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Making activated carbon from the wood of the Juniperus excelsa and investigating arsenic adsorption in the effluents of factories containing arsenic

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ABSTRACT:
Nowadays, industries are developing so fast that often only their economic dimension is considered and their environmental consequences are overlooked. Industrial and municipal effluents mostly contain metal ions that cannot be easily destroyed, metabolized, and transformed into less hazardous substances. Their presence in the environment is considered a very serious threat. Arsenic is one of these metal ions. It is among the heavy metal ions. Arsenic disrupts the nervous system, blood circulation, digestion, and skin and threatens human health. It causes the death of people with acute poisoning. Thus, the removal of heavy metals, including arsenic, is crucial to prevent the destruction of the environment and damage to humans and animals. This study aimed to achieve the desired outcome with less energy and cost to help design the process of removing pollutants and accelerating the cleaning of industrial effluents containing various metals. This study investigated the separation of arsenic toxic pollutants from aqueous solution by surface adsorption on activated carbon obtained from the sawdust of the Juniperus excelsa. First, carbon was prepared from the sawdust of the Juniperus excelsa and chemically activated using phosphoric acid. To identify the properties of the studied activated carbon, iodine number, and electron microscope were used. Then, the adsorption characteristics of activated carbon in the removal of arsenic metal pollutants were evaluated. Design Expert statistical software was used in the design of experiments and analysis of the results. In pollutant adsorption experiments, the effect of adsorbent dosage, contact time, and solution pH factors were studied. By evaluating the results of the desirable separation conditions as described in contact time of 4 hours, adsorbent dose of 1.0 g, and pH of 9, the adsorption value of 75.967% was obtained.

Keywords: Adsorption, , Activated carbon, Arsenic, Sawdust

INTRODUCTION:

Nowadays, surface and underground waters are at risk of pollution due to the development of industries and the entry of industrial effluents into the ecosystem surrounding the factories. It leaves harmful effects on the living organisms of the soil, plants, and animals of these areas in the short and long term. The discharge of raw sewage into the environment is followed by many health and environmental risks. Thus, a suitable method is needed for wastewater treatment that can be justified economically and operationally. Nowadays, there are various methods for treating industrial effluents, including physical, biological, chemical, and surface adsorption methods based on the existing technology. The surface adsorption process generally is through mechanisms such as the external transfer of the mass of the dissolved substance to the adsorbent followed by intra-particle distribution.

This phenomenon applies to physical, chemical, and biological systems. It can be used for purification in gas and liquid systems synthetically and for water treatment systems and air pollution removal. Physical adsorption or van der Waals adsorption is a type of surface adsorption that is a reversible phenomenon that is the result of attractive forces between solid molecules and the adsorbed substance. Chemical adsorption or activated surface adsorption is another type of surface adsorption that is the result of chemical interactions between the solid substance and the adsorbed substance. Ganjidoust et al. investigated the removal of dye from aqueous solution with sawdust and bentonite clay. The dye removal efficiency was obtained at 25, 50, and 100, respectively, from solutions containing initial dye concentrations, 96.75, 91.11, and 79.26% respectively, for sawdust, and 97.32, 96.78, and 94.69, respectively, for bentonite

clay. Sheikh Mohammadi et al. investigated the use of elemental iron in removing Azo dyes from industrial wastewater. The results of the experiments revealed that pH plays a crucial role in the dye removal efficiency and the highest dye removal efficiency was observed at low pH. By increasing the pH from 3 to 11 with iron powder 3.33 g/l and a contact time of 100 minutes, the removal percentage decreases from about 94.84% to 16.24%.

Sharifi et al. investigated the potential application of adsorption of activated carbon prepared from myrtle fruit and pomegranate peel, which were used as adsorbents to remove concord from aqueous solutions. The adsorption capacity of activated carbon made from myrtle fruit and pomegranate peel to extract concord was 19.23 mg/g and 10.00 mg/g, respectively. Khomrnia et al. investigated the application of powdered activated carbon in removing azo dye (BV 16) from the aqueous environment. In this study, a pH of 3, contact time of 60 minutes, and dye concentration of 50 mg/liter were obtained as desirable conditions for dye removal. Keihani et al. investigated the efficiency of ash and sawdust of kiwi trees in removing Congo red (CR) dye from textile effluents. It was revealed that the pH variable did not have a significant impact on the adsorption by ash. However, regarding sawdust, the adsorption rate decreased with increasing pH. By increasing the adsorbent dosage from 2 g to 20 g, the adsorption rate for ash and sawdust increased from 70 to 95 and from 50 to 80 percent, respectively. Also, an increase in dye concentration led to a reduction in the adsorption rate. Zohri Jaliseh et al. investigated the removal of green acid dye 20 from simulated textile effluent using surface adsorption. The result indicated an efficiency of 93%.

