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Preparation and characterization of some Heterocyclic compounds linked to graphene oxide and study of their effect to remove Cd (II) and Pb (II) from industrial wastewater

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Abstract

In this study, several heterocyclic compounds were prepared and combined with graphene oxide to produce composite materials with high adsorption efficiency, exceeding that of each individual component. This is attributed to the unique surface properties of graphene oxide, particularly the presence of functional groups such as hydroxyl, carboxyl, and epoxy groups, which facilitate bonding with the prepared compounds. Furthermore, the heterocyclic compounds possess donor atoms such as nitrogen and sulfur, enhancing their ability to form strong coordinate bonds with heavy metal ions. The efficiency of the prepared composite materials in removing cadmium (II) (Cd^{2+}) and palladium (II) (Pd^{2+}) ions from contaminated aqueous solutions simulating industrial wastewater, was investigated. Adsorption experiments were conducted under various time and pH conditions, with mass and temperature constants, and the concentrations of residual ions were determined using atomic absorption spectroscopy. Advanced analytical techniques, including FTIR, XRD, AFM, and FESEM, were used to characterize the composite materials to determine their chemical composition, surface properties, and morphology. The results demonstrated a significant improvement in the adsorption efficiency of the composite materials compared to the individual materials, confirming their potential as effective materials for treating water contaminated with heavy metals.

Keywords : Heterocyclic compounds ، graphene oxide, heavy metals.

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Introduction

Over the centuries, thiazole has been a heterocyclic ring of significant synthetic importance in chemistry. The thiazole ring consists of sulfur and nitrogen in a bond that allows pi electrons (π) to transfer from one bond to another, giving it the properties of an aromatic ring. Due to these properties, the ring has many sites where donor-acceptor interactions, oxidation reactions, etc. may occur. (*Leoni et al., 2014)*

Graphene oxide has attracted the attention of researchers since its discovery(*Agharkar et al., 2014*) . Graphene is widely used in many fields such as biological engineering, and lightweight/durable composite materials after its reduction to graphene, which is of great importance in photovoltaics and energy storage. It is an excellent electrical conductor with high mechanical strength and relatively high thermal conductivity, and is characterized by its high gas permeability and optical transparency*(Randviir et al, 2014*), (*Stankovich et al., p. 1560, 2007*), (*Ramesha et al., 2011*) . One of the methods used in its manufacture is mechanical exfoliation.*(Stephenson & Duff, p. 783, 1996*), (*Cheng et al., p. 40, 2024*), bottom-up synthesis(*Novoselov et al., 2004*), Growth of graphene layers by chemical vapor deposition (CVD) on substrates(*Moon et al., 2023*), (*Farahani et al., 2024*), Freeing carbon nanotubes from their compressed state(*Mishra et al., 2023*), (*Ebrahimi Naghani et al., 2023*), In addition to the reduction of graphene oxide(*Liu et al., 2015*),(*Dey et al., 2025*). Graphene oxide(GO) is the oxidized form of graphene(*Das et al., 2022*), which is completed by the addition of oxygen functional groups(*Peng et al., 2019*),(*Wei et al., 2016*). The oxidation of graphite to graphene oxide sheets that carry oxygen-based functional groups, especially epoxy and hydroxyl groups at the base level and carboxyl groups at the edges.(*Peng et al., 2016*). The oxygen functional groups on graphene significantly contribute to its hydrophilicity and negative charge density(*Wu et al., 2017*) .

Heavy metal pollutants in water are among the most hazardous environmental pollutants, leading to the contamination of water resources. Common methods for their removal include adsorption,

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precipitation, ion exchange, filtration, oxidation processes, and other techniques., Among these methods, adsorption using graphene oxide is one of the most efficient and widely used in industrial applications due to its relatively low cost and ease of operation (*Salih, 2003*). Graphene oxide is considered the most effective material for the adsorption of various heavy metal ions compared to rice husks, orange peels, red clay, sugarcane ash, sawdust, conductive polymers, activated carbon, and carbon nanotubes. This is due to its surface area, high surface charge density, and the ease of its synthesis from abundant natural graphite using chemical oxidation and exfoliation methods. The study focused on adsorption affinity and influencing factors, and aims to develop graphene-based adsorbents for the absorption of several heavy metal pollutants, leading to the commercial application of graphene and its compounds in the future.

2- Experimental

2-1 preparation of 2 – amino –6- nitro benzothiazole(R1)

At first, the compound(2 – amino –6- nitro benzothiazole) was prepared by adding a mixture of (1.2) ml of liquid bromine dissolved in (15) ml of glacial acetic acid drop by drop to a mixture of (3.450) grams (0.025) mol of para-nitroaniline and (3.8) grams (0.05) mol of ammonium thiocyanate dissolved in (70) ml of glacial acetic acid. The mixture was cooled to (10°C) and stirred continuously. After the mixture of bromine and glacial acetic acid was completed, the mixture was left to settle for 15 minutes, then it was gradually diluted with cold water. After that, a concentrated, cold sodium hydroxide solution was gradually added to the reacted mixture to make it a base, where the thiazole derivative, which was suspended in water, precipitated as a solid with a light yellow color. The precipitate was washed with distilled water several times and recrystallized with pure ethanol to obtain a pure precipitate, which was left to air dry. (*Salih, 2003*).

