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Nanomaterial in the treatment of heavy metals in industrial wastewater from the Northern Refineries Company, Iraq

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ABSTRACT: In this study, a nanomaterial was synthesized from attapulgite clay to act as a new adsorption material. The synthesis of a compound (Attapulgite-$NiCoFe_2O_4$) was demonstrated within nanoscale measurements using X-ray Diffraction Measurements (XRD), Scanning Electron Microscopy (SEM), Surface Area Analysis, Vibrating Sample Magnetometer (VSM) and Transmission Electron Microscopy (TEM). Batch operations were carried out to study each of the effects of acidity, contact time, adsorbent dose, and ion concentration. The ideal values for the adsorption of lead were: pH (7), contact time (30) minutes, weight of the adsorption material (200 mg/L) and ion concentration (0.2 mg/L). The adsorption rate of lead was (88%).
Keywords: attapulgite ($NiCoFe_2O_4$); copper (Cu); chromium (Cr); zinc (Zn); cadmium (Cd); lead (Pb).

Nanomaterial no tratamento de metais pesados em águas residuais industriais da Companhia de Refinarias do Norte, Iraque

RESUMO: Neste estudo, um nanomaterial foi sintetizado a partir da argila attapulgita para atuar como um novo material adsorvente. A síntese de um composto (Attapulgite-$NiCoFe_2O_4$) foi demonstrada em nanoescala por meio de medições de difração de raios X (XRD), microscopia eletrônica de varredura (SEM), análise de área superficial, magnetômetro de amostra vibrante (VSM) e microscopia eletrônica de transmissão (TEM). Operações em lote foram realizadas para estudar o efeito da acidez, tempo de contato, dose do adsorvente e concentração de íons. Os valores ideais para a adsorção de chumbo foram: pH (7), tempo de contato (30) minutos, peso do material de adsorção (200 mg/L) e concentração de íons (0,2 mg/L). A taxa de adsorção de chumbo foi de (88%).
Palavras-chave: attapulgite ($NiCoFe_2O_4$), cobre (Cu); cromo (Cr); zinco (Zn); cadmium (Cd); prata (Pb).

1. INTRODUCTION

Heavy metal pollution is an environmental problem of great concern worldwide. Soil and water are often polluted, through which pollution enters into other systems such as plants, animals and humans (UDDIN, 2017). Although some heavy metals are required in small amounts for proper metabolism in living organisms, increasing their concentration beyond normal limits in living organisms poses significant health risks (DURAND, 2012). Some are harmful even in their low concentrations, and represent dangerous environmental pollutants, being non-dissolvable. They remain suspended or partially dissolved in the water column, enter the body through food, air, or polluted water, and accumulate in it over time, causing various harm to the organism (BLANCO, 2005). This prompted many industries to build industrial wastewater treatment units, so that they are suitable for reuse again, by establishing internal networks and conducting studies to dispose of waste left behind in these units (PORTER, 1989).

Many traditional methods have been used to purify and remove heavy metals from water, examples of which are coagulation (Kumar et al., 2004), reverse osmosis (Ning, 2002), ion exchange (Kim; Benjamin, 2004) and others. However, these techniques have many drawbacks; therefore, the need for alternative and effective techniques is imperative (TUUTIJARVI et al., 2009). It was found that the adsorption procedure is the best technique compared to other techniques for removing heavy metals from water in terms of cost, flexibility, simplicity of design, ease of use, insensitivity to toxic substances, and improvement in removal efficiency (AMER et al., 2010).

The current study aims at: 1 - Studying the concentrations of (5) heavy metals in the liquid waste generated from the North Refineries Company, which are copper (Cu), chromium (Cr), zinc (Zn), cadmium (Cd), and lead (Pb). This is conducted over the course of a year in a quarterly system and compared with the specified global ratios; 2-Evaluating the feasibility of using a locally available natural material, which is attapulgite clay, to adsorb lead ion from the industrial wastewater of the North Refineries Company; 3-Identifying the optimal conditions for lead removal, namely the acidity function (pH), the dose of the adsorbent, the reaction time, and the concentration of solute ions; 4-Determining the concentrations of the heavy metals in the industrial drainage wastewater, such as Zinc and lead.

