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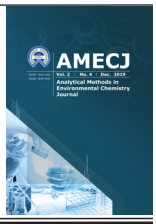

Enhancing the effect of zinc oxide on the absorption of heavy metals from wastewater by using silica in graphene bed

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ABSTRACT

In this study, the effects of nanostructure absorbent of zinc oxide (ZnO) in graphene bed for wastewater treatment were studied. Initial analysis was undertaken to identify the existing metals and their concentration in the prepared wastewater. It was seen that the diluted solution consisted of the ambivalence ions of lead, copper, nickel, cadmium, and silver with the concentration of 73.31, 81.19, 54.6, 98.1and 76.1 milligram per liter, respectively. Trivalent chrome, with a concentration of 98.1 milligram per liter was also observed. Therefore, by adding various amounts of absorbent (20, 30 and 50 mg) to the wastewater sample and adjusting the pH to 5 and 6, each metal was separately absorbed. Consequently, the concentration of the remaining metals was measured, and it was observed that absorbent was effective for the absorption of lead, copper and silver (with a reduction of up to 80%), however, the absorbent was weak in the absorption of nickel and chrome. Hence, the silicon nanoparticles added to absorbent and the experiments repeated. It was observed that the presence of silicone resulted in higher absorption of nickel and chrome but negatively affected the absorption of copper and silver. Electrical charges at lower pH's have an inverse impact on the absorption of metal ions that is due to the electrostatic repelling forces between the positive charges. In more acidic solutions, the carbonyl groups in the surface of composite create positive charges and hence repel the metal ions. Hence, the performance of the absorbent improves by reducing the acidity of the solution. At the pH of six, the number of hydroxide increases and the capacity of absorbing metal cations increase.

1. Introduction

During the past decades, due to population growth and industrial development, the demand for freshwater and wastewater treatment has significantly increased. This includes the treatment of industrial wastewater that contains considerable amounts of heavy metals, such as; lead, copper, chrome, cadmium, nickel, iron, zinc, arsenic, manganese and mercury. Among the various industries, the weaving industries produce a significant amount of chemically contaminated wastewater. The concentration of these chemicals depends on the raw materials and the methods of production. Due to the variety of different processes and their demand for large amounts of water, these industries are responsible for the production of considerable contaminated water. Production of textile requires several mechanical processes such as spinning, weaving, knitting, etc. [1]. Besides, there exist the "wet processes" that include wool washing, bleaching, and dying. During the production processes of fibers, cloth and, clothes several contaminators will be added

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to the wastewater. As such, during the dying process, heavy metals such as chrome and copper contaminate wastewater. It is vital to reduce and remove these metals, as these will disable the bacteria's in the biological treatment units. Additionally, these metals can contaminate surface and underground water sources, which due to their toxicity can cause death by affecting nerves and kidneys [2]. There are national and international codes and standards that define a limit for disposing of the wastewater containing heavy metals to the water resources. Exceeding these limits could be fatal and harm the environment. The main concern in wastewater treatment in weaving industries is the quantity of disposed wastewater. Dispose of wastewater in these industries are often to the absorbent wells, which will cause irreversible damages to the environment. The sampling results show high values of pH, BOD, COD and dye. The quantity of COD, BOD and TSS was reported 750 - 3500 milligram per liter, 300-1800 milligram per liter, 18-155 milligram per liter, respectively [3]. In the same study, the pH was observed to be varying from 5 to 12 and the quantity of dye was reported to be from 30 to 550 units. The dying weaves produce a significant amount of wastewater. If these were disposed to the environment without proper treatment, the damages to the environment would be significant. This reveals a pressing need for efficient methods of treatment. Cadmium and nickel are examples of toxic elements, which traces of these are seen in wastewater from mining, alloying and battery production industries. Adsorption is one of the methods used to reduce these elements. Nowadays various adsorbents are used with the capability of removing organic and inorganic contaminators, which the most common adsorbent is activated carbon. The activated carbon is not an economical solution for large scale treatment units, as there are significant losses of carbon in the regeneration process [4]. The critical elements for selecting the reduction methods are environmental issues, regeneration and economic matters. In the past few years, improvement in nanotechnologies helped in the production of nanostructures that are distinct due to their larger surface. The unique structure of nano adsorbents caused them to be high capacity adsorbents. On this basis, wastewater treatment is considered one of the main applications of nanotechnologies that have the potential to considerably improve the quality and capacity of the water and wastewater treatment units.

