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Preparation Composites To Remove Some Pollutants From Its Aqueous Solutions

A Thesis Submitted to the

Council of the College of Science, University of Diyala in Partial Fulfillment of the Requirements for the Degree of Master of Science in Chemistry

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B.Sc. In Chemistry Science 2021 College of Science – University of Diyala

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2024 AD 1446 AH



In this study, copper oxide (CuO) was synthesized from plant extract (Capparis spinosa) using a green method, then graphene oxide (GO) was prepared using the Hummer modified method and reduced graphene oxide (rGO) was obtained by reducing it with hydrazine. The conductive polyaniline (PANI) was then synthesized. The binary nanocomposites (GO-CuO) and (rGO-CuO) were synthesized utilizing the aforementioned basic ingredients. Ultimately, the aforementioned binary composites with polyaniline (PANI) were used to prepare the ternary nanocomposites (GO-CuO-PANI), (rGO-CuO-PANI) using in situ polymerization. Nanocomposites were analyzed utilizing infrared techniques (FTIR), X-ray diffraction (X-ray), and Field emission scanning electron microscopy (FESEM). The memberans were synthesized by dissolving polyvinyl alcohol (PVA) in heated water and subsequently combining the resultant solution with the ternary nanocomposite in various weight ratios to produce the films. The electrical properties were examined using (PVA) single and (PVA) hybrid polymer membrane techniques (LCR) by incorporating a manufactured ternary nanocomposite (GO-CuO-PANI) at varying weight ratios (1, 2, 3, 4, 5%) and a nanocomposite (rGO-CuO-PANI) at weight ratios (2, 4, 6, 8, 10%) within a specified frequency range (1KHz-5KHz) at a designated temperature (25°C). The electrical conductivity measurements indicated decrease in the dielectric constant (ϵ') where the highest value was (9.322) with the ratio (8%) for (PVA/rGO-CuO-PANI) membrane, the highest value for (PVA/GO-CuO-PANI membrane with the ratio (3%) was (9.718) and the dielectric loss factor (tanδ) for hybrid membranes (PVA) showed a decrease

with increasing frequency, reaching the highest value of (1.031) with a ratio of (10%) for (PVA/rGO-CuO-PANI) membrane and the highest value of (PVA/GO-CuO-PANI) membrane with a ratio of (3%) was (1.019). Alternating electrical conductivity (σ a.c) showed an increase with increasing frequency. The alternating electrical conductivity ($\sigma_{a,c}$) rises with higher frequency. Measurements of thermal characteristics indicated that the thermal conductivity coefficient rises with an increase in the weight ratio of the reinforced nanoparticles. The ternary nanocomposite (GO-CuO-PANI) was employed to remove lead ion contamination and the using nanocomposites (GO-CuO), (rGO-CuO) to remove contamination orange-G dye from dilute aqueous solutions. A number of factors influencing the lead ion and orange-G dye removal rate in the adsorbat were examined. The time required to remove lead ions and achieve equilibrium was (20 minutes) while the best surface removal (GO-CuO) was (30minutes) and the surface (rGO-CuO) was the best removal was (10 minutes). The optimal weight for removal (0.05g) at all the surfaces nanocomposites. The best Lead ion adsorption on the prepared surface was observed at (pH=8) while the best of removal for two surfaces (GO-CuO), (rGO-CuO) was (pH=2). The findings indicated that the adsorption process conforms to pseudo-second-order kinetics for two surfaces (GO-CuO), (rGO-CuO). The various thermodynamic functions of adsorption were studied as well the process is obviously exothermic for the lead ion and surface (GO-CuO), while the surface (rGO-CuO) was endothermic (ΔG) has exhibits negative values for binary the indicating that adsorption nanocomposites, processes transpire spontaneously. The molecules have become less constrained, according to positive (ΔS) values. Because the study findings showed linear associations with strong correlation coefficients and were generally compatible with each other, the isotherm of the dye orange-G for the surface (rGO-CuO) exhibited greater conformity with the Freundlich isotherm, and the surface (GO-CuO) was exhibited greater conformity with the Temken isotherm.