2. MATERIAL AND METHODS

2.1. Purification and sieving of Attapulgite

Attapulgite clay was obtained from the Department of Geological Survey of the Ministry of Industry and Minerals in Baghdad, where 100 grams of attapulgite ore were taken and grounded using an electric agate mill for half an hour. After the process of grinding the attapulgite and obtaining a powder, the attapulgite powder was sifted by a sieve with a mesh size of 75 μm, and then the ground powder was collected and dried in the oven at 70 ºC for 24hrs.

2.2. Preparation of $NiCoFe_2O_4$

An amount (2.9712g) of $NiCl_2$ dissolved in (100 mL) deionized water was mixed with (2.9741g) of $CoCl_2$ dissolved in (100mL) deionized water and the mixture was left to stir for 15 minutes. Then, (8.11g) of $FeCl_3$ dissolved in (200 mL) of deionized water was added, and the mixture was stirred for 15 minutes. The mixture was heated to 60ºC, and NaOH was added until reaching a pH value of (11) to form a gel solution. After that, the formed gel was collected using a centrifuge. Then, the resulting product was calcined (heat treatment) at 600 ºC for 2 hours to obtain $NiCoFe_2O_4$. One gram of the substance was added with (100 mL) deionized water and then placed in the ultrasonic device for 15 minutes to regulate the area.

2.3. Preparation of Attapulgite-$NiCoFe_2O_4$

The proportions of 10 grams of attapulgite suspended in 250 ml of deionized water were mixed with 5 grams of $NiCoFe_2O_4$. The process of suspending the materials with water was carried out using an ultrasonic processor, after which the mixture was heated with stirring for four hours at 100 ºC. Then, the formation of the product was tested by bringing a neodemium magnet closer together. The entire clay containing $NiCoFe_2O_4$ was attracted to the magnet, confirming the transformation of the clay into the magnetic form.

2.4. Testing nanoparticles for heavy metal removal

The concentrations of (5) heavy metals were measured using a visible spectrophotometer of the type (HACH DR3900). The heavy metals studied are copper (Cu), chromium (Cr), zinc (Zn), cadmium (Cd), and lead (pb). These minerals were chosen because they are the most common in industrial wastewater within the aforementioned study stations. Lead (Pb) was chosen for treatment from among the heavy metals studied, and this is since this element is the most polluting element for the wastewater of the North Refineries Company within the aforementioned study sites and throughout the study period. A solution of lead ions was prepared together with the following ratios (0.1, 0.5, 0.11, 0.2 mg/L). It was chosen with these concentrations, as it represents the same concentrations present in the study samples during the study period and for the third station, which is before the mouth of the canal in the Tigris River.

The adsorption process took place under the influence of the conditions (adsorbent dose, pH, solute concentration, reaction time) by adding different doses of the adsorbent (Attapulgite-$NiCoFe_2O_4$), and with the following weights: (10mg,100mg, 200mg, 250mg) to (50 ML) of water (prepared ion solution) in glass flasks measuring (250m/L). Then, the pH was changed to be in the following concentrations (3,5,7,9) with the adsorbent (prepared ion solution) concentration for lead was studied according to the same previously prepared concentrations. After that, it was placed on the shaker after closing the mouth of each beaker using Parafilm and at different times of (5,15,25,30) minutes. The mixing speed was (180 rpm) rotation per minute. Each mixture was filtered separately using filter paper (0.45 μm) to separate the adsorbent solids. Finally, the resulting extract was taken and examined with a visible spectrophotometer.

All experiments were conducted at the laboratory temperature, as the device readings showed a high removal rate for the studied heavy metals.

3. RESULTS

3.1. Measurements of Nanoparticles

It has been also proven that the prepared nanomaterials of (Attapulgite-$NiCoFe_2O_4$) compound are within the diffraction of nanoscale measurements. This has been proven using X-ray Diffraction Measurements (XRD), Scanning Electron Microscopy (SEM), Vibrating Sample Magnetometer (VSM), and Transmission Electron Microscopy (TEM).

