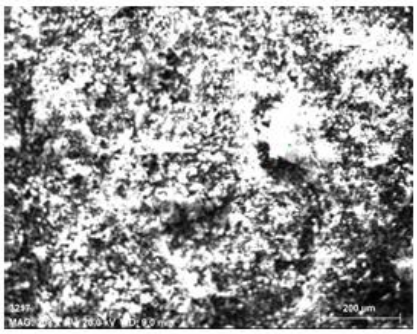
EDX Analysis For Graphene oxide with Silanization

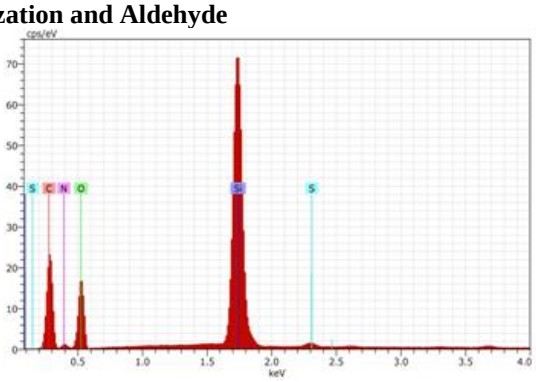




Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [wt.%]
Carbon	K-series	48.35	47.21	57.03	5.6
Oxygen	K-series	32.88	32.11	29.12	3.9
Silicon	K-series	14.66	14.31	7.39	0.7
Nitrogen	K-series	6.27	6.12	6.34	1.1
Sulfur	K-series	0.26	0.26	0.12	0.0
----------		Total:			
		102.42	100.00		
		100.00			

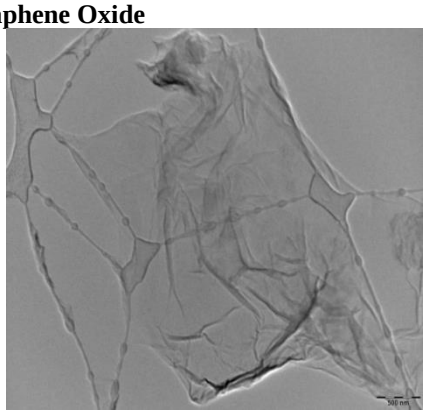
EDX Analysis For Graphene oxide with Silanization and Aldehyde



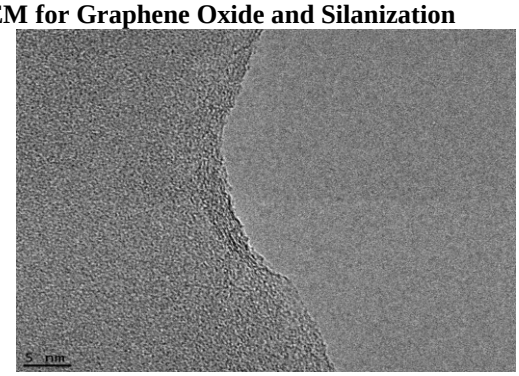


Spectrum: Element	Acquisition Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	[wt.%]	Error
Carbon	K-series	28.53	55.60	66.96	3.5	
Oxygen	K-series	10.78	21.02	19.00	1.4	
Silicon	K-series	9.02	17.59	9.06	0.4	
Nitrogen	K-series	2.09	4.06	4.20	0.5	
Sulfur	K-series	0.89	1.73	0.78	0.1	
		Total:				
		51.31	100.00			
		100.00				

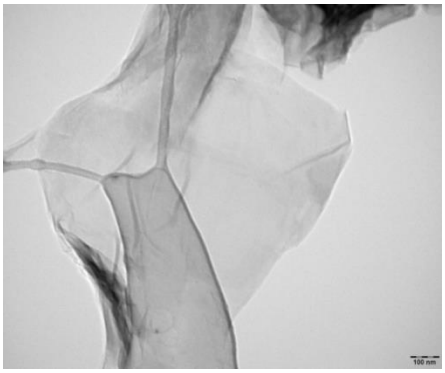
Transmission electron icroscopy (TEM) Analysis for Graphene Oxide