Chapter one Chapter one

Introduction & Literature

survey

1.1 Introduction

An increasing number of enterprises are releasing a wide variety of contaminants into the environment through water as a result of the ongoing trend toward industrialization. The petroleum, paint, textile, paper and pulp, food and drink, plastic tannery, and a host of other related sectors are all part of this category. Industrial effluents contain a variety of pollutants, such as dyes, heavy metals, and medications. Research in recent years has focused on developing environmentally friendly technologies for the removal of pollutants from effluents, as these contaminants can be harmful to ecosystem flora and fauna, carcinogenic, and exhibit other hazardous features [1]. Because of the increasing danger posed by these dyes to aquatic life and their toxic effects on humans, physical and chemical methods such as adsorption, sedimentation and filtration, as well as the photo oxidation method were used to remove these dyes [2]. Among the methods used to remove these dyes, adsorption is the best in terms of cost and energy required [3]. Pigments are mainly classified based on the structural structure into dyes (acid - basic - dispersed - azo - pigments of metal compounds)[4]. Graphene oxide nano sheets were used as a highefficiency material to remove pigments because they contain functional oxygen groups such as alcohols, carboxylic acids and epoxides, and thus interact with the effective groups of those pollutants and their advantages are easily dispersed in water [5]. For ecological, evolutionary, and nutritional reasons as well as environmental ones, heavy metal toxicity is becoming an increasingly pressing concern. Heavy metals are major environmental contaminants [6][7]. The nonbiodegradable nature of heavy metals makes them extremely dangerous [8]. Unlike organic contaminants, heavy metals in soil cannot be eliminated; instead, they can only be moved to a new location, a process that is extremely costly. Soil heavy metals pose a threat to plant and animal life as well as water quality and, by extension, human health, prompting the development of other

approaches to mitigate these dangers and contaminations [9]. Metals with a particular density more than 5 g/cm³ are known as heavy metals, and they have negative effects on both the environment and living things [10]. Green chemistry focuses on the production of desired products without generation of hazardous intermediate by products in chemical reaction processes. Integrating green chemistry principles into nanotechnology has led to the identification of environmentally friendly reagents that are multifunctional, which they can serve as a reducing agent as well as a capping agent

1.2 Green Nanotechnology

Green Nanotechnology is a method for creating nanomaterials that either use very little or no hazardous chemicals [11]. Nanomaterial synthesis is a significant technological challenge as well as a hub for innovation [12]. Metal nanoparticles can help treat a variety of environmental contaminants, including heavy metals, toxic dyes, and antimicrobial substances [13]. The synthesis of nanoparticles can be accomplished by physical, chemical, or biological processes [14]. Chemical methods are expensive and environmentally dangerous, but they can produce clean, well-defined nanoparticles. As an alternative to current nanoparticle production processes, the numerous uses of biological biomasses—such as yeast, bacteria, fungi, various plant extracts, and various algal extracts—offer low-cost, eco-friendly, and safe options. To make nanoparticles that are very stable, scientists employ a process called green synthesis [15].

1.3 Capparis spinosa

The Capparidaceae family includes the perennial shrub genus Capparis, more often known as caper. Around 250 species belong to this genus. The plant, which goes by more than one name, makes good use of the abundant sunshine all summer long and shows no signs of water stress or photo-inhibition [16]. It has been used in the preparation of some nanocomposites in the green way due to the availability of effective phytochemicals in plant extract, especially in leaves such as ketones, aldehydes, flavones, amides, terpenoids, carboxylic acids, phenols, and ascorbic acids. These components are capable of reducing metal salts into metal nanoparticles[17].



Figure (1-1) Capparis spinosa plant

1.4 Literature Survey

- **J. Hua et al. in 2013** conducted a study on the removal of methylene blue and orange pigments, as well as cadmium ions, utilizing magnetized graphene oxide nanomaterial. The study indicated that the highest adsorption capacity for the two dyes was (64.23 mg/g). For cadmium ions, the adsorption amplitude was 20.85 mg/g [18].
- J. Yang et al. in 2014 in a study conducted on the production of a novel nanocomposite designated as (PANI-GO-LS). To make this nanocomposite, aniline was polymerized in situ with graphene oxide and lignosulfonate. (FESEM, TEM, FTIR, and UV-VIS) spectroscopy were used to analyze the shape and structure of the (PANI-GO-LS) triple nanocomposite. Additionally, researchers investigated the nanocomposite's ability to absorb lead ions (II). Batch experiments were used to examine the effects on lead ion (II) absorption in aqueous solutions of different adsorption periods, starting pH values, adsorbent concentrations, and initial absorption concentrations. The results showed that the (PANI-GO-LS) triple nanocomposite shows great potential to remove (Pb⁺²) ions from wastewater [19].
- **H. Raja et al. in 2015** concentrated on the manufacture of copper oxide nanoparticles using solution combustion utilizing plant extract from Gloriosa superba L. as a fuel source. Particles possess a monoclinic crystal structure, as shown by the XRD investigation. According to the scanning electron micrographs, the particles have a spherical shape [20].
- H. Mahdi et al in 2016 synthesized graphene oxide with magnesium oxide in order to eliminate the methylene blue color from its aqueous solution. The compound was characterized using transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction

(XRD). Several variables were investigated, including acid function and concentration. The oxides were mixed in different proportions, corresponding to different ratios of acid function (10.5 and 9.7, respectively). The adsorption isotherms showed the best fit with the Langmuir isotherm[21].

K. Henikish et al. in 2017 looked into the developed sol-gel method for producing nanoparticles of copper oxide. Numerous methods, such as X-ray diffraction (XRD), atomic force microscopy (AFM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM), were used to analyze their surface. The XRD examination indicated the presence of CuO based on the reflection peaks and their relative intensities, while the spectrum revealed a particle size of around (21.11 nm). This size estimation was consistent with the values obtained from SEM and TEM. Analyzing CuO using AFM, SEM, and TEM revealed that the particle sizes fall within the nanoscale [22].

S. Hariprasad et al. in 2018 conducted a study. The copper nanoparticles were synthesized utilizing a straightforward approach of environmentally friendly chemical reduction. This technology is both environmentally benign and economically efficient. The synthesis of copper nanoparticles at room temperature can be achieved effectively using this technology. The research paper presented the synthesis of copper nanoparticles using an tack out derived from the leaves of the plant Arivalanata. A (UV-VIS) spectrophotometer was used to confirm the presence of copper nanoparticles, and a combination of (UV-VIS, FTIR, SEM, and TEM) were employed to study their characteristics. E. coli, Staphylococcus aureus, Bacillus cereus, and Pseudomonas aeruginosa were used to test the antibacterial activity of copper nanoparticles[23].

Sally. K. S in 2018 conducted an experiment using two different ways to create copper oxide nanoparticles. The motivation of the experiment was to investigate the effectiveness of these nanoparticles in removing nickel and

cadmium(II) ions. Several parameters that influence the demineralization in the binary system on adsorbent (nanomaterials) have been investigated in this sector. Research shows that achieving equilibrium in a binary system including copper oxide nanoparticles made using the solution-gel approach takes (30 minutes), while nanoparticles prepared using fig tree leaf extract require (21 minutes). At a pH of (6), the ions were most effectively adsorbed on both surfaces. However, increasing the temperature was found to decrease the percentage of removal for both metals in the binary system, indicating that the process generates heat[24].

E. T. Bakir . et al in 2018 and his colleagues synthesized graphene oxide, graphene, and Graphene-1,3,4-oxadiazole-2thiol (rGS) by combining varying proportions of this material. They compound was diagnosed using techniques such as (FT-IR, XRD, SEM) to analyze the properties of the resulting membranes and assess their electrical conductivity. Electrical measurements were conducted within the frequency range of (5 KH to 0.5 MH) at standard room temperature. The findings indicated that there is Electrical conductivity increases with increasing frequency and decreasing the real and imaginary dielectric constant with increasing frequency [25]

Aseel. J. A in 2019 conducted a study on the removal of lead ions from aqueous solutions using prepared (NiMO/-Al₂O₃, new NiMO/-Al₂O₃, regenerated NiMO/-Al₂O₃) catalysts. The study examined various factors that affect the percentage removal of the metal onto the adsorbents. It was found that a contact time of (20 minutes) was sufficient for the removal of Pb⁺² ions. The calculated values of the thermodynamic functions (ΔG , ΔH , ΔS) indicated that the adsorption process was spontaneous, exothermic, and exhibited less randomness when lead metal was present on NiMO/-Al₂O₃ catalysts[26].