XRD demonstrated the synthesis of a compound (Attapulgite-$NiCoFe_2O_4$) by the one-step mixing method. The XRD measurement in Figure 1 shows the characteristic peaks of attapulgite and ferrite together, which is an indication of the success of the reaction to form the required nanocomposite. The presence of some other peaks, such as the peak at 11.66 degrees, is an indication of the presence of other components within the Iraqi clay (attapulgite), and we could not identify them because the Iraqi ores are considered mixed ores (ABDULKAREEM et al., 2022).

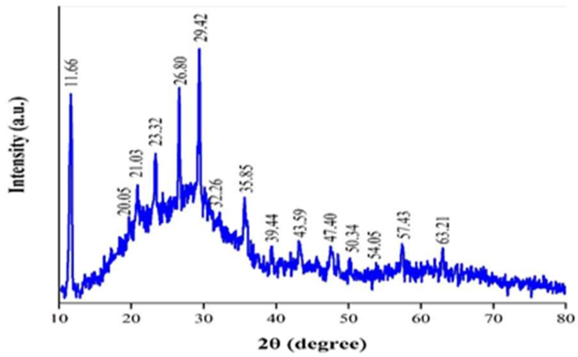

Figure 1. XRD of (Attapulgite - $NiCoFe_2O_4$).
Figura 1. DRX de (Atapulgita - $NiCoFe_2O_4$).

The characteristic structure of attapulgite is a structure similar to a bundle of interconnected channels, resulting from the bonding of alumina and silica layers to each other within the crystal lattice with the presence of hydroxyl groups (ZENG et al., 2022). Accordingly, this measurement was used to establish the presence of this structure and to record the success of the reaction by monitoring other components on these channels. The SEM measurements in Figure 2 show the presence of separate channels with a diameter of 29.74 nm.

The diameter of the un-interacting Attapulgite channels is approximately 160 nm, which was recorded by the researcher confirming the presence of ferrite. Moreover, measurement at a lower magnification in the second measurement shown in Figure 2 proves a material with very high porosity and a surface with high roughness. Thus, this provides a high ability to absorb pollutants. However, the high porosity is achieved through the nanomaterial ferrite acting and creating high porosity on the surface by perforating it (ALLAWI et al., 2022).

A magnetic analysis of the slurry was carried out after it was hardened with $NiCoFe_2O_4$ to determine the possibility of the material being attracted to magnets and whether it had superior magnetic properties. The measurement proved that the VSM value was approximately 75.1 emu, indicating that the attraction would need a strong magnet because it is weak. This is due to the presence of clay in its composition and the percentage of $NiCoFe_2O_4$ was low compared to clay. Figure 3 shows this value is lower than that recorded for $NiCoFe_2O_4$ (ALLAWI et al., 2022).

As shown in the TEM measurement in Figure 4 and by several measurements with different magnifications, the composite was prepared successfully, because it clearly shows the presence of each of the characteristic structure of Attapulgite in the form of separate channels (AL KAABI et al., 2022). Moreover, there are the dark structures and shapes approaching the spherical shape that characterize $NiCoFe_2O_4$.

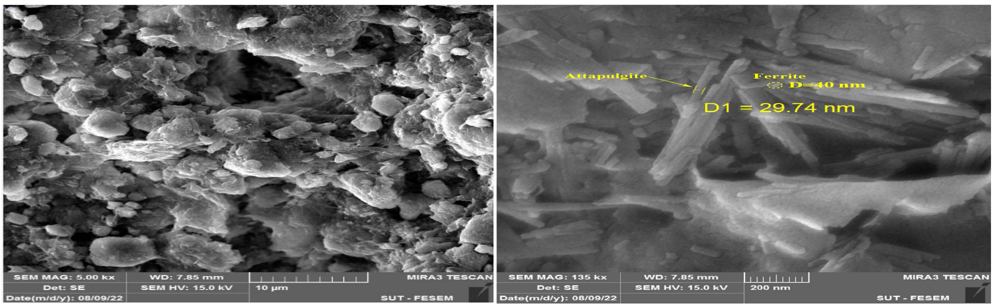

Figure 2. SEM with different magnifications for (Attapulgite-$NiCoFe_2O_4$).
Figura 2. MEV com diferentes ampliações para (Atapulgita- $NiCoFe_2O_4$).