2. Experimental

2.1. ZnO nanostructure synthesis in graphene bed

Firstly, synthesis of the graphene sheets by oxidation process in accordance with hummer's method in the concentrated acidic media that contains mixing ratio of 1:2:46 of concentrated sulphuric acid, graphite powder, and sodium nitrate, respectively in 2°C temperature with continuous mixing. Afterward, potassium permanganate to a ratio of 6 added to mixture slowly and after oxidation reaction, mixture temperature rose to 40°C and mixing plateaued for 1 hour. Added distilled water and sodium hypochlorite solution stopped the reaction and trended the pH to neutral and then filtration, washing and drying mixture, respectively. The yellowish powder remained was graphene. In order to extend synthesized graphene sheets completely, pour 1mg graphene oxide powder in 100ml distilled water and apply ultrasound for 3 hours. The resulting solution, centrifuged for half an hour by 6500rpm in order to get out unexpended graphene sheets by sedimentation process from the solution. Then added 3 grams of zinc oxide salt powder ($6H_2O*Zn(NO_3)_2$) to remain solvent and apply ultrasound spanned 1 hour then stirred it slowly for more than 3 hours in 90 centigrade degrees. Poured the produced mixture in an autoclave tank and carried out a hydrothermal synthesis method for 6 hours at 180 °C. Then cooled it down to room temperature naturally and washed it with extra ethanol. The amount of resulting graphene was about 20 percent [1]. The final structure was a hybrid form of zinc monoxide at the surface of

graphene (GO/ ZnO) that was used as the first type of absorbent. Figure (1) illustrates the TEM of this structure. As shown in Figure (1) use of graphene sheets leads to produce an appropriate culture for bonding nanoparticles of zinc oxide on it without aggregation of zinc particles, in the second step silica nanoparticles added on this hybrid structure. Therefore, in order to build graphene/zinc/silica nano-composite firstly applied ultrasound for the solution of 0.5 grams synthesized GO/ZnO in 100 ml of Dionysius water for half an hour in room temperature. After that added 2 g of CTAB and mixed completely, then by using caustic soda adjusted pH around 9 and dropped 1mg of tetraethyl orthosilicate solution and heated by 40°C of temperature the mixture in a closed system with magnetic agitator for 24 hours. The next step was to dry out the solution after filtration the resulting mixture and washed it with extra Dionysius water in Avon with 60 °C for one day. This resulting powder is the second type absorbent of this study. Figure (2) illustrates the TEM of that. As shown in Figure (2) darkness particles of SiO_2 bonded and distributed correctly within ZnO nanoparticles that stabilized on graphene culture.so it can be seen that the second type absorbent structure formed as well.

2.2. Apparatus and Reagents

The furnace atomic absorption spectrophotometer (GF-AAS, GBC 932 plus, Australia) were used for the determination of Cu, Ni, Cd, Pb, Cr and Ag in samples. First, the manufacturer's manual book of GF-AAS was prepared. The hollow cathode lamp (HCL) with wavelength and current favorite for

Table 1. The instrumental conditions of heavy metals by GF-AAS

Element	Current(mA)	Wavelength(nm)	Slit(nm)	*LR	*LOD	*LOQ
Cu	4.0	327.4	0.5	1-30	0.3	1.0
Ni	9.0	229.0	0.2	1.5-60	0.4	1.5
Cd	3.0	228.8	0.5	0.2-6.5	0.05	0.2
Pb	3.0	283.3	0.4	2.0-70	0.5	2.0
Cr	7.0	357.9	1.5	1.0-15	0.3	1.0
Ag	4.0	328.1	0.5	0.2-4.2	0.05	0.2

*Linear range (LR, µg L^{-1}); Limit of detection (LOD, µg L^{-1}); Limit of quantification (LOQ, µg L^{-1})

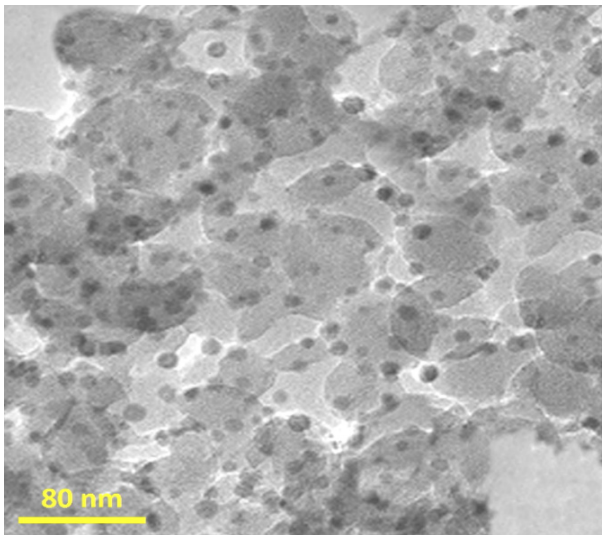

Fig. 2. TEM nanostructure of GO /ZnO/SiO_2

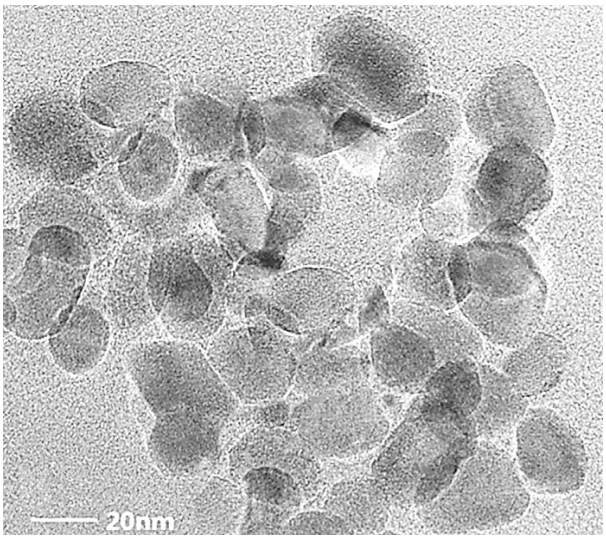

Fig. 1. TEM nanostructure of GO /ZnO

Table 2. The initial quantity of metal ions in sample wastewater

Pollutant	Metal ions	Initial quantity
Copper	Cu^{++}	19.8
Nickel	Ni^{++}	6.5
Cadmium	Cd^{++}	2.3
Lead	Pb^{++}	31.7
Chromium	Cr^{3+}	1.98
Silver	Ag^{++}	1.76