Omar. G. H in 2019 synthesized Graphene oxide, graphene, graphene with molybdate cobalt nanoparticles, graphene with ferrite oxide Cobalt The electrical properties of these membranes were examined, and the scientific findings demonstrated that the polymeric membranes incorporating graphene and its derivatives exhibited higher conductivity compared to films without nanomaterials. Additionally, these polymeric films were utilized in the production of highly efficient electrical amplifiers, with the results indicating a preference for dilators from the prepared cells. The materials used are graphene foils, cobalt ferrite oxide, and polyaniline[27].

Abdulilah. A. O in 2019 prepared MgO by the gel-liquid method, (GO) by modified hummer method and secondary composite (GO/MgO). The prepared oxides were used to study the adsorption of orange-G dye, this study included the optimal conditions of adsorption. They found that the best equilibration time is (125 min), the acid function is (3), the weight of the adsorbed surface is (0.05 g), and the concentration is (10ppm). the results of the study showed high correlation coefficients of the pseudo-second order [28]

Ali. A. S in 2019 created pure PVA polymer and polymeric mixes membranes, specifically PVA:PVP, with varying weight ratios. An investigation was conducted on the optical, thermal, and electrical characteristics, specifically insulation, of polymeric alloys. The scientific findings indicate that the thermal conductivity coefficient exhibits irregular behavior when compared to the pure PVA polymer membrane. This irregular behavior is observed when the weight ratios of the additive are increased. Additionally, the impact of the weight ratio of the polymeric mixture on the insulating electrical properties of the membranes was investigated. The results demonstrate a decrease in both the insulation constant and the loss factor. As the frequency increases, the insulation also increases for all ratios, and the electrical conductivity of the membranes grow with the growing frequency [29].

Abdullwahab. H. M in 2020 conducted a study involving the preparation of graphene oxide and graphene, as well as various nanomaterials such as titanium oxide and manganese oxide. Additionally, conductive polymers including poly aniline (PANI), poly (ortho phenylene di amine) and poly (anthralic acid) (PANA) were prepared. The researcher also created binary and ternary nanocomposites using the aforementioned materials. The composition and shape of all materials were determined through diagnostic techniques such as (TEM, SEM, EDX, and FTIR) measurements. The electrical properties of these composites were analyzed by blending them with (PVA) to fabricate the membranes, and it was observed that at low frequency At the maximum values of both real and imaginary dielectric permittivity, it was observed that the electrical characteristics enhance with the addition of nanomaterials and an increase in their concentration[30].

Raghad. L. K in 2020 synthesized (MnO₂) using the sol-gel method and (GO) using the Hummer method. Additionally, a (GO-MnO₂) nanocomposite was prepared with a weight ratio of (1:1). The prepared oxide was characterized using (AFM, FTIR, XRD, BET, and SEM). The produced nanocomposites were utilized for the adsorption of Congo Red and Rhodamin B from aqueous solutions. The study focused on the variables that influence the process of adsorption[31].

Zamn. R . A in 2021 studied the properties of graphene oxide, reduced graphene oxide, zinc oxide, and copper oxide and their conductivity in PoPDA polymer. He synthesized binary nanocomposites (ZnO-GO, ZnO-rGO, CuO-GO, and CuO-rGO), and novel triple nanocomposites using (PoPDA-CuO-GO). The electrical properties of these nanomaterials were analyzed using LCR techniques. The results showed an increase in dielectric constant values for hybrid PVA membranes, with the nanocomposite (ZnO-rGO-PoPDA:PVA) having higher values. The dielectric constant values for composites were also provided[32].

Nagham. J. A in 2021 created zinc oxide nanoparticles by utilizing Capparis Leaves Extract. Modern imaging modalities like atomic force energy dispersive X-ray spectroscopy, X-ray diffraction, , microscopy, Fourier transform infrared spectroscopy, and field emission scanning electron microscopy are utilized to portray each feature. This study examined several factors that influence the percentage of metal removal in a single system, including the volume of adsorbents and the time needed to achieve equilibrium in the removal of copper (II) and nickel (II) ions. A study demonstrated that it took (50 minutes) to eliminate the two metals from the surfaces of (CLE.ZnO.NP) and conventional zinc oxide. Additionally, it was observed that the removal of copper (II) and nickel (II) ions decreases slightly as the amount of adsorbents increases, but increases with an increase in surface weight. This process appears to be exothermic, since the impact of temperature on the elimination % seems to diminish with increasing temperature. Both surfaces' adsorption processes adhere to the pseudo second order equation, according to the data [33].