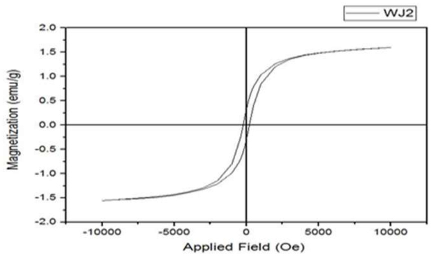

Figure 3. VSM of CLAY-$NiCoFe_2O_4$.
Figura 3. VSM de CLAY- $NiCoFe_2O_4$.

3.2. Lead Treatment

The results of the study showed that the concentrations of the studied heavy metals, which are lead, cadmium, copper, zinc and chromium, did not match the Iraqi determinants of the system for protecting rivers from pollution and the environmental determinants according to the specifications of the US Environmental Protection Agency, as shown in Tables 1, 2, 3, 4. The results of the current study, shown in Table 5, clarified that the treatment using the prepared nanoparticles showed efficiency in removing lead. The highest removal rate was 88% when using conditions with a constant acidic function (pH = 7 and a concentration of 0.2 mg/L) for the ion of the adsorbent element (lead) and the dose of the prepared nanomaterial (adsorbent) with a weight of (200 mg/L) and a reaction time of 30 min.

This is compared with the lowest removal rate, which reached 44%, when using conditions with an acidic function of pH = 7, and doses with the reaction times with the concentration of the ion of the adsorbent element being constant. This is shown in Table 2.

The removal rate when using a function of acidity, periods of time, and fixed adsorbent ion concentrations was 67% and at a dose of adsorbent (200 mg/L), as shown in Table 3. Whereas, when using a pH function, doses and fixed reaction times, the highest removal rate at the concentration (0.2mg/L) of the adsorbent element ion reached 86 %, as shown in Table 4.

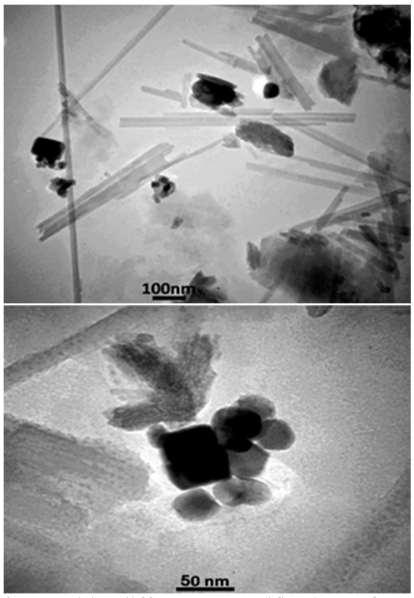

Figure 4. TEM with different magnifications for (Attapulgite-$NiCoFe_2O_4$).
Figura 4. TEM com diferentes ampliações para (Atapulgita-$NiCoFe_2O_4$).

Table 1. Results of an examination of the determination of some heavy elements in the industrial water of the North Refineries Company for the month of December (winter season).
Tabela 1. Resultados do exame de determinação de alguns elementos pesados nas águas industriais da Companhia de Refinarias do Norte referente ao mês de dezembro (época de inverno).

The site	Cr ppm	Cu ppm	Zn ppm	Cd ppm	Pb ppm
The main company	0.057	0.029	0.8	0.04	0.17
Post-treatment (Nouri channel)	0.043	0.021	0.7	0.03	0.9
Before the downstream point 500 meters	0.016	0.07	0.5	0.013	0.1
estuary point in the Tigris River	0.047	0.09	0.6	0.01	0.3
1000 meters after the downstream point	0.051	0.05	0.5	0.009	0.1

Table 2. Results of an examination of the determination of some heavy elements in the industrial water of the North Refineries Company for the month of March (spring season).
Tabela 2. Resultados do exame de determinação de alguns elementos pesados nas águas industriais da Companhia de Refinarias do Norte referente ao mês de março (época da primavera).