Cu, Ni, Cd, Pb, Cr and Ag was applied by 20 µL of sample injection to graphite tube of GF-AAS. For measuring pH, a Metrohm pH meter based on glass electrode was used (E-744, Switzerland) in wastewater samples**.** The instrumental conditions are listed in Table1.

2.3. Analytical Procedure

The adsorption of heavy metals such as, Cu, Ni, Cd, Pb, Cr and Ag based on GO/ZnO and GO/ZnO-SiO2 as sorbent were studied. All metals determined by electrothermal atomic absorption spectrometry (GBC, 932; ET-AAS). The different concentration heavy metals (1-50 ppm) in waters was used based on GO/ZnO and GO/ZnO-SiO2 (10-50 mg) for heavy meal adsorption at pH=5 and 6. After shacking, the heavy metals separated and adsorbed by GO/ZnO or GO/ZnO-SiO2. Other parameters such temperature, pH and sonication time were studied and optimized. In optimized conditions, temperature (25^{o}C), pH=5-6, and shacking of 5 min was achieved.

3. Results and Discussion

3.1. Dilution process

The wastewater used in this study belongs to an active center of the dyeing and printing industry, which kept motionless in a closed container for 24 hours span until sedimentation had been done. Then add an equal amount of Dionysius water into solution in order to half the pollutants concentration. Firstly, in order to assess the wastewater condition, measured the levels of metal ions by the atomic absorption method as listed in a Table (2). According to Table (1) the despite the dilution of prepared wastewater a large variety of heavy metals in significant amounts exists. In order to pursue the used absorbent function firstly added 0.2, 0.3 and 0.5 grams of it into 50 ml of selected wastewater. For pH adjustment used 0.1solution of NaOH. Each test carried out on 5-6 pH range.

Table (3) represents the samples and pH of experiments. In every experiment samples put on the ultrasonic device for 12 hours and afterward the liquid part of solution separated from the solid part by centrifuge equipment and then the concentration of remaining metals in solutions measured by atomic absorption machine. All experiments carried out at room temperature.

3.2. Discussion

As represented in Table (3), the existence of silica nanoparticles within the absorbent structure generally improves the adsorption function and increases adsorption efficiency that strongly subordinates with pH level, so that, in general view, the maximum efficiency of adsorption occurs when pH equals 6. Results in adsorption rate deal with pH value represent that it was rose up by increasing of the adsorbent amount, that the main reason for it is due to the total increase in adsorption valence for each adsorbent unit, in other words, by increasing of the adsorbent content, more adsorption bed provided for heavy metals. Also, it can be seen that the adsorption value varies for different metals which process differently within the dissimilar experimental conditions for each metal. Hence, in the following, the adsorption conditions for metals were investigated.

3.3. Lead adsorption

Results of Pb adsorption on used adsorbent shows that in pH of 5 the rate of adsorption increases by rising in amount of adsorbent, which is adsorbed 57.8% of Pb in case of 20 mg of adsorbent existence and by increase this till 30 mg, adsorption rate rises to 64.9%, in other way the adsorption rate improves 12 percent. Also in the concentration of

Table 3. Samples and pH status at the experiments

Adsorbent	conc.	pH	Remained concentration(mg L^{-1})					
			pb^{2+}	cu^{2+}	Ni^{2+}	cd^{2+}	Cr^{3+}	Ag^{2+}
Non	0		31.73	19.81	6.54	2.32	1.98	1.76
GO/ZnO-20	20		11.10	4.36	6.24	1.23	1.86	0.49
GO/ZnO-30	30		9.19	3.37	6.24	1.25	1.84	0.29
GO/ZnO-50	50	6	6.34	2.18	6.18	1.23	1.82	0.19
GO/ZnO-SiO2-20	20		8.94	8.12	3.12	1.18	1.43	0.68
GO/ZnO-SiO2-30	30		7.39	6.93	2.75	1.13	1.20	0.46
GO/ZnO-SiO2-50	50		5.05	5.38	2.62	1.04	1.15	0.34
Non	0		31.73	19.81	6.54	2.32	1.98	1.76
GO/ZnO	20		13.39	4.82	6.37	1.78	1.92	0.90
GO/ZnO	30		11.14	3.63	6.37	1.80	1.89	0.67
GO/ZnO	50	5	7.79	2.19	6.31	1.78	1.87	0.55
GO/ZnO-SiO2	20		12.85	10.36	4.00	1.72	1.81	1.13
GO/ZnO-SiO2	30		10.02	8.95	3.56	1.66	1.74	0.87
GO/ZnO-SiO2	50		6.27	7.26	3.41	1.55	1.68	0.73

50 milligrams of adsorbent, it reaches 75.4% that is equivalent to 30 percent of enhancement. Therefore, the first type adsorbent structure (graphene hybrid/ zinc oxide) represents an appropriate adsorbent for lead adsorption. Although the existence of silica within this adsorbent amplifies their ability of adsorption, this may not be significant, in a way, that adsorption improves 3% and 6% in the concentration of 20 milligrams and 50 milligrams, respectively, the results demonstrate in the Figure (3).