The analysis of the laboratory results revealed that the adsorption of dye has a favorable match with the Langmuir isotherm. They also showed that kaolin is a suitable adsorbent for removing textile dyes from the effluents containing it. Heibati et al. evaluated activated carbon prepared from walnut wood in removing azo dyes in textile effluents and determining Acid Red18 isotherms. The results indicated that Acid Red18 dye follows the Freundlich isotherm ($R2>0.962$). The maximum adsorption capacity of this dye was calculated to be 32.8 mg/g using the Langmuir model. Generally, the results of this study revealed that activated carbon made from walnut wood can be used as an effective adsorbent to remove azo dyes. Darvishi et al. concluded that the preparation of carbon from Astragaluswood has a high potential for the removal of dye molecules. The values of dye adsorption capacity at pH 7.3 and 11 for the initial dye concentration of 50 mg/L and the adsorbent mass of 0.1 g/1 0.0 liter in 30 minutes 38.66, 40.48, and 48.5 mg/g, respectively, and the values of dye adsorption in concentrations of 50, 100 and 150 mg/l at pH 7, the adsorbent mass is 0.1 liter in 30 minutes was 66, 40.40, and 78.69 mg/g, respectively.

Industrial and urban effluents mostly contain metal ions that cannot be easily destroyed, metabolized, and transformed into less hazardous substances. Their presence in the environment is considered a very serious threat. Some of these metal ions include chromium, mercury, Lead, copper, nickel, zinc, cadmium, cobalt, uranium, and arsenic considered among the heavy metal ions. Arsenic threatens human health by disrupting the nervous system, blood circulation, digestion, and skin, so it causes the death of people with acute poisoning. The use of surface and underground water contaminated with arsenic to irrigate agricultural fields increases the concentration of this pollutant in the soil and increases its transfer to different parts of the plant, leading to disruption of the natural growth of the plant with toxic symptoms such as decreased root and stem biomass, necrosis of leaf buds, reduction of photosynthesis level, etc. The method of surface adsorption using cheap and natural adsorbents was significant due to its simplicity and high efficiency in removing heavy metals and many micro-pollutants. Young et al. (2013) used walnut shell charcoal to remove cesium from water. Vitela-Rodrigues and Rangel-Mendez (2013) used modified activated carbon to remove arsenic. This study investigated activated carbon from Juniperus excelsa wood to remove arsenic from effluent and the effect of different laboratory conditions such as pH, contact time, arsenic concentration, and adsorbent dosage in the adsorption process. The present study examined the possibility of using cheap activated carbon in the treatment of water containing arsenic pollution.

MATERIALS AND METHODS
Materials
- Sawdust
The sawdust used for activated carbon was prepared from the wood of Juniperus excels located on the road of Darghaz-Quchan City.
-Phosphoric acid:
85% phosphoric acid, made by Merck Company, Germany, was used to prepare an acidic solution with a concentration of 30% by weight.
- Arsenic stock solution:
Arsenic stock solution was obtained by adding 0.001 g of arsenic in 1 liter of deionized water.
- Hydrochloric acid:
To adjust the pH of the pollutant solution, normal HCL 0.1 with a purity of 37%, Arman Brand, made in Iran, was used.
- Caustic soda:
To adjust the pH of the pollutant solution to arsenic, normal NAOH 0.1, made by Merck, Germany, was used.
Iodine test materials
-hydrochloric acid:
In this experiment, hydrochloric acid with a purity of 37% and a density of 18.1 gr/cm³, made by Merck Company, Germany, was used.
- Sodium thiosulfate:

In this experiment, Sodium thiosulfate with a purity of 99% and a density of 1.67 gr/cm³, made by Merck Company, Germany, was used.