The site	Cr ppm	Cu ppm	Zn ppm	Cd ppm	Pb ppm
The main company	0.086	0.032	0.13	0.045	0.41
Post-treatment (Nouri channel)	0.072	0.026	0.11	0.036	0.29
Before the downstream point 500 meters	0.038	0.08	0.8	0.03	0.5
estuary point in the Tigris River	0.078	0.1	0.9	0.027	0.8
1000 meters after the downstream point	0.082	0.09	0.7	0.019	0.7

Table 3. Results of an examination of the determination of some heavy elements in the industrial water of the North Refineries Company for the month of July (summer season).
Tabela 3. Resultados do exame de determinação de alguns elementos pesados nas águas industriais da Companhia de Refinarias do Norte referente ao mês de Julho (época do verão).

The site	Cr ppm	Cu ppm	Zn ppm	Cd ppm	Pb ppm
The main company	0.091	0.031	0.11	0.038	0.49
Post-treatment (Nouri channel)	0.078	0.022	0.9	0.027	0.34
Before the downstream point 500 meters	0.042	0.09	0.7	0.023	0.11
estuary point in the Tigris River	0.080	0.1	0.8	0.02	0.13
1000 meters after the downstream point	0.082	0.08	0.7	0.016	0.12

Table 4. Results of an examination of the determination of some heavy elements in the industrial water of the North Refineries Company for the month of October (autumn season).
Tabela 4. Resultados do exame de determinação de alguns elementos pesados nas águas industriais da Companhia de Refinarias do Norte referente ao mês de setembro (época do outono).

The site	Cr ppm	Cu ppm	Zn ppm	Cd ppm	Pb ppm
The main company	0.065	0.027	0.10	0.03	0.24
Post-treatment (Nouri channel)	0.052	0.021	0.8	0.026	0.15
Before the downstream point 500 meters	0.027	0.05	0.6	0.021	0.2
estuary point in the Tigris River	0.061	0.09	0.7	0.018	0.4
1000 meters after the downstream point	0.053	0.06	0.5	0.015	0.2

Table 5. The optimal conditions for the adsorption of lead from industrial water.
Tabela 5. Condições ótimas para adsorção de chumbo de água industrial.

No.	Element	pH	Adsorbent dose (mg/L)	Reaction time (min)	Prepared solution (mg/L)	Concentration after treatment	Removal Ratio
1-	Lead	7	200	15	0.2	0.04	80%
2-	Lead	7	200	25	0.2	0.032	84%
3-	Lead	7	200	30	0.2	0.024	88%

Table 6. The stability of the effects of doses, times and concentrations of the adsorbent ion with the change in the pH value.
Tabela 6. Estabilidade dos efeitos das doses, tempos e concentrações do íon adsorvente com a alteração do valor do pH.

No.	Element	pH	Adsorbent dose (mg/L)	Reaction time (min)	Prepared solution (mg/L)	Concentration after treatment	Removal Ratio
1-	Lead	3	10	5	0.1	0.085	15%
2-	Lead	5	10	5	0.1	0.064	36%
3-	Lead	7	10	5	0.1	0.056	44%
4-	Lead	9	10	5	0.1	0.068	32%

Table 7. The stability of the pH conditions, times and concentrations of the adsorbent ion with the change in the weight of the dose of the adsorbent.
Tabela 7. Estabilidade das condições de pH, tempos e concentrações do íon adsorvente com a mudança no peso da dose do adsorvente.

No.	Element	pH	Adsorbent dose (mg/L)	Reaction time (min)	Prepared solution (mg/L)	Concentration after treatment	Removal Ratio
1-	Lead	7	100	5	0.1	0.049	51%
2-	Lead	7	200	5	0.1	0.033	67%
3-	Lead	7	250	5	0.1	0.034	66%

Table 8. The stability of the pH conditions, doses and times with the change in the adsorbent ion concentration.
Tabela 8. Estabilidade das condições de pH, doses e tempos com a mudança na concentração do íon adsorvente.