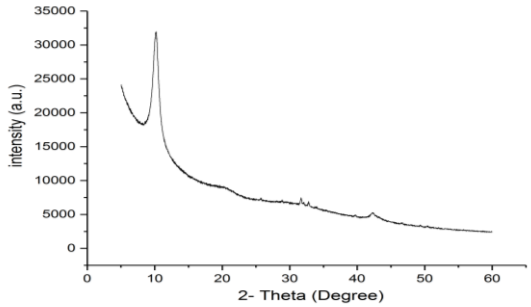
TEM for Graphene Oxide and Silanization

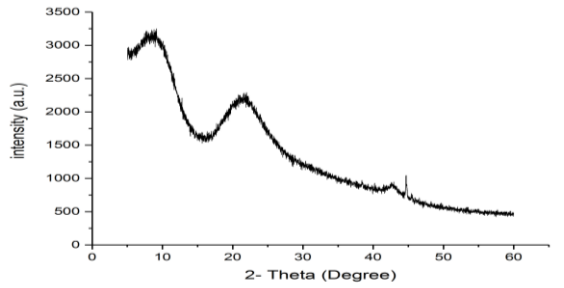


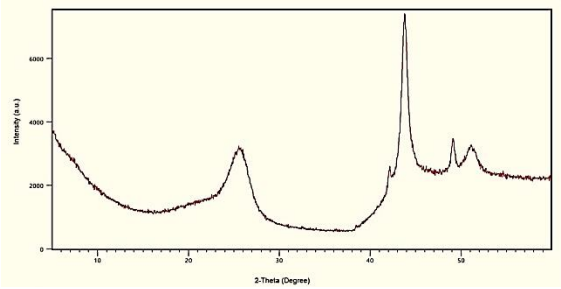
TEM for Graphene Oxide And Silanization With Aldehydes



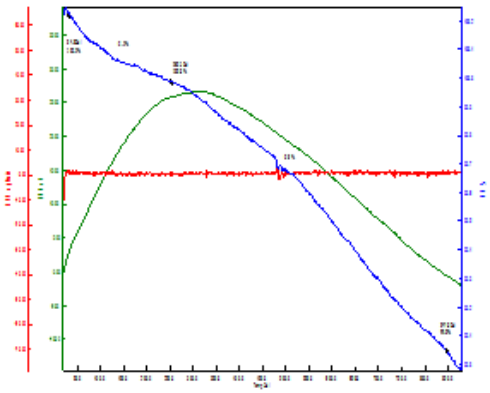
X-Ray Diffraction (XRD) Analysis For Go+SILAN+ALDEHYDE