- Iodine:

In this experiment, Iodine with a purity of 99% and a density of 93.4 gr/cm³, made by Merck Company, Germany, was used

-Potassium iodate:

In this experiment, Potassium iodate with a density of 3.89 gr/cm³, made by Merck Company, Germany, was used.

- Potassium iodide:

In this experiment, potassium iodide with a density of 23.3 gr/cm³, made by Merck Company, Germany, was used.

- Iodine test:

The guideline of Iran's Standard and Industrial Research Institute was used to conduct the iodine test.

- Hydrochloric acid solution - 5% by mass

A milliliter of concentrated hydrochloric acid was added to 550 ml of water and mixed well. A graduated cylinder was used to remove the mentioned volumes.

-Sodium thiosulfate solution- 0.100 N

24.820 g of thiosulfate was dissolved in 75ml ± 25ml of freshly boiled water and 10.01g ± 0.01g of sodium carbonate was added to minimize the decomposition of sodium thiosulfate dissolved bacteria. Then, it was transferred to a volumetric flask and brought to volume.

- Iodine solution 0.100 N ± 0.001 N

12.700g of iodine and 100.19g of potassium iodide were weighted. Then, we mixed them, added 2 to 5 ml of water, and stirred well. While stirring, we continued the increase of water little by little (about 5 ml each time) and up to the volume of 50 to ml 60 ml. The solution was quantitatively transferred to a one-liter volumetric flask and was brought to volume with water. In a standard iodine solution, the mass ratio of iodide to iodine should be 1.5 to 1.

- Potassium iodate solution 0.100 N

Four grams (or more) of potassium iodate of the initial standard type was cooled for two hours at ambient temperature in a desiccator. 3.5667 ± 0.1 mg of dry potassium was dissolved in about 100 ml of water. It was quantitatively transferred to a volumetric flask and brought to volume.

- Starch solution

0.5 g ± 0.1 g of starch was mixed with 5 ml to 10 ml of cold water to make a paste. While stirring the starch paste, 25 ml ± 5 ml of water was added. This mixture was added to one liter of boiling water and boiled for 5 minutes while stirring. Then, two sodium thiosulfate solutions and an iodine solution were standardized for the iodine test.

Measured tools

-Electric furnaces

To carbonize the samples up to a temperature of 1100 C°, an electric furnace model CARBOLITE ELF\6B made in England was used.

-Oven

To dry the glassware, activated carbon from the oven model 3487 of Behdad Company (Labtron) made in Iran was used.

- Magnetic stirrer

A magnetic stirrer was used to mix the samples.

PH meter

ISTEK brand pH meter made in Japan, which includes an electrode to be placed inside the sample to determine the pH, was used.

- Scanning electron microscope device

Scanning Electron Microscope or SEM has a very high-resolution limit due to the use of an electron beam. To examine the morphological condition of the prepared adsorbent particles, the samples were analyzed by SEM.

- ICP (Inductively Coupled Plasma) device

ICP device model Flash EA-1112 series from Spectro Genesis was used to determine the concentration of the sample.

Carbon activation method

Raw material preparation method

The sawdust prepared from the wood of the Juniperus excelsa was washed first by using normal water and then by double-distilled water to remove the soil on the wood. Then, the resulting sawdust was heated in the oven for one hour. After reaching a uniform moisture level, it was kept in a closed container until the next test.

Preparation of carbon and activation with phosphoric acid

The particles obtained from sawdust with dimensions of 125 μm were converted into activated carbon. In this method, in a porcelain plant, three grams of the raw material were mixed with 11 ml of phosphoric acid with a concentration of 30% by weight for 48 hours by a magnetic magnet at a speed of 500 rpm at ambient temperature so the acid was allowed to penetrate the structure of the raw material. The samples were heated at an activation temperature of 600 °C to carbonize and destroy the molecular structure of the raw material and remained at this temperature for one hour. Then, the samples are removed from the oven to cool. The samples were washed several times with boiling distilled water and finally dried in an oven at 100°C for 3 hours.