According to Figure (3), the presence of silica within the GO/ZnO adsorbent structure was ineffective to reach more adsorption of

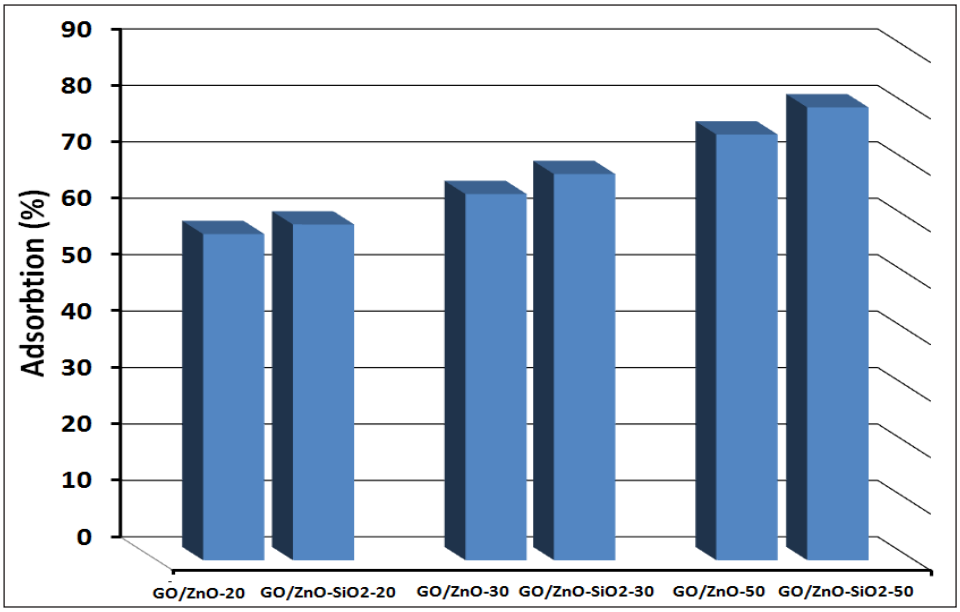

Fig. 3. Silica effect in GO/ZnO structure for Pb adsorption (pH=5)

Pb. Therefore, in existence 20 mg of adsorbent in wastewater 65%of Pb has adsorbed and by increasing this quantity to 30 mg adsorption yield rose up to 71%, in the other word adsorption improved 9% and in 50 milligrams of adsorbent, it reached 80%, equivalent 23% of increase. The same results obtained in pH of 6. Although the adsorption shows a little improvement in this pH, it is not noticeable. Figure (4) demonstrate the pH effect in different quantities of the second type of adsorbent in which silica added to their structure.

As illustrated the adsorption process is better in all amount of adsorbent (second type) in pH of 6 than 5, but the effect of pH in lower amounts of adsorbent (20 mg) is more significant, in a way, in higher concentrations 4% improvement has seen whereas in lower points of adsorbent's concentration it has been more than 20% (Fig.4).

3.4. Copper adsorption

Results of Cu adsorption on used adsorbent shows that in pH of 5 the rate of adsorption increases by rising in amount of adsorbent, which is adsorbed 75.7% of Pb in case of 20 milligrams of adsorbent existence and by increase this till 30 milligrams, adsorption rate rises to 81.7%, in other way the adsorption rate improves 8 percent. Also in the concentration of 50 milligrams of adsorbent, it reaches 88.9% that is equivalent to 17 percent of enhancement. Therefore, the adsorbent structure (GO/ZnO) is considered as an approximately appropriate adsorbent for copper adsorption. The existence of silica within this adsorbent structure reduces their function and abilities significantly, in a way that in this situation,20 milligrams of adsorbent low off about 37% in adsorption or 29% reduction for 50 milligrams of adsorbent's concentration. The results represent in Figure (5).

According to the bar chart in Figure (5) it is considered that added silica nanoparticles to GO/ZnO structure in order to more adsorption of copper not only didn't be efficient but also reduced it. The same results obtained in pH of 6. Although the adsorption level is better in this pH, it's behavior and the proceeding similarly continues, for 20 mg concentration of adsorbent in wastewater solution,78% of copper adsorbed and by its increase to 30 mg adsorption rose up to 83%, in other words, adsorption efficiency improved 6% improved. Also, 50 milligrams of adsorbent lead to 89% copper adsorption that is equivalent to 14% of improvement. Due to the function of both adsorbent structure, the existence of silica within this adsorbent structure strongly reduces the copper