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2-2 **preparation of 2 – amino –6- nitro benzothiazole -** schiff base [R1(a, c,)]

2-amino-6-nitrobenzothiazole (0.010 mol) with aldehyde (0.010 mol) (4-methylbenzaldehyde, 4-hydroxybenzaldehyde) was prepared by heated until it melted in beaker for 10 min. The reaction was followed by TLC . The precipitated yellow Schiff bases were filtered, washed, and dried**.**

2-3 preparation of 4-(6- nitro-2- benzothiozolylazo)resorcinol R2

(1.950) grams (0.010) mol of the derivative prepared in paragraph (2-1) were dissolved in a mixture consisting of (5) milliliters of concentrated hydrochloric acid, (20) milliliters of glacial acetic acid, and (25) milliliters of distilled water. The mixture was cooled to (0) °C. Then, the sodium nitrite solution prepared by dissolving (0.69) grams (0.010) mol of sodium nitrite in (5) milliliters of distilled water was added drop by drop to the mixture while stirring, noting that the temperature did not rise above zero degrees Celsius. After that, the solution was left for (5) minutes. Then, this diazonium salt solution was added drop by drop to the solution consisting of (1.100) grams (0.010) mol of resorcinol and (1.000) grams of sodium hydroxide dissolved in (50) milliliters of distilled water prepared below zero degrees Celsius. At that time, it was observed that the mixture turned orange and two hours after the addition was completed, a quantity of distilled water was added and the medium was adjusted to pH = 6. Then the solution was left for (24) hours, during which the orange-colored compound precipitated. It was filtered and washed several times with distilled water, then recrystallized using ethanol and its purity was confirmed by thin layer chromatography (TLC) (*Salih, 2003*).

2-4 **preparation of Gaphene Oxide R3**

Place 23 ml of concentrated sulfuric acid in an ice bath to homogeneity, then add (0.005) mol, 0.5 g of sodium nitrate gradually for 15 minutes while stirring at a temperature of 0°C. 0.2 g of graphite dust was added, then gradually added over 10 minutes with stirring. 0.021 mol of potassium permanganate was then added over 15 minutes with stirring. The mixture was then left in an ice bath for an additional 5 minutes, then

stirred at room temperature (25°C) for 60 minutes. 46 ml of distilled water was added dropwise over 20 minutes. The temperature was then raised to 98°C for 20 minutes. 140 ml of warm distilled water was added, and the mixture was left to stir for 10 minutes. 9 ml of hydrogen peroxide (30%) was then added, and stirring continued for 30 minutes. The mixture was cooled, filtered using special filter paper, washed with distilled water (3.5 ml), and dried at 60-70°C until the weight stabilized (4.5 hours) (*Agharkar et al., 2014*).

2-5 **preparation of Gaphene Oxide –2 – amino –6- nitro benzothiazole** - schiff base[GO- **R1(a) (c)]**

Add (0.1) g 0.0005 mol of 2-amino-6-nitrobenzothiazole-schiff base to 0.1 g of graphene oxide in a suitable flask, then heat in a sand bath at the melting point for about ten minutes, stirring until noting the change in color and nature of the molten substance and remove from heat and collect the product. (*Hussain et al., 2017*)

2-6 **preparation of Gaphene Oxide-4-(6- nitro-2-enzothiozolylazo)resorcinol(GO- R2)**

Dissolve 0.1 g of the prepared reduced graphene oxide in 10 mL of ethylene glycol in an ultrasonic bath for 2 hours until a clear nanoparticle solution is obtained. Then, dissolve 0.01 g of 4-(6-Nitro-2-benzothiazolyl azo)-resorcinol in 10 mL of ethylene glycol in an ultrasonic bath for 2 hours at 50 Hz for disintegration to obtain clear solutions. Then, take 1 mL of the reduced graphene oxide solution by pipetting and place it on a glass microscope slide using a spin coater at a speed of 6000. rpm rotations per minute to obtain a thin layer and expose it to a temperature of 60-70°C for 10-15 minutes. The sample was then removed and placed at room temperature until it reached a temperature close to that of a normal glass microscope slide. The remaining graphene oxide was then spread. After the graphene oxide solution was finished, 4-(6-Nitro-2-benzothiazolyl azo)-resorcinol was spread on the same slide above graphene oxide. The color of the slide was black.

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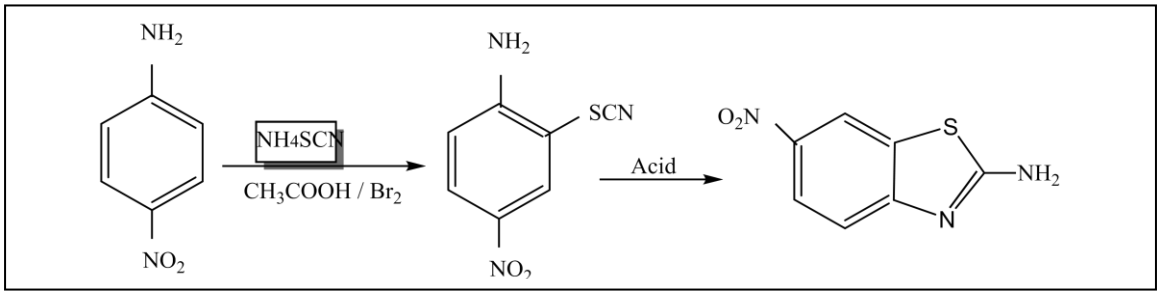
3-Results and Discussion