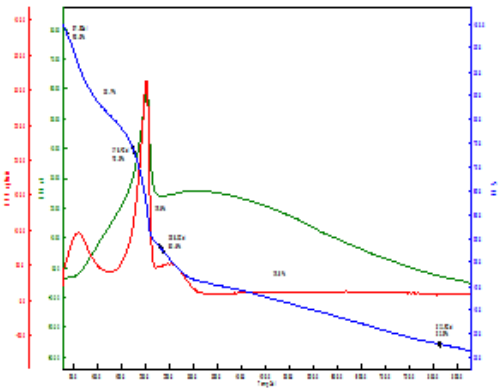


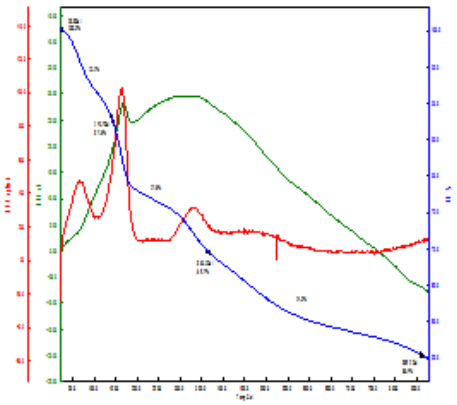


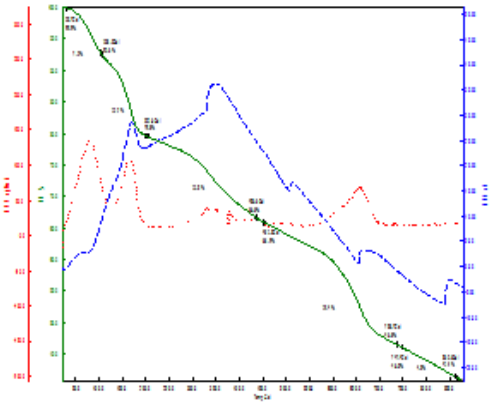


Thermogravometric TGA Analysis









BATCH EXPERIMENTS (ADSORPTION ISOTHERM)

Adsorption is the adhesion of atoms, ions, or molecules from a gas, liquid, or dissolved solid to a surface. Adsorption is a surface-based totally method at the same time as absorption consists of the enter volume of material, Adsorption is the manner which heavy metals are adsorbed on the Graphene oxide Schiff base surface, and the equilibrium is mounted when the concentrations of heavy metal adsorbed and in water grow to be steady, at equilibrium the relationship between amounts of heavy metal ions adsorbed and in water is referred to as an adsorption isotherm, the adsorption of Zn(II) and Co(II) particles on GO-Schiff base material surface from aqueous solution turned into studied in batch system [10].

After preparing Graphene oxide Schiff base material and after characterized it with several instrumental analysis such as FT-IR, EDX, XRD, Uv-visible, TGA, TEM, SEM, then we will make several reactions to investigate the maximum adsorption capacity with metal (Zn and Cobalt) ions, different pH, different stirring time, different temperature, different concentrations of metals, these four reactions is

A) First reaction is, additional Graphene oxide Schiff base material with metals at different pH values, in this step we prepared metals of Zn(II) that prepared from $(CH_3COOH)_2Zn.2H_2O$ and Co(II) from $CoCl_2.6H_2O$, taking (5) small beakers each one contain (25 mg) of GO base Schiff with (25mg/l) of both metals, these 5 small beakers putting on stirring instrument at different pH at (3, 5, 7, 9, 11) in ($25C^o$), then regulate pH with

HCL in acidic medium and NaOH in basic medium of each beaker at pH instrument, then making filtration of all beakers, after that the filtered characterized by ICP analysis instrument this process as shown in figure (15) Below:-

Fig.15: GO-Schiff base with metals (Zn & Co) in different pH values; 25 ^{o}C

B) Second reaction, is GO Schiff base material with different stirring time, by preparing also (5) small beakers, each one contain (25 mg) Graphene Schiff base material react with (25 mg/L) of both (Zn &Co) metals, putting it on stirring instrument at different stirring time (10, 30, 50, 70, 120 min.) respectively, with regulating pH (7) for both Zn(II) & Co(II) metals, then making filtrations for all beakers, the filtered is characterized by ICP instrumental analysis, this process as shown in figure (16). Below.

Fig.16: GO-Schiff base material with metals (Zn & Co) in different stirring time

C)Third reaction is Graphene oxide Schiff base material react with metals (Zn & Co) at different temperature in (25, 30, 40, 50, 60 ^{o}C), also we prepared (5) small beakers each one contain (25 mg) of GO Schiff base material with (25 mg/L) of metals, then regulating pH (7) for both Zn(II) and Co(II) metals, putting each beaker on stirring instrument, with oil bath for (1 h.) to stabling of temperatures, then making filtrations for all beakers, the filtered characterized by ICP instrumental analysis, this process as shown in figure (17) Below.