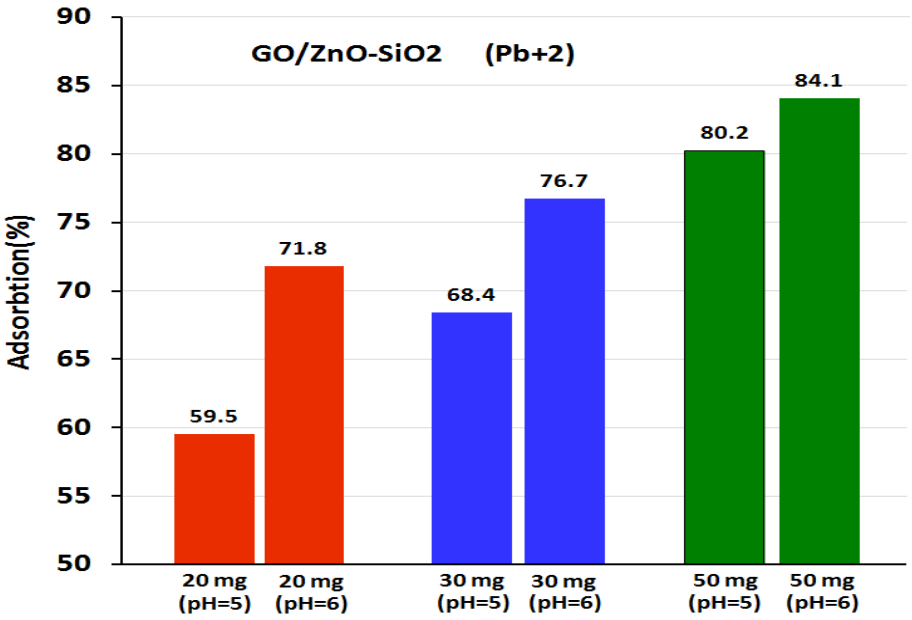

Fig. 4. Comparison of the pH effect by the different quantity of adsorbent GO/ZnO/SiO2

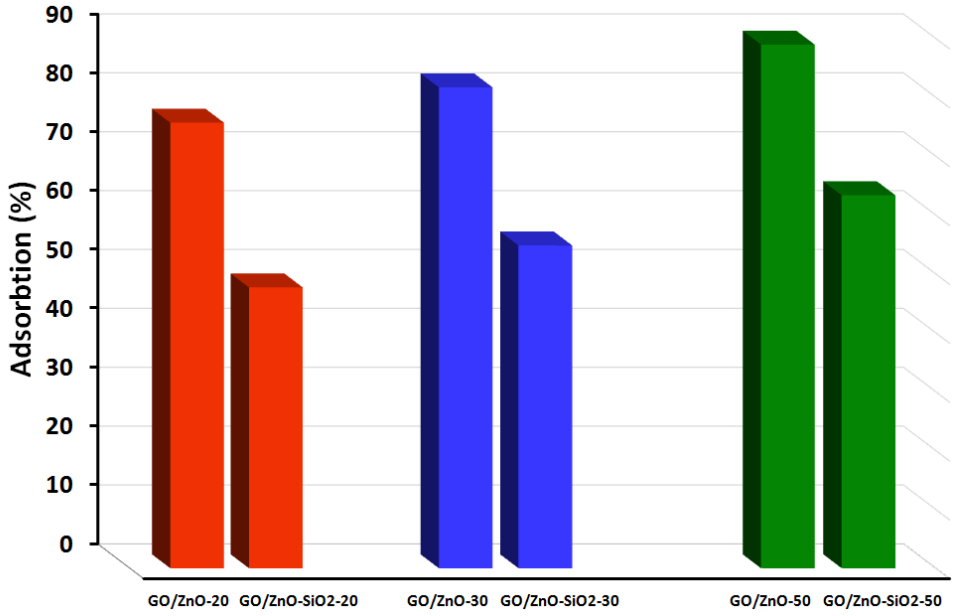

Fig. 5. Effect of silica within the GO/ZnO adsorbent structure to copper adsorption(pH=5)

adsorption which is subordinating to wastewater pH. Figure (6) represents the effect of pH for defined quantities of the second type adsorbent.

As represented in the Figure (6), the adsorption process is better in all amount of adsorbent (second type) in pH of 6 than 5. But, the similar to lead adsorption the effect of pH in lower amounts of adsorbent (20 mg) is more significant, in a way, in higher concentrations 14% improvement has seen whereas in lower points of adsorbent's concentration it has been more than 23%.

3.5. Nickel adsorption

Results of nickel absorption by the specified adsorbents express that in the pH of 5, adsorption rate rises by increasing adsorbent concentration.