The compound 2-amino-6-nitro-benzothiazole was prepared mechanically, in the first step of preparation, the (SCN-) group was introduced by the thiocyanogen method at the ortho position on the aromatic amine substituted with the nitro group at the para position in an acidic medium using bromine liquid dissolved in glacial acetic acid. This was followed by the second step, which includes a secondary reaction between the amine group and the thiocyanate group, where the ring closure occurs in a cold acidic medium to form the thiazole derivative (scheme 1). After that, it was precipitated in a basic environment, as the melting point of this derivative was equal to 200-245 °C, which is different from the melting point of nitroaniline, and the product percentage was 79% and the powder was yellow in color. The infrared spectrum showed (ν cm-1): 3445 (NH str), 3020 (CH-Ar), 1640 (C=N), 1449 (C=C), 1279 (C–NO2), 696 (C– S–C).



Scheme 1 preparation (R1)

The 2 – amino –6- nitro benzothiazole - schiff base [R1(a, c)] scheme 2, product percentage was 74% and 78%, respectively, and the melting point was 265-268 for R1(a), IR 1630 (C=N), 1440 (C=C) and 250-252 for R1(c) , IR 1630 (C=N), 1445 (C=C) ,3330 (-OH).

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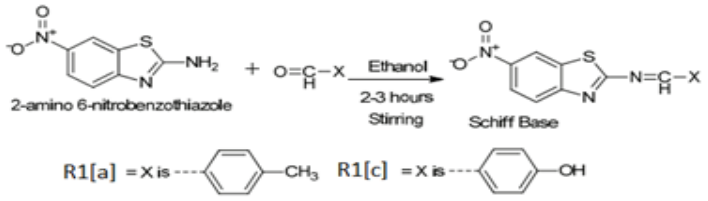
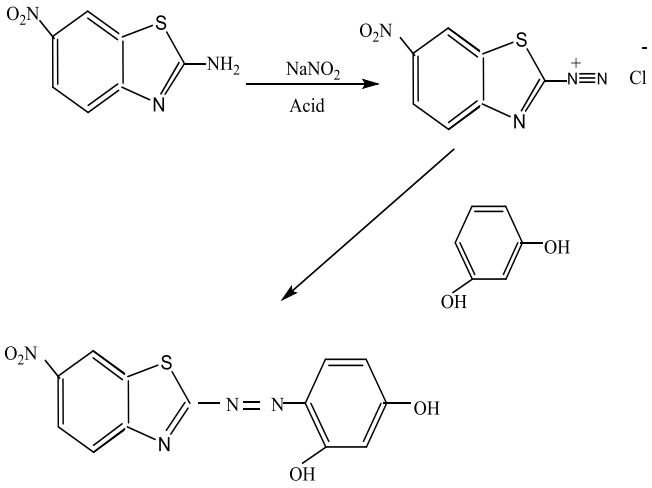


Scheme 2 preparation R1(a,c)

The compound 4-(6- nitro-2- benzothiozolylazo)resorcinol was prepared by diazolation of the thiazole derivative, by adding sodium nitrite solution .scheme 3, The infrared spectrum showed in fig (1)



scheme 3 **preparation (R2)**

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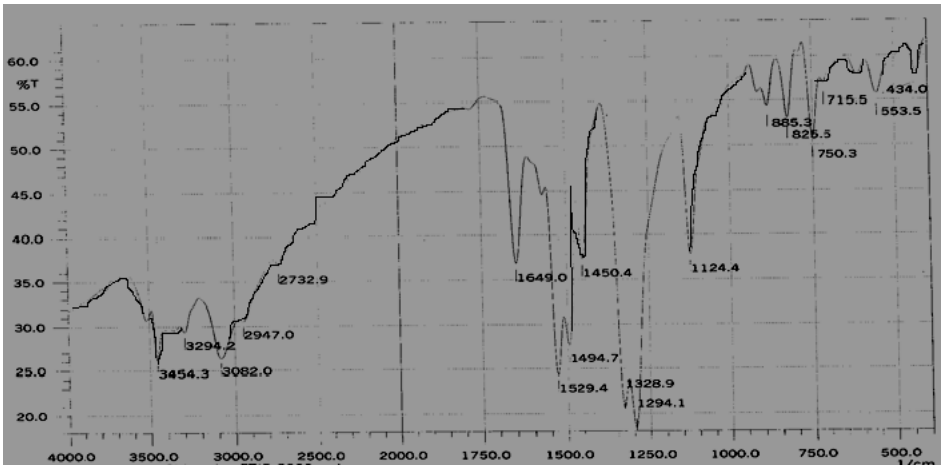

Fig 1 IR for (R2)

The infrared spectrum of graphene oxide showed a broad band associated with the stretching of the carboxylic and alcoholic (OH) groups, as well as the stretching band of the carboxylic and ketonic (CO) bonds (overlapping due to the repetition of these groups at the ends), the stretching of the (C-C) bond, and another band associated with the symmetric and asymmetric stretching of the epoxy rings, with the appearance of the unreacted (C=C) bond stretching band on the graphene oxide molecule. Fig 2, X-ray diffraction showed in Fig 3