Iodine number (Iodine test)

This method is based on the three-point graph of adsorption isotherm. The standard iodine solution is mixed with three different masses of activated carbon under certain conditions. The obtained mixtures are filtered to separate the carbon from the iodine solution. The amount of remaining iodine in the filter solution is determined by titration of the results. For each carbon dose, the amount of adsorbed iodine per gram of carbon is calculated and the results are used to draw the adsorption isotherm graph. The amount of adsorbed iodine (in mg) per gram of carbon in the remaining iodine concentration of 0.02 is reported as the iodine number in the graph below. The

concentration of iodine in the standard solution affects the adsorption capacity of activated carbon iodine. Thus, the concentration of iodine should be kept within the normal range. The amount of activated carbon product is obtained by dividing the weight of activated carbon after washing by the amount of raw materials. The total ash percentage was calculated based on the ASTMD 2866-94 standard method. The iodine number, indicating the milligrams of adsorbed iodine per gram of adsorbent, was determined by ASTMD 4607-94. Figure 3 presents the properties of prepared activated carbon.
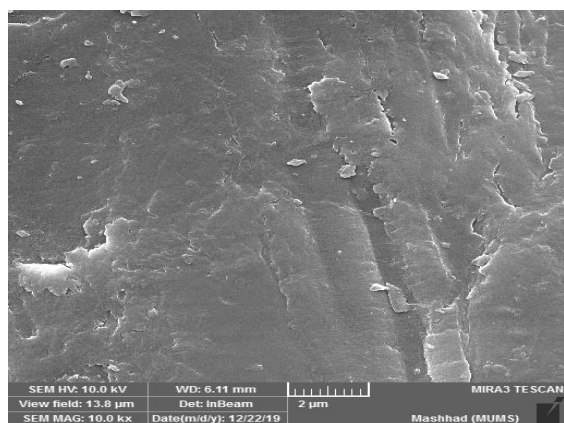
Figure 1- SEM images of sawdust surface before and after activation by phosphoric acid in 2 mµ size
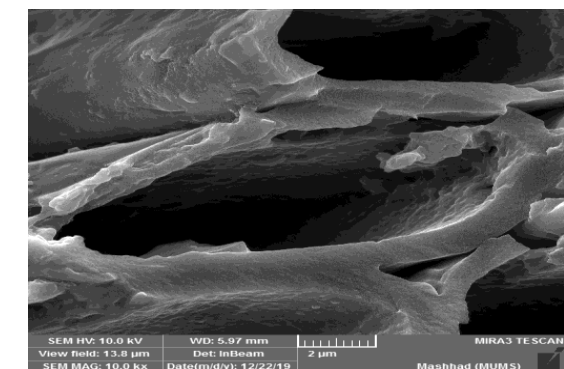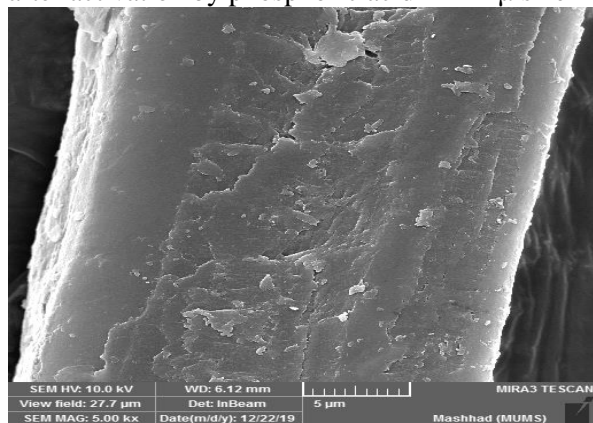
Figure 2- SEM images of the sawdust surface before and after activation by phosphoric acid in the 5 µm size
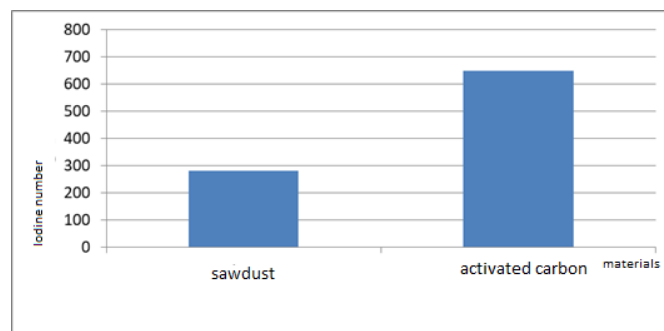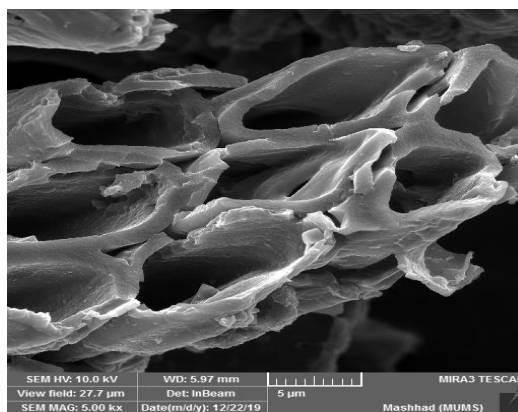
Figure 3. Changes in iodine number before and after activated carbon