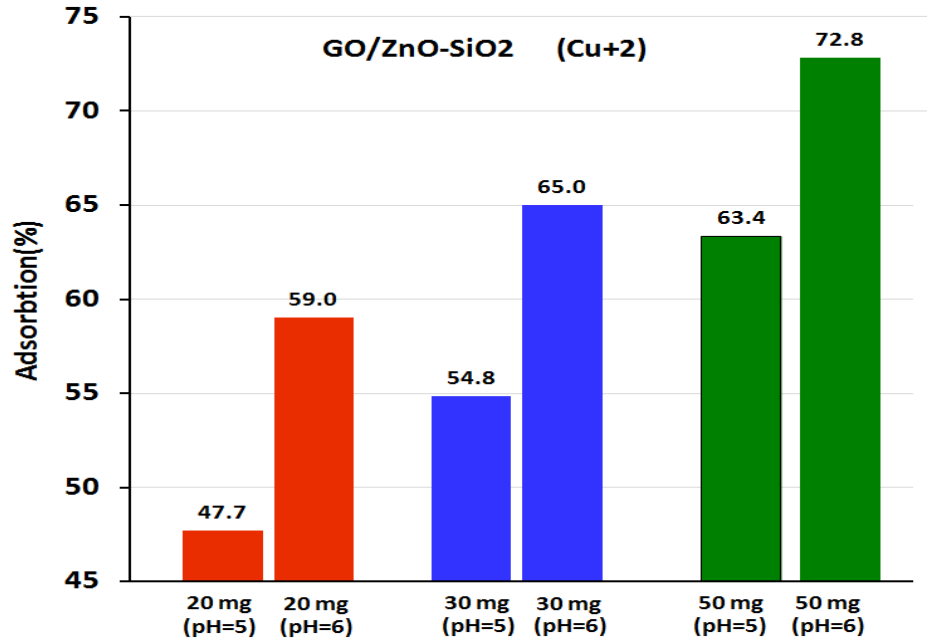

Fig. 6. Comparison of the pH effect on Cu adsorption by the different quantity of adsorbent GO/ZnO/SiO2

The first type of adsorbent acts weakly in nickel adsorption, in a way that due to the existence of 20 milligrams of adsorbent in wastewater, only 2.6% of nickel adsorbed and by rising its concentration to 30 milligrams adsorption plateaued and then went up slowly till 3.5% for 50 mg of the adsorbent concentration. Therefore, GO/ZnO structures have known as an inefficient adsorbent for nickel. Chang the adsorbent structure to GO/ZnO/SiO_2 strongly improved its ability in nickel adsorption, in a way that by added 20 milligrams of this structure into wastewater adsorption yield reached to 38.8%, despite of it is not acceptable yet but it shows that silica is the main factor to the nickel adsorption from wastewater. Also in 50 milligrams of adsorbent, the adsorption amount raised up to 45.6% that equivalent by 17% more nickel adsorption. The results demonstrate a bar chart in Figure (7).

As shown in Figure (7) the existence of silica nanoparticles within the GO/ZnO in order to more adsorption of nickel act effectively and strongly increased the adsorption results. Similar proceeding to this has shown in the pH of 6 meanwhile the results are better. Results showed that the adsorbent concentration of 20 milligrams in per liter of wastewater, adsorbed 52.3% of its nickel and by increasing that until 30 mg adsorption range reach to 58%, means adsorption efficiency improved 10 percent and also in 50 milligrams of adsorbent concentration, the adsorption amount was 59.9% that is equal to 14% increase in adsorption yield. On the other hand, the existence of silica in the adsorbent structure enhances nickel adsorption efficiently. As considered in both types of adsorbent function, it is obvious that silica leads to a significant increase of nickel adsorption. it is subordinated to the pH of wastewater. The pH effect on nickel adsorption in different quantities of the second type of adsorbent that is included silica represented as a bar chart in Figure (8).

As represented in the Figure (8) adsorption process is better in all amount of adsorbent (second type) in pH of 6 than 5, but unlike with lead metal adsorption, the pH's effect shows off with the same pattern in different quantities of the adsorbent, which shows 14% of improvement for higher concentration and 23% for lower ones.

3.6. Cadmium adsorption

The study results of cadmium adsorption on defined adsorbent represent that in pH of 5 adsorptions weren't changed by increasing of

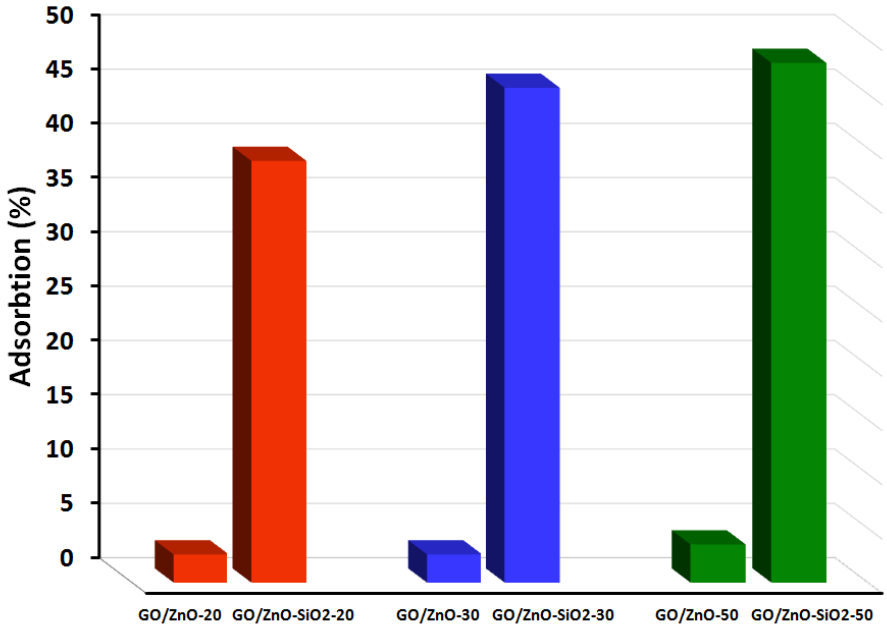

Fig. 7. Effect of silica within the GO/ZnO adsorbent structure to nickel adsorption (pH=5)