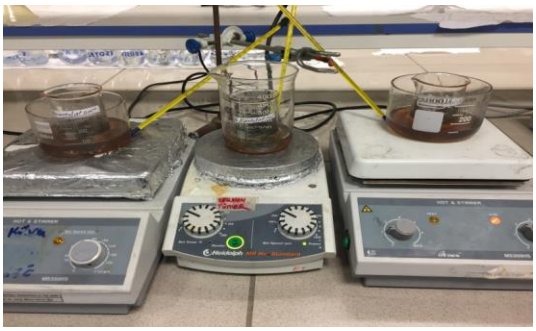


Fig.17: GO-Schiff base material with metals (Zn & Co) at different temperature

D) Fourth reaction step is preparing stock solution is (1000 mg/L) of Zn (II) that prepared from $(CH_3COOH)_2$ $Zn.2H_2O$ and Co (II) from $CoCl_2.6H_2O$. The metals Zn (II) and Co (II) in batch system is adsorption studies for adsorptive of metal ions (Zn and Co) on GO-Schiff base surface, additional of Graphene oxide Schiff base material with different concentrations of metals that starting from (25 mg/L to 1000 mg/L), preparing (10 small beakers) each one contain (25 mg) of Graphene Schiff base material with different concentrations of metals on stirring instrument and regulating pH (7) for both Zn(II) and Co(II) metals by HCl in acidic medium and NaOH at basic medium, at (60^{o}C) then making filtrations for all beakers, the filtered substance characterized by ICP analysis instrument, this process as shown in figure (18) Below.

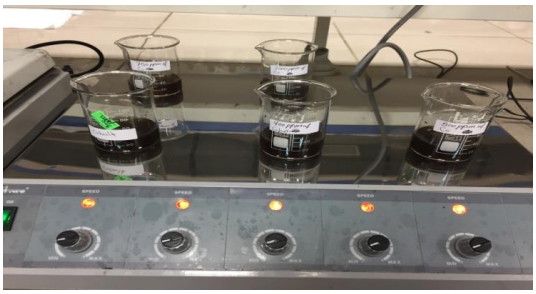


Fig.18: GO-Schiff base material with different concentrations of metals (Zn & Co)

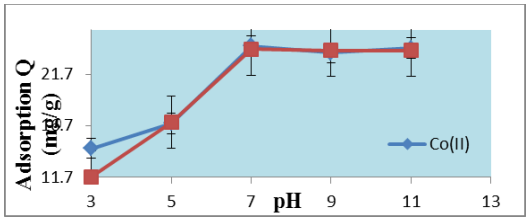
These four reactions steps that carried out to investigate the effect of different laboratories conditions, such as the effect of pH and the effect of adsorbent dosage of metals (different concentrations of metals), with contact time (different stirring time), and temperature (different temperature) of metals (Zn &Co) on GO-Schiff base material.

That can be measured by ICP analysis s a type of mass spectrometry which detecting metals and several non-metals at concentrations, that ionizing the sample with inductively coupled plasma and then using a mass spectrometer to separate and quantify those ions, the Maximum absorption capacity Q (mg/g) were determined by the following equation (1) below (Najafi,F., 2015).

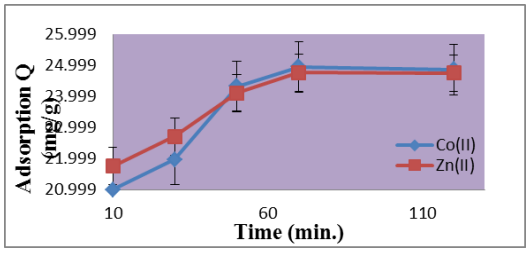
Q = [(Co-Ce). V] / m

Where Co and Ce (mg/L) are the liquid-phase concentrations of metals before and after adsorption respectively. V is the volume of the solution (L) and m is the mass of GO-Schiff base material used (g), this Experimental work occurs, as explained below:-