Design of experiments

Design Expert software was used to design the experiments and analyze the results. To investigate the effect of each of the three factors including pH, contact time, and adsorbent concentration on the adsorption rate, the experiments were first designed based on the Box-Behnken statistical scheme with three levels for each factor. After the experiments, data were analyzed for each of the desired responses. Table 1 presents the removal percentage and the arsenic concentration values after each stage. Finally, variables were optimized based on the information obtained from the experiments. Table 2 also presents the selection of the range of variables introduced to the software. In this study, which includes 12 steps, according to the variables affected by the software, the percentage of arsenic adsorption was calculated according to the

$$R = \frac{C_o - C_t}{C_O} \times 100$$

formula. R% represents the adsorption rate of metals. C_O and C_t, respectively, are the initial and residual concentrations of metals (mg/liter). The initial concentration of the solution is the same as the concentration of the effluent used in this study, which was 1 ppm.

Table 1. The studied factors along with their relevant levels

Std	Run	Factor 1 A:m gr	Factor 2 B:pH	Factor 3 C:Time h	Response 1 C mgr/litr	Response 2 %R mgr/litr
12	2	0.75	9	4	0.215	78.5
4	3	1	9	2.5	0.296	70.4
3	4	0.5	9	2.5	0.391	60.9
10	10	0.75	9	1	0.468	53.2
8	5	1	7	4	0.53	47
7	12	0.5	7	4	0.646	35.4
6	8	1	7	1	0.724	27.6
5	9	0.5	7	1	0.767	23.3
11	11	0.75	5	4	0.879	12.1
2	7	1	5	2.5	0.924	7.6
1	6	0.5	5	2.5	0.929	7.1
9	1	0.75	5	1	0.96	4

Table 2. Selection of the range of variables introduced to the software

pH	9	7	5
Time (h)	4	5.2	1
Adsorbent value (g)	1	75.0	5.0

RESULTS AND DISCUSSION
Changes in the adsorption rate based on pH
Figure 4 shows the adsorption rate in an adsorbent dose of 0.75 g and the contact time of 2.5 hours based on different pH. Based on the graph, the adsorption rate was better in the alkaline medium. In alkaline pH, the capability to absorb activated carbon is more than in acidic pH. It can be justified in this way that as pH decreases, the hydrogen ion concentration in the medium increases, and its tendency to adsorb with the active carboxyl groups on the surface of the adsorbent, which has a negative charge, increases. For this reason, the hydrogen ion adsorbed on the surface of activated carbon decreases from the level of places available for pollutant adsorption. This result is in line with the result of a study by Alizadeh et al. (2013), who modeled and optimized the removal of copper from water by plantain fruit using the experimental design method. Figures 5, 6, 7, and 8 show the changes in adsorption rate at different pH levels at the lowest and highest levels of adsorbent dose and the lowest and highestcontacttime. Basedonthegraphs, with increasing adsorbent dose and contact time, the rate of adsorption or removal of arsenic from the solution increases.