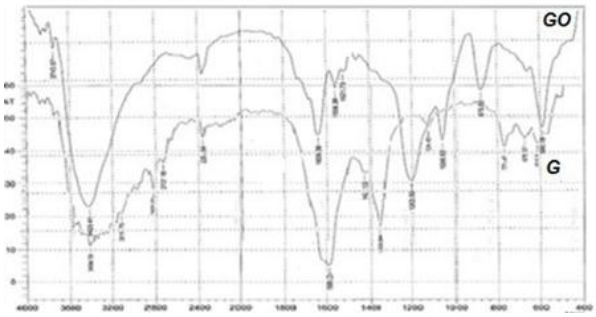

Fig 2 IR for GO

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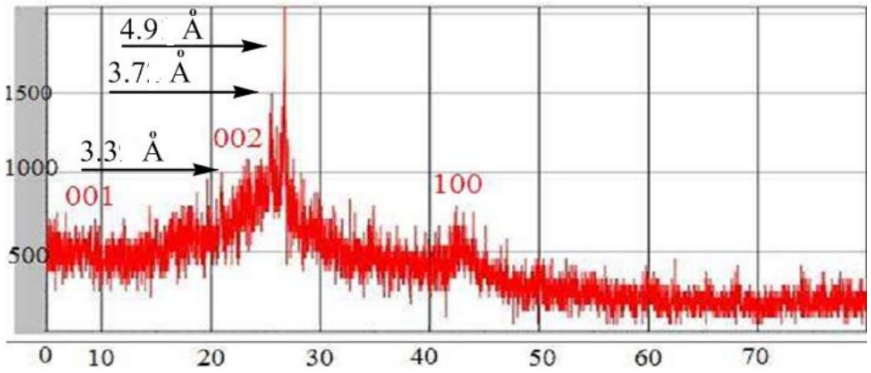

Fig 3 x-ray for GO

XRD spectrum of the compound (GO-R1(a)) as shown in Fig (4), appeared at 2θ = 21.9, and the average distance between the layers is given by d = 3 A0 with the number of the layer (n = 155) and the grain size (D = 62.7 nm). Atomic force microscopy (AFM) was also used to examine the morphology and thickness of the sheets of this composite. The morphology of this composite was imaged and measured using AFM in the fig(5). and FESEM images in Fig (6)

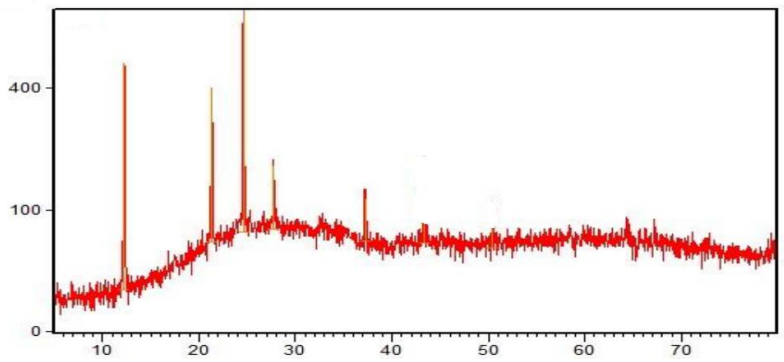

Fig 4 x-ray for GO-R1(a)

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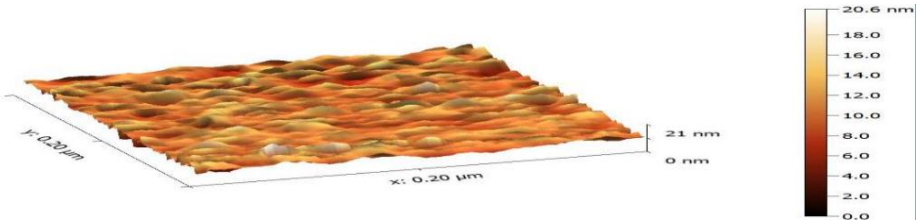

Fig (5): AFM image of GO-R1(a)

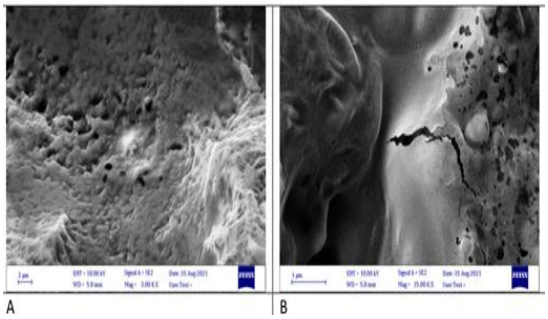

Fig.(6): FESEM images of GO-R1(a)

XRD spectrum of the compound (GO-R1(c)) as shown in Fig (7), appeared at 2θ = 15.109, and the average distance between the layers is given by d = 5 A^{o} with the number of the layer (n =74) and the grain size (D =37.17 nm). Atomic force microscopy (AFM) was also used to examine the morphology and thickness of the sheets of this composite. The morphology of this composite was imaged and measured using AFM in the fig(8). and FESEM images in Fig (9)