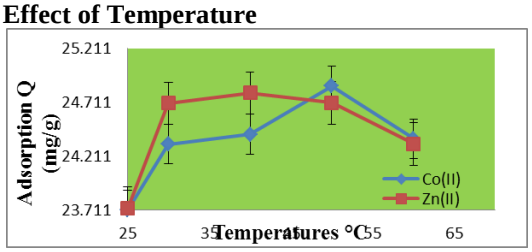
A) Effect of PH



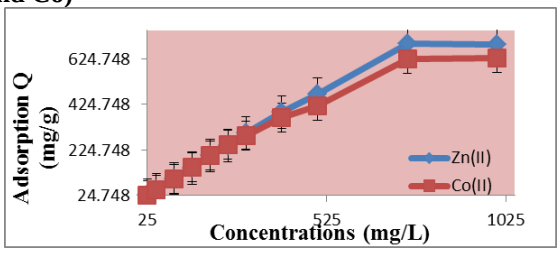
B) Effect of Contact Time



C) Effect of Temperature



C) Effect of Different concentration of metals (Zn and Co)



Conclusion

In this study, Graphene oxide considered one of methods that we can use it for treatment of waste water and decrease of pollution of waste water that absorption of heavy metals ion from water by a new sorbent material (GO) having functionalized for solid phase extraction (SPE) and discovered its compliance based on the removal of some heavy metal ions. Were included by aqueous samples under the various laboratory conditions such as pH, concentration, temperature, and time effect on their adsorption capacity and reusability. This method considered one of newest methods for remediation of pollution in environment that come from industrial wastes that cause harm effect on human health and other animals with plants.

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امتصاص وادمصاص معالجة الملوثات البيئية لإزالة أيونات المعادن الثقيلة من مياه الصرف الصحي بأستخدام النظام المائي

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الجامعة اللبنانية الفرنسية ، اربيل ، العراق

الملخص

الجرافيت هو مادة متكونة من أشكال الكربون مع الماس، والكربون غير المتبلور ، يعتبر الجرافيت إحدى الطرق التي يمكننا استخدامها لامتصاص أيون المعادن الثقيلة من الماء، بواسطة مادة ماصة جديدة تعمل على استخراج المعادن الصلبة (SPE) (الزنك والكوبالت) من المحاليل المائية. أولاً، استخدمنا مادة الجرافيت من خلال عملية كيميائية لتحويلها إلى الجرافين، وقد تم استخدام أكسيد الجرافين في الأكسدة عن طريق عملية همر باستخدام H_2SO_4 و $KMnO_4$ التي تعتبر مواد مؤكسدة جيدة. أكسيد الجرافين هو مادة مركبة مصنعة من الجرافيت. يتم أيضًا تسمية الأغشية السميكة للميكرومتر من ورق أكسيد الجرافين بأغشية أكسيد الجرافيت، وثانيًا قمنا بتحسينها باستخدام 3− (تريميثوكسيسيليل) بروبيلامين للحصول على منتج (GO-Si) ، ثالثًا ، تفاعل هذا المنتج مع (ألدهيد) (3،5-Di-tert-butyle-2-hydroxy-benzenaldehyde) للحصول على (Go-Si-Ald) التي تشكل مادة الجرافين Schiff الأساسية، التي استخدمناها في العديد من العمليات تحت عوامل مختبرات مختلفة مثل (الأس الهيدروجيني، تركيزات مختلفة من المعادن / الزنك والكوبالت، اختلاف زمن التفاعل، مع درجات حرارة مختلفة)، ثم بعد ذلك فحص امتزاز المعادن (Zn and Co) بنجاح، وهي إحدى طرق التطوير لمعالجة النفايات ومياه الشرب لإعادة استخدامها. هذه الطريقة مفيدة لحل المشكلة البيئية الضارة بالصحة. كل هذه الأعمال التي قام بها العديد من التحليلات الآلية مثل طيف الانبعاث الذري و جهاز طيف الاشعة تحت الحمراء وجهاز طيف الاشعة السينية وطيف الاشعة فوق البنفسجية و جهاز تحليل الوزن الحراري و جهاز الكتروني نافذ ومجهر الكتروني ماسح و جهاز حيود الاشعة السينية .