Design-Expert® Software
Factor Coding: Actual

%R (mgr/litr)
-- 95% CI Bands

X1 = B: pH

Actual Factors
A: m = 0.75
C: Time = 2.5

One Factor
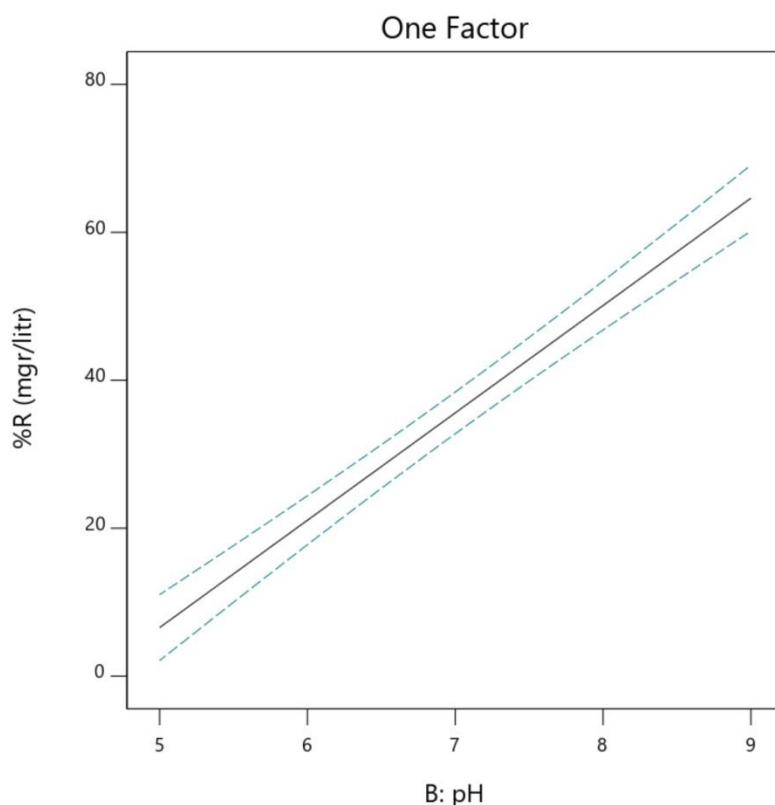
Figure 4. Changes in the adsorption rate based on pH during the contact time of 2.5 hours and 0.75 g of adsorbent