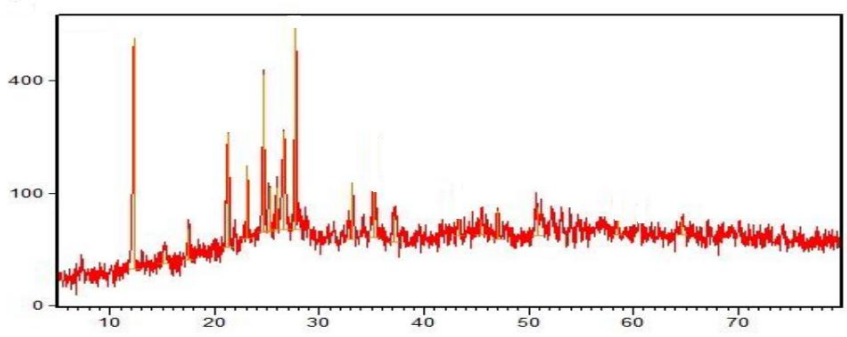

Fig 7 x-ray for GO-R1(c)

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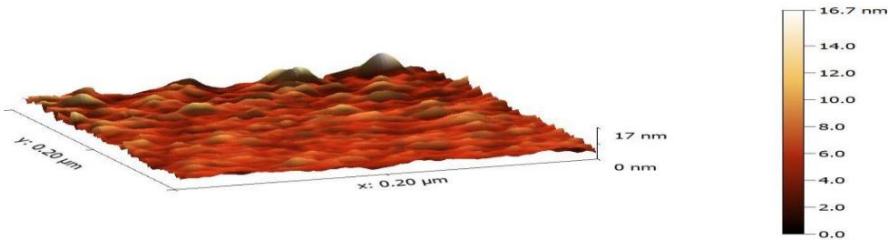

Fig (8): AFM image of GO-R1(c)

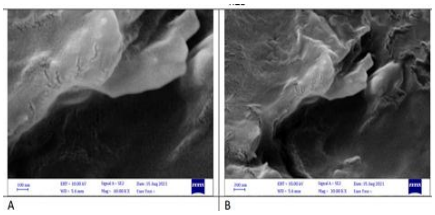

Fig.(9): FESEM images of GO-R1(c)

Also XRD spectrum of the compound (GO-R2) as shown in Fig (10), appeared at 2θ = 12.10, and the average distance between the layers is given by d = 10 Aº with the number of the layer (n =4) and the grain size (D =47.9 nm). Atomic force microscopy (AFM) was also used to examine the morphology and thickness of the sheets of this composite. The morphology of this composite was imaged and measured using AFM in the fig(11). and FESEM images in Fig (12)

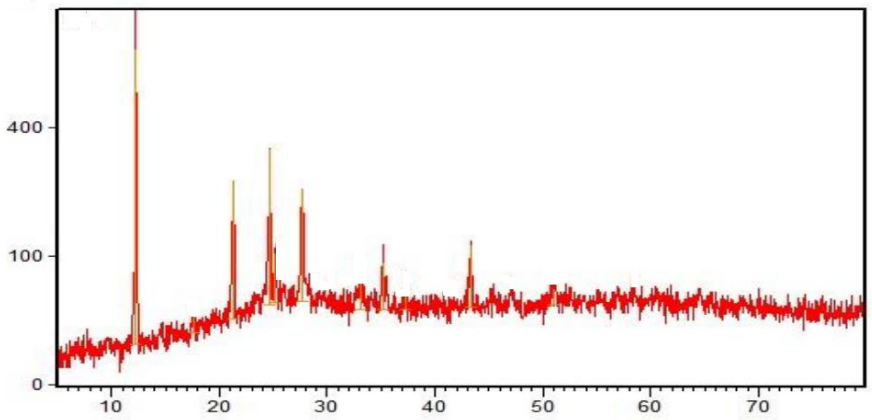

Fig 10 x-ray for GO-R2

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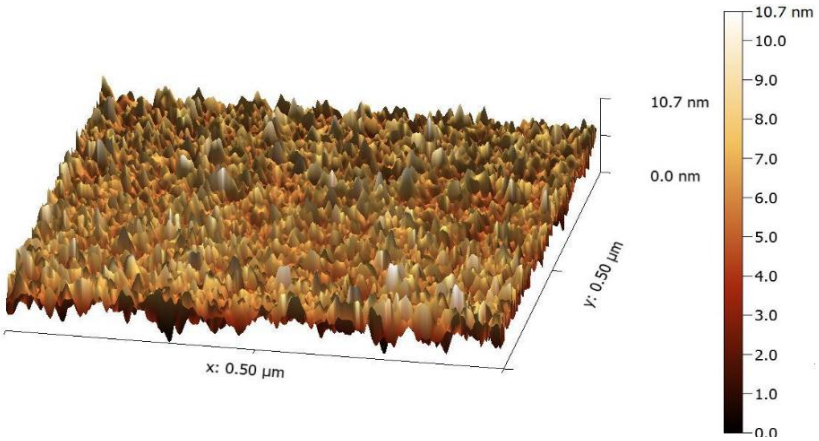