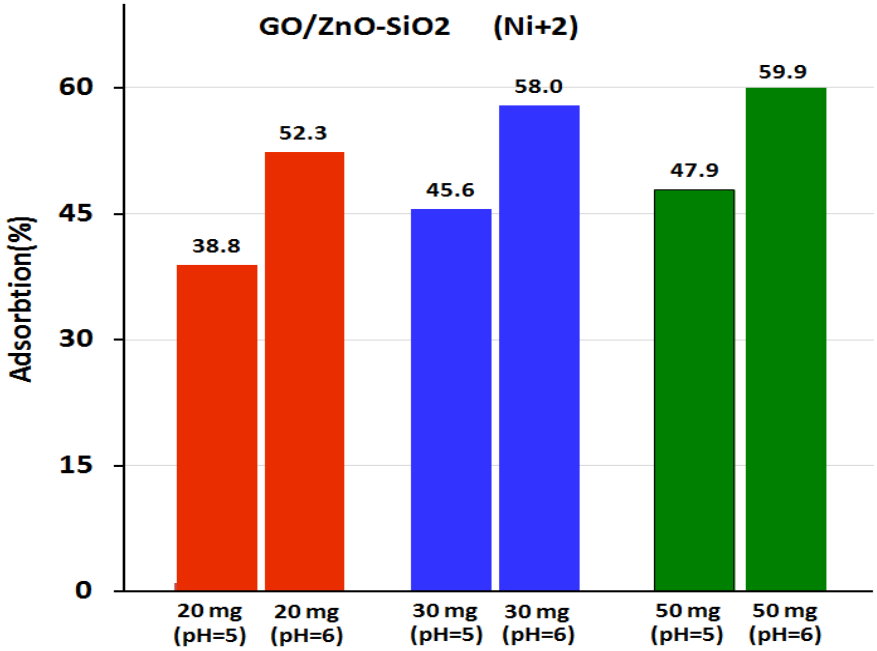

Fig. 8. Comparison of the pH effect on Ni adsorption by the different quantity of adsorbent GO/ZnO/SiO_2

adsorbent concentration. The first type of adsorbent ‘s function was weakness, in which,20 milligrams of its concentration leads to adsorb 23.3% of cadmium and by increasing that to 30 and then 50 milligrams per liter of wastewater the adsorption of cadmium plateaued. So, the GO/ZnO structure had seen as an adsorbent with moderate efficiency that absorbs a few amounts of cadmium at low levels and then remains constant. Even added silica to adsorbent structure did not change their ability to adsorb cadmium. The existence of 20 milligrams of adsorbent with silica could adsorb 25.9 % of cadmium that didn’t have noticeable difference by the first type of adsorbent. so, it seems that not only silica isn’t be adsorbing factor of cadmium in wastewater, but also due to steric hindrance of cadmium ions with atomic number of 48, adsorption of this heavy metal is based on surface of nanostructure that most of the time adsorption quantities overmatch by steric competition on adsorbent surface by increasing of adsorbent concentration. Also in 50 milligrams of adsorbent, the quantity of adsorption reaches 33.2% that it is 42 percent more cadmium adsorption than the case of silica absence. The results illustrated in Figure (9).

According to Figure (9), it showed that the existence of silica nanoparticles in the GO/ZnO structure in order to more cadmium adsorption act slowly and somewhat increases the adsorption. Similar results with about double improvement obtained that represents the strong effect of pH on the adsorption process. For this number of pH, 20 milligrams GO/ZnO/SiO_2 ،adsorbed 47% of cadmium from wastewater and by increasing in adsorbent concentration, the adsorption trends up insignificantly. Moreover, silica in the structure didn’t change the adsorption rate noticeable. Figure (10) demonstrates the effect of pH in different concentrations of the second type of adsorbent and it represents that in all quantities of GO/ZnO/SiO_2 adsorption yield is better in pH of 6 than 5, in a way that in high concentration of adsorbent 66% and in its lower concentration 89% improvement is seen. Therefore, adsorption gets better in the lower concentrations of adsorbent and changes the pH number makes it double.