F. Amin. et. al in 2021, Synthesized copper oxide nanoparticles are taken from the leaves of the Aerva javanica plant. X-Ray diffraction, Fourier transform infrared spectroscopy, and scanning electron microscopy were the three methods used to characterize copper oxide nanoparticles that were manufactured using the green way. The crystalline structure of CuO-NPs is shown by X-ray diffraction (XRD), with an average crystal size of 15 nm. Scanning electron microscopy revealed that the particles are spherical in shape and have a size ranging from 15 to 23 nanometers. The antibacterial potential of the produced CuO-NPs was investigated against several bacterial and fungal diseases. The results showed that CuO-NPs had the strongest antibacterial effects against all of the tested fungi and bacteria. Nanoparticles of copper oxide were tested for their antimicrobial activity in comparison to the gold standard

antibiotics, Norfloxacin and amphotericin B. Antimicrobial agent with the lowest inhibitory effectiveness. It was claimed that toxicity was minimal at doses below 60 μ g/mL based on the cytotoxic activity of the produced CuO-NPs [34]

Zahraa. M. Sh in 2021 investigated how three dyes—Orange-G, Reactive Yellow 145, and Acid Fuchsin—adsorb onto a Nano Co-Polymer surface. Using condensation polymerization, the Nanoparticle Co-polymer was produced and subsequently evaluated using a variety of methods. The adsorption process was determined to be spontaneous, exothermic, and more random through the use of thermodynamic functions in the study. Using Freundlich, Temkin, and Langmuir assumptions, the data was evaluated, and the outcomes demonstrated a strong concordance with the isotherms [35].

Aya. M. N in 2022 searched into four varieties of ternary nano composites: GO-ZnO-PANI, rGO-ZnO-PANI, GO-Fe₃O₄-PANI, and rGO-Fe₃O₄-PANI. The methods of scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR) are used to analyze all nanomaterials. Next, the films were created by dissolving polyvinyl alcohol (PVA) in hot water and combining the resultant solution with the previously synthesized compounds to produce the films. The electrical properties of the films and nano materials were analyzed using LCR measurements, while the thermal properties of the films were also examined. The polymer sheets with electrical characteristics were utilized for the fabrication of a supercapacitor[36].

L. A. Mohammed et al. in 2023 studied preparation, a ternary nanocomposite consisting of reduced graphene oxide (rGO), manganese oxide nanorods (MnO₂), and poly (o-phenylenediamine) (PoPDA) was synthesized. Furthermore, the nanocomposite (rGO-MnO₂-PoPDA/PVA) was utilized to produce films with different weight percentages (ranging from 1% to 5%) of

PVA. Regarding these manufactured films, the study examined the dielectric properties of real (ε), imaginary part of dielectric constant (ε "), and electrical conductivity (ac). The investigation demonstrated that the real and imaginary permittivity reached their maximum value at the lowest frequency and subsequently commenced. The frequency exhibited a progressive drop, whereas the electrical conductivity demonstrated a gradual increase. Surge in occurrence. Where there is a noticeable rise in the value of electrical conductivity[37].

Afrah. A. J in 2023 prepared of (CuO) by aqueous solution of the plant extract (cumin). As well as the preparation of nanocomposite (MWCNTs/CuO). Research was conducted on the adsorption process of rhodamine B and red Congo dyes, taking into account the effects of equilibrium time, surface weight, pH function, initial concentration, and temperature [38].

D. Bejjanki et al. in 2023 The study presents a chemical oxidation polymerization method for creating a ternary nanocomposite, SnO₂/rGO/PANI, with superior specific capacitance, energy density, and performance, making it a promising supercapacitor electrode material.[39].