Fig (11): AFM image of GO-R2

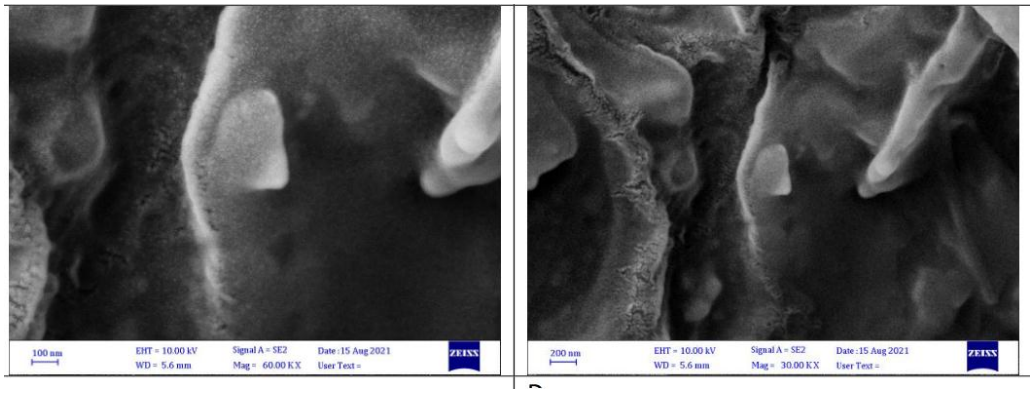

Fig.(12): FESEM images of GO-R2

4- Adsorption study results

Stock solutions Pb (II) and Cd (II) of ions (1000 ppm) were prepared, 10 mg [GO- R1(a) (c)], GO-R2 in 10 ml of ion solutions (100ppm) were placed in 10 ml volumetric flask and Shaked continuously for20, 40,60,80 minutes at 25 ºC, pH of 2,4,6,8. Table 1,2,3,4,5,6. Fig 13,14,15,16,17,18. The metal ions concentrations in the filtrate were measured by atomic absorption spectroscopy .The removal efficiency R% was calculated by using the equation .

$R\% = \frac{C_i - C_e}{C_i} \times 100$ (C_i and C_e are the initial and equilibrium con of ions)

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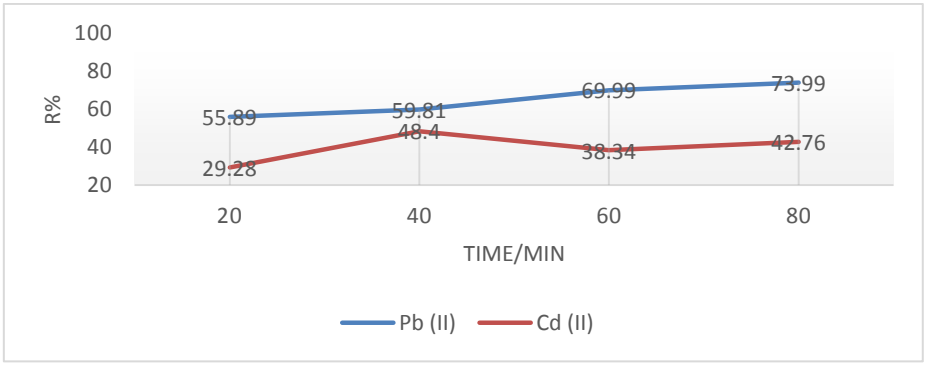
Table (1) result of contact time on adsorption of Pb (II) and Cd (II) **ions for(**GO- R1(a)**)**

Ions	C_i(mg/L)	Time/min	C_e(mg/L)	$C_i - C_e / C_i * 100$
Pb (II)	140.O2	20	99.01	29.28
		40	72.33	48.40
		60	86.44	38.34
		80	80.14	42.76
Cd (II)	220.02	20	97.03	55.89
		40	88.42	59.81
		60	66.01	69.99
		80	57.22	73.99



Fig(13) Effect of the Time on metal ions removal by (GO- R1(a))

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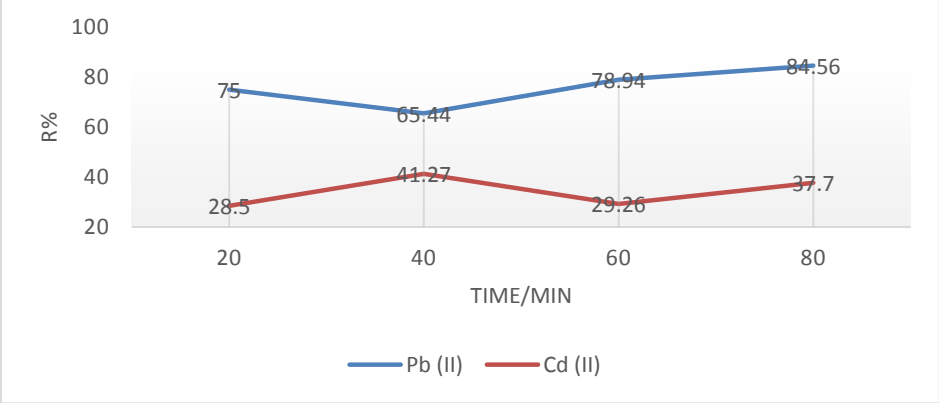
Table (2) result of contact time on adsorption of Pb (II) and Cd (II) **ions for(**GO- R1(c)**)**

Ions	C_i(mg/L)	Time/min	C_e(mg/L)	$C_i - C_e / C_i * 100$
Pb (II)	140.O2	20	100.11	28.50
		40	82.22	41.27
		60	99.04	29.26
		80	87.23	37.70
Cd (II)	220.02	20	55.00	75.00
		40	76.03	65.44
		60	46.33	78.94
		80	33.97	84.56