4. Conclusions

The most important parameter in absorbing heavy

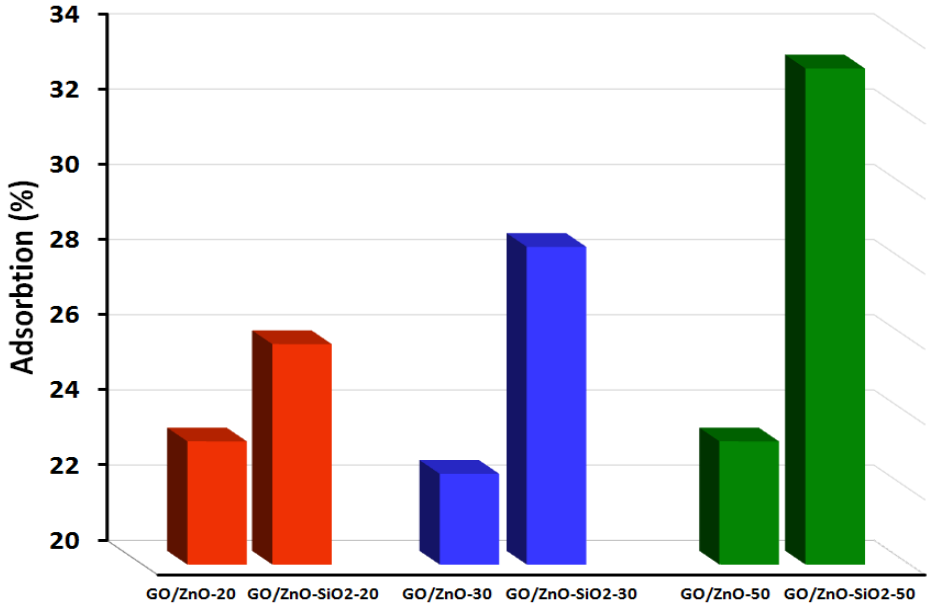

Fig. 9. Effect of silica within the GO/ZnO adsorbent structure to cadmium adsorption (pH=5)

metals in industrial wastewaters that contain a high concentration of metals is the acidity of the wastewater. The results of this experiment have shown that metals often exist in wastewater as ions, in which their interactions are controlled by the acidity of the wastewater. The metal cations in acidic environments are repelled by the cations in the solution, and hence their effects are converged. Under these circumstances, the adsorption processes are emulative and hence the adsorption efficiency diminishes [11]. Hence, one would be able to enhance the efficiency by adjusting pH, that is a function of metal ions in the wastewater. However, it must be considered that both basic and acidic environments are negatively affecting the adsorption efficiency. Hence the type and quantity of the existing metals and the adsorbent used can affect the adsorption process. In this study, it was also observed that the best adsorption process takes place at pH of around 6. The effect of silicon on the structure of nanocomposite, causes the absorbing surface to be positively charged, which are inappropriate for heavy metals. The electro-statically force between anions and cations, at increased pH, causes the efficiency of adsorbing nickel and cadmium to improve [12]. Additionally, the silicon nanostructure used in the adsorbent, oddly resulted in higher adsorption in pH of 6 as compared to pH of 5. Presence of silicon significantly increases the adsorption chrome but reduces the copper adsorption, and has a neutral effect on cadmium adsorption. From this, it can be concluded that the adsorption processes depend on chemistry of the adsorbs as well as the reaction of the existing dissolved metals. Therefore, in order to improve the performance of the graphene-based adsorbents with ZnO, it recommended that the heavy metals removed first, then reduced the chrome, and nickel using silicon. Also in pH of around 6, improves the performance of graphene bases to some extent and enhances the efficiency. Overall, the graphene-ZnO recommended for pre-treatment of wastewater by adsorbing heavy metals. Afterwards, there is a need for the main treatment to reduce the metals to their acceptable limits according to the standards. During the initial stages of adsorption, several adsorbing sites are available. However, as time passes, due to repelling forces between the absorbed matter and the dissolved molecules, the free sites are hardly usable. It was also observed that increasing the concentration of metals, causes the absorbing efficiency to drop. In lower concentrations the chance of adsorption increases and ions are able to react with the

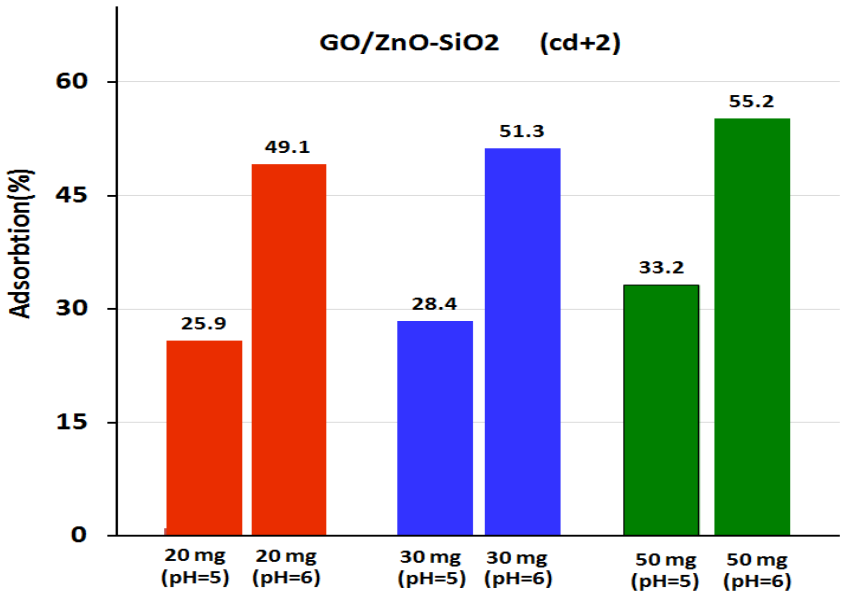

Fig. 10. Comparison of the pH effect on Cd adsorption by the different quantity of adsorbent GO/ZnO/SiO_2

adsorbing surface, which improves efficiency.

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