No.	Element	pH	Adsorbent dose (mg/L)	Reaction time (min)	Prepared solution (mg/L)	Concentration after treatment	Removal Ratio
1-	Lead	7	200	5	0.5	0.115	77%
2-	Lead	7	200	5	0.2	0.028	86%
3-	Lead	7	200	5	0.11	0.0341	69%

4. DISCUSSION

The physiochemical path of lead ion adsorption results from the physical, chemical and biological effects in the environmental systems in which this ion is present, affecting its biogeochemical cycle in those systems. The specific adsorption is characterized by high binding energy and depends on the electronic arrangement of the surface group and the positive ion (SHIT, 2008; ALI; SALEM, 2012).

4.1. Effect of pH

In the current study, the effect of the pH function on the adsorption of lead ions by Attapulgite-$NiCoFe_2O_4$ at different pH levels (0.3-0.9) is studied. This range was chosen to avoid the hard metal hydroxide. It was found that the rate of removal of ions by the adsorbent surface depends on the acidity of the solutions, and the rate of removal of acidic ions increases. The quality of the acid paste on the charge of the surface is adsorbent and the degree of ionization, as it covered the percentage of removal in the strong acidic medium. Confidence confirms that it is between hydrogen ions and lead ions in determining the locations because it is for the surface of the adsorbent and because of the small size of sand, it prefers rapid desorption compared to lead ions. In the contrast medium, the percentage of charge removed due to a negative increase, increasing the bonding with lead ions. In this contract, the reduction in the percentage of lead ions is due to the phenomenon of electrostatic adsorption. The negative charges define the amount of the adsorbed material (LIU et al., 2016).

4.2. Effect of Adsorbent Dose

The effect of half of the adsorbent material was studied from the results of the composite Attapulgite-$NiCoFe_2O_4$ in the adsorption process using different weights of the adsorbent material, which are (100, 200, 250 mg/L). It goes back to the adsorption rhyme that initially requires the presence of the vehicle between the adsorbent and the adsorbent so that it occupies all sites, but the (Active Size) then the adsorbent, which makes the adsorption process on the surface and the amount of adsorption reaches the value that represents the amount of adsorbent material in the saturation stage, but the increase in the weight of the surface The adsorption then leads to an unstable spread of the large adsorbent surface compared to the amount of the adsorbent material and for this, it will overcome the muttering energy as the adsorption on the adsorption energy on the surface, which leads to a decrease in the amount of the adsorbed material on the surface (ES-SAID et al., 2021).

4.3. Effect of Adsorbate

The initial effect of lead ions on the surface of the prepared superimposed was studied by using a range of concentrations (0.11, 0.2, 0.5 mg/L) with fixing the touches of the savory function, the dose of the adsorbent, and the touch time as written in Table 8. It found the best percentage reaching 86% at a concentration of 0.2 mg/L. This is due to the balance between the amount of the adsorbent and the amount of superimposed surface, and thus all organic matter becomes organic free of the surface molecule. At star concentrations, the removal rate is due to the separation of lead ions at the adsorbent surface sites (ARIAS; SEN, 2019).

4.4. Effect of Reaction Time

The equilibrium time of a solution of contaminated lead ions on the surface of the adsorbent superimposed was studied at several different time periods (15, 25, 30 min) and at a temperature of 20 °C, to detect the optimal time for the adsorption process. The results are shown in Table 5. shows that the process of removing lead increases with the increase in the reaction time, and after the time is 25 minutes, the increase is rather minor. This is because the active sites present in the adsorbent surface at the beginning of time are ready for adsorption until they are occupied, after which adsorption becomes slow and more difficult due to the occupancy of all the active sites of the surface with lead ions (UDDIN, 2017).

5. CONCLUSIONS

High concentrations of some heavy metals in the wastewater of the Northern Refineries Company (Zn, Pb, Cd, Cu, Cr), which exceeded the permissible limits due to the discharge of industrial wastewater issued from the company's operational and production units, and the inefficiency of the company's treatment unit in removing these Metals.

Attapulgite-$NiCoFe_2O_4$ showed high efficiency in removing lead (Pb) from the liquid waste of the Northern Refineries Company in the study sites near the Tigris River.

Attapulgite-$NiCoFe_2O_4$ achieved high efficiency in removing lead metal at the optimum pH (7), adsorbent dose (200 mg/L) and ion concentration (0.2 mg/L) with a reaction time (30 min).

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