Fig(14) Effect of the Time on metal ions removal by (GO- R1(c))

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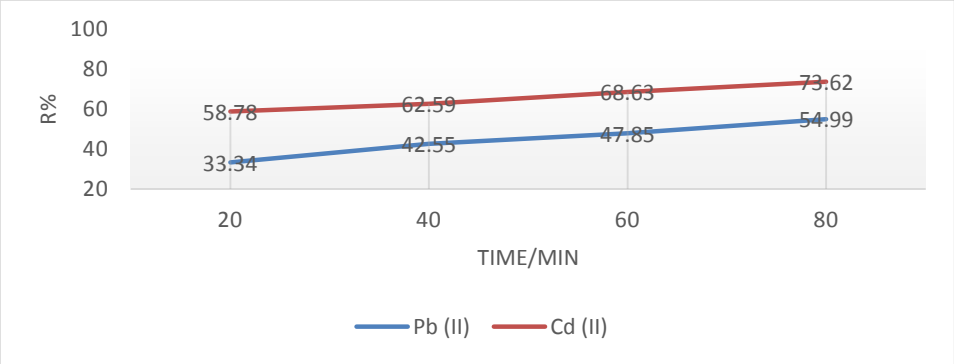
Table (3) result of contact time on adsorption of Pb (II) and Cd (II) **ions for(**GO- R2**)**

Ions	C_i(mg/L)	Time/min	C_e(mg/L)	$C_i - C_e / C_i * 100$
Pb (II)	140.O2	20	93.33	33.34
		40	80.44	42.55
		60	73.33	47.85
		80	63.01	54.99
Cd (II)	220.02	20	90.68	58.78
		40	82.30	62.59
		60	69.02	68.63
		80	58.04	73.62



Fig(15) Effect of the Time on metal ions removal by (GO- R2)

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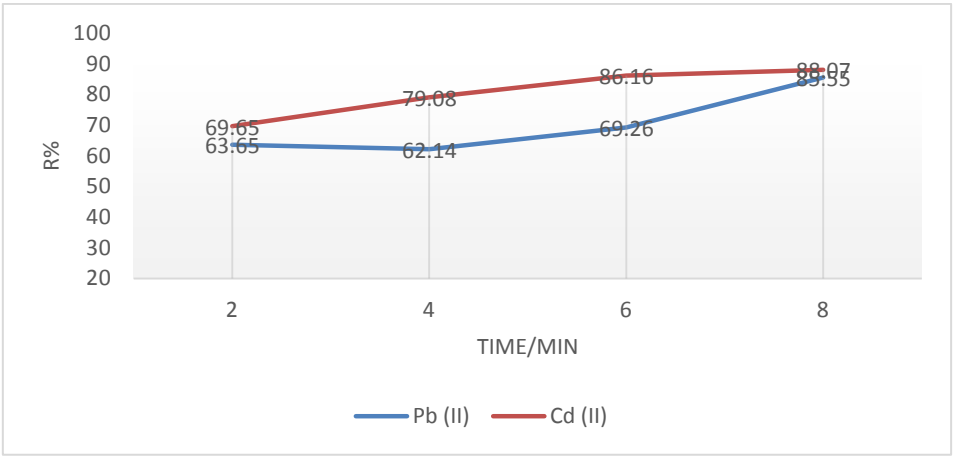
Table (4) result of PH on adsorption of Pb (II) and Cd (II) **ions for(**GO- R1(a)**)**

Ions	C_i(mg/L)	pH	C_e(mg/L)	$C_i - C_e / C_i * 100$
Pb (II)	140.O2	2	50.89	63.65
		4	53.01	62.14
		6	43.03	69.26
		8	20.23	85.55
Cd (II)	220.02	2	66.77	69.65
		4	46.01	79.08
		6	30.39	86.18
		8	26.24	88.07



Fig(16) Effect of the Time on metal ions removal by (GO- R1(a))

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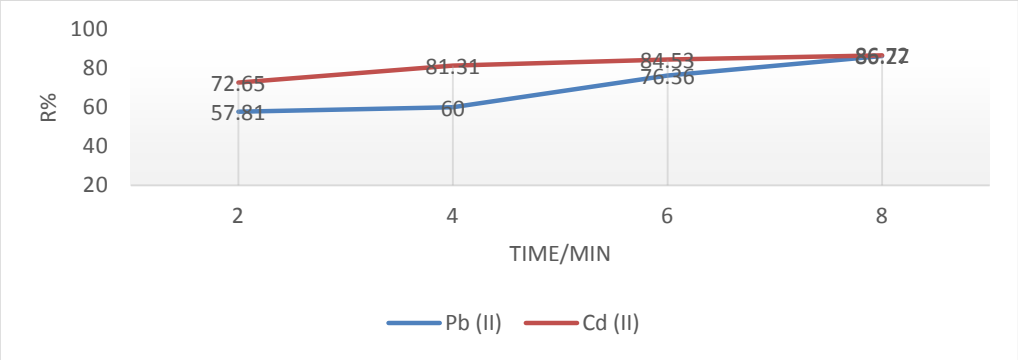
Table (5) result of pH on adsorption of Pb (II) and Cd (II) **ions for(**GO- R1(c))