1.5 Aim of study

- **1.** Preparation of nano oxides: graphene oxide (GO), reduced graphene oxide (rGO), and copper oxide (CuO) using a green method that included a reducing agent made from a plant extract from Capparies spinosa.
- **2.** Fabrication of pure PVA films and supported utilizing ternary nanocomposites (GO-CuO-PANI), (rGO-CuO-PANI) and analyze the characteristics of these membranes in electrical and thermal conductivity.
- **3.** The removal of both orange-G dye and lead ion from their aqueous solutions with eco-friendly nano chemicals.
- **4.** Investigation of the optimal conditions for the adsorption of orange-G dye on the synthesized nano-surfaces (GO-CuO), (rGO-CuO), and lead ion on the surface (GO-CuO-PANI) including the weight of the adsorbent surface, equilibrium time, pH, concentration, and temperature.
- 5. Determine the activation energy value and the rate constant for the adsorption process for each surface and with of the orange-G dye by using the first and second order models to study the reaction's kinetics.

الخلاصة

في هذه الدراسة تم تصنيع أوكسيد النحاس (CuO) من المستخلص النباتي (Capparis spinosa) باستخدام الطريقة المخضراء، ثم تم تحضير أوكسيد الجرافين (GO) باستخدام طريقة هامر المعدلة وتم الحصول على أوكسيد الجرافين المختزل (rGO) عن طريق اختزاله بالهيدرازين. ثم تم تصنيع البولي أنيلين الموصل (PANI). تم تصنيع المتراكبات النانوية الثنائية (GO-CuO) و (rGO-CuO) باستخدام المكونات الأساسية المذكورة أعلاه. في النهاية ، تم استخدام المتراكبات الثنائية المذكورة أعلاه مع البولي انيلين (PANI) لتحضير المتراكبات النانوية الثلاثية (rGO-CuO-PANI) باستخدام البلمرة في الموقع. تم تحليل المتراكبات النانوية باستخدام تقنيات الأشعة تحت الحمراء (FTIR) ، وحيود الأشعة السينية (XRD) ، والمجهر الإلكتروني الماسح للانبعاث الحمراء (FTIR)). تم تصنيع الأغشية عن طريق إذابة بولي فينيل الكحول (PVA) في الماء الساخن ثم دمج المحلول الناتج مع المتراكب النانوي الثلاثي بنسب وزن مختلفة لإنتاج الأفلام.

أشارت قياسات الخصائص الحرارية إلى أن معامل التوصيل الحراري يرتفع مع زيادة نسبة وزن الجسيمات النانوية الهجينة. تم استخدام المتراكب النانوي الثلاثي (GO-CuO-PANI) لإزالة تلوث أيونات الرصاص واستخدام المتراكبات النانوية (GO-CuO)، (GO-CuO) لإزالة تلوث الصبغة البرتقالية (orange-G) من محاليلها المائية المخففة. تم فحص عدد من العوامل المؤثرة على معدل إزالة تلوث أيون الرصاص والصبغة البرتقالية (OG) للمادة الممتزة. كانت المدة اللازمة لإزالة أيونات الرصاص وتحقيق الاتزان هي (20 دقيقة) بينما كانت افضل ازالة للسطح (GO-CuO) هي (GC-Cuō) هي

والسطح (rGO-CuO) كانت أفضل إزالة له هي (10 دقيقة) ، الوزن الأمثل للإزالة (0.05 جرام) في جميع اسطح المتراكبات النانوية. لوحظ أفضل امتزاز لأيون الرصاص على السطح المحضر عند الاس الهيدروجيني (PH=2) بينما كانت أفضل إزالة للسطحين (rGO-CuO) (rGO-CuO) عند (PH=2). أشارت النتائج إلى أن عملية الامتزاز تتوافق مع حركية المرتبة الثانية الكاذبة للسطحين (GO-CuO) أشارت النتائج إلى أن عملية الوظائف الديناميكية الحرارية المختلفة للامتزاز أيضا ، ومن الواضح أن العملية باعثة للحرارة بالنسبة لايون الرصاص والسطح (GO-CuO) ، في حين أن السطح (-GO-cuO) كان ماصا للحرارة ,(Δ G) له قيم سالبة للمتراكبات النانوية الثنائية مما يشير إلى أن عمليات الامتزاز تحدث تلقائيا. أصبحت الجزيئات أقل تقييدا ، وفقا للقيم الموجبة (Δ G). أظهر ايزوثيرم الصبغة البرتقالية (Δ G) للسطح (-rGO-cuO) تطابقا أكبر مع ايزوثيرم فريندلش وأظهر السطح (-GO-cuO) تطابقا أكبر مع ايزوثيرم تيمكن .



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2024 هـ 1446