Ions	C_i(mg/L)	pH	C_e(mg/L)	$C_i - C_e / C_i * 100$
Pb (II)	140.O2	2	59.07	57.81
		4	56.00	60.00
		6	33.09	76.36
		8	19.22	86.27
Cd (II)	220.02	2	60.17	72.65
		4	41.11	81.31
		6	34.03	84.53
		8	29.20	86.72



Fig(17) Effect of pH on metal ions removal by (GO- R1)

Table (6) result of pH on adsorption of Pb (II) and Cd (II) **ions for(**GO- R2**)**

Ions	C_i(mg/L)	pH	C_e(mg/L)	$C_i - C_e / C_i * 100$
Pb (II)	140.O2	2	33.49	76.08
		4	20.29	85.50
		6	19.00	86.43
		8	11.09	92.07
Cd (II)	220.02	2	41.51	81.13
		4	12.22	94.44
		6	5.22	97.59
		8	3.11	98.58

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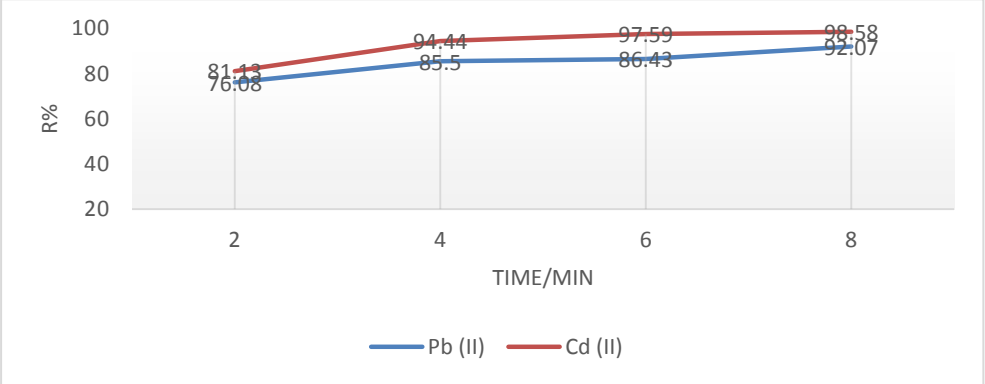

Fig(18) Effect of pH on metal ions removal by (GO- R2)

The adsorption results were as shown in Table 1 and Figure 13, for GO-R1(a)with a significant increase in adsorption at 80 minutes for Cd(II) and at 40 minutes for Pb(II). The compounds GO- R1(c), the results were as shown in Table 2,3 and Figure 14, 15 a significant increase in the removal rate over time for both ions in 80 minutes . When the pH varied, the basic medium at pH = 8 showed the highest removal rate for GO-R1(a) ,GO-R1(c), GO-R2 , shown in Table4,5,6 and Figure 16, 17,18

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تحضير وتشخيص بعض المركبات الحلقية غير المتجانسة المرتبطة بأوكسيد الكرافين ودراسة تأثيرها في إزالة أيونات الكادميوم والرصاص من مياه الصرف الصناعي

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مستخلص البحث:
في هذه الدراسة، تم تحضير عدة مركبات حلقية غير متجانسة وربطها مع أوكسيد الكرافين لإنتاج مواد ذات كفاءة امتزاز عالية. يُعزى ذلك إلى الخصائص السطحية الفريدة لوأكسيد الكرافين، ولا سيما وجود مجموعات وظيفية مثل الهيدروكسيل والكربوكسيل والإيبوكسي، التي تُسهّل الارتباط بالمركبات المُحضّرة. علاوة على ذلك، تحتوي المركبات الحلقية غير المتجانسة على ذرات مانحة مثل النيتروجين والكبريت، مما يُعزز قدرتها على تكوين روابط تناسقية قوية مع أيونات المعادن الثقيلة. تم التحقق من كفاءة المواد المركبة المُحضّرة في إزالة أيونات (II) (Cd^{2+})و (Pb^{2+}) (II) من محاليل مائية ملوثة تُمثل مياه الصرف الصناعي. أُجريت تجارب الامتزاز في ظل ظروف زمنية وpH مختلفة، مع ثبات الكتلة ودرجة الحرارة، وتم تحديد تراكيزالأيونات باستخدام مطيافية الامتصاص الذري. استُخدمت تقنيات للتشخيص، تشمل مطيافية الأشعة تحت الحمراء (FTIR)، وحيود الأشعة السينية(XRD) ، والمجهر الذري الماسح(AFM) ، والمجهر الإلكتروني الماسح ذي الانبعاث الميداني(FESEM) ، لتوصيف المواد المركبة وتحديد تركيبها الكيميائي، وخصائص سطحها، وبنيتها المورفولوجية. وأظهرت النتائج تحسنًا ملحوظًا في كفاءة امتصاص المواد المركبة ، مما يؤكد إمكانية استخدامها كمواد فعالة لمعالجة المياه الملوثة بالمعادن الثقيلة.

الكلمات المفتاحية: مركبات حلقية غير متجانسة، أوكسيد الكرافين، عناصر ثقيلة.

ملاحظة: هل البحث مستل من رسالة الماجستير او اطروحة الدكتوراه ؟ كلا