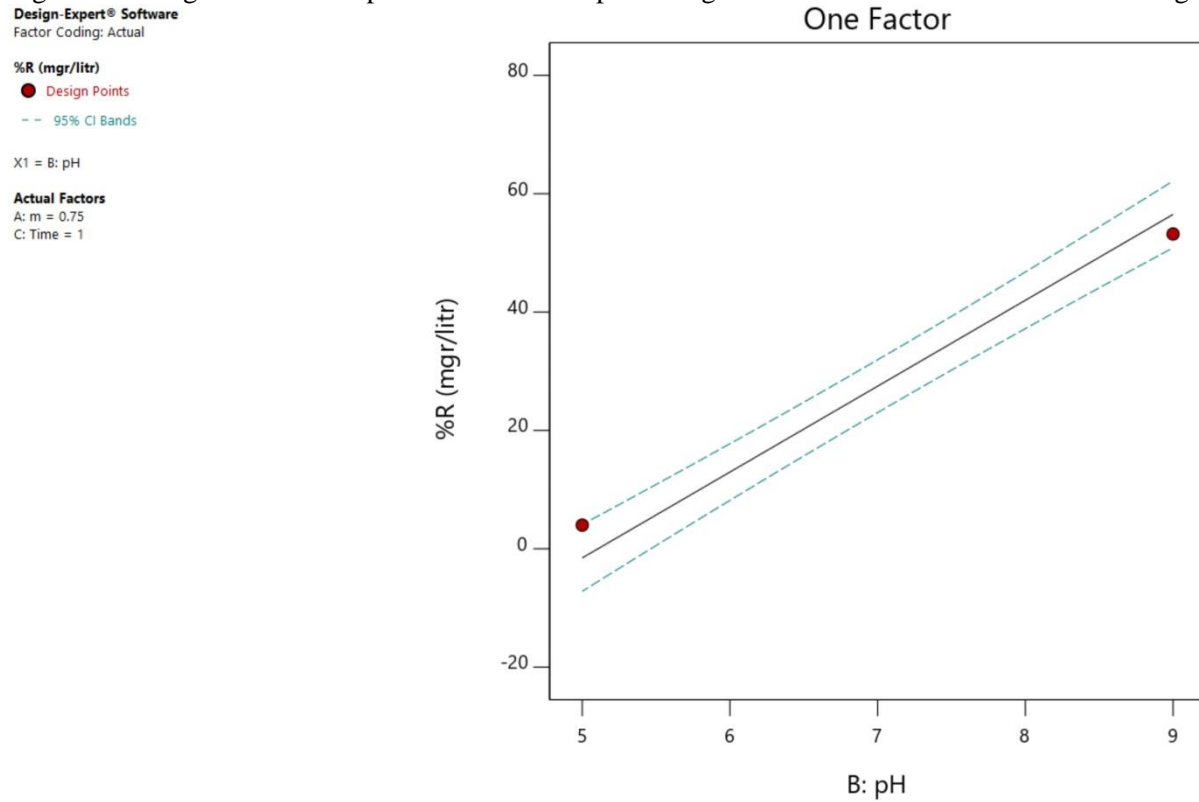
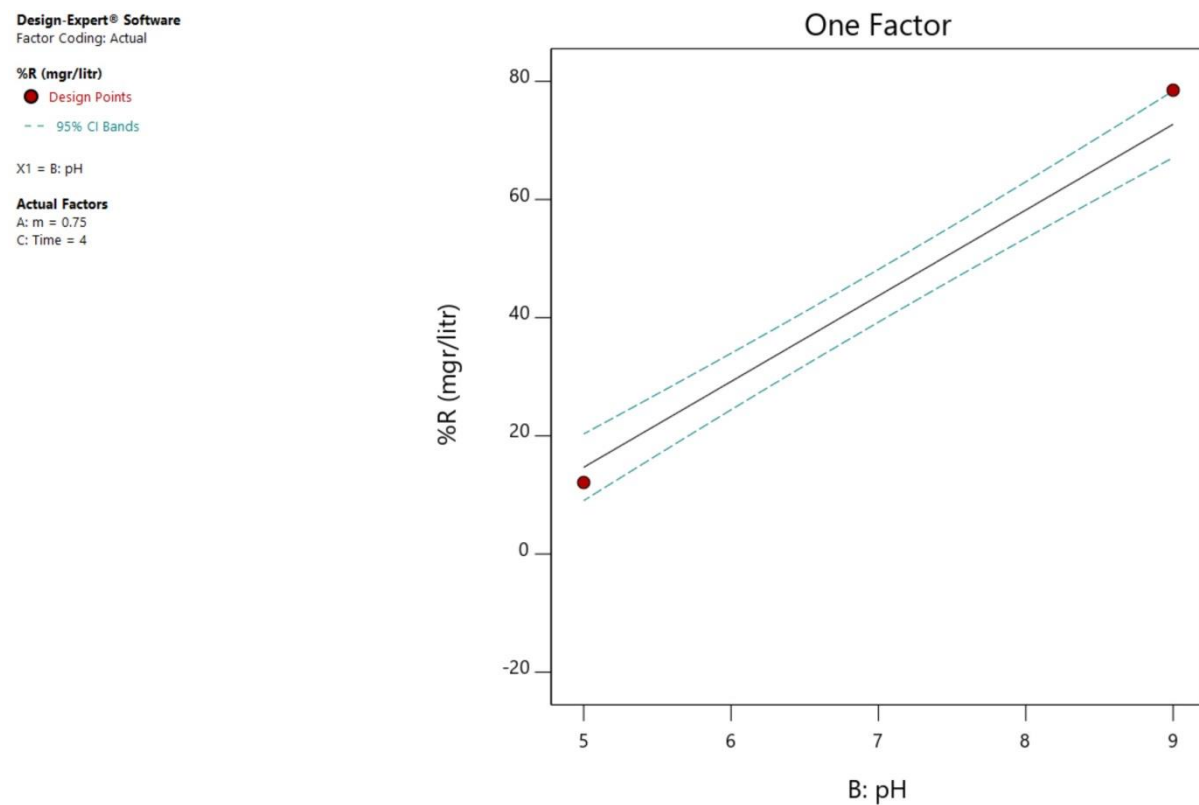Figure 5. Changes in the level of adsorption based on pH in the shortest contact time and 0.75 g of adsorbent

Figure 6. Changes in the adsorption rate based on pH at the maximum contact time and 0.75 g of adsorbent

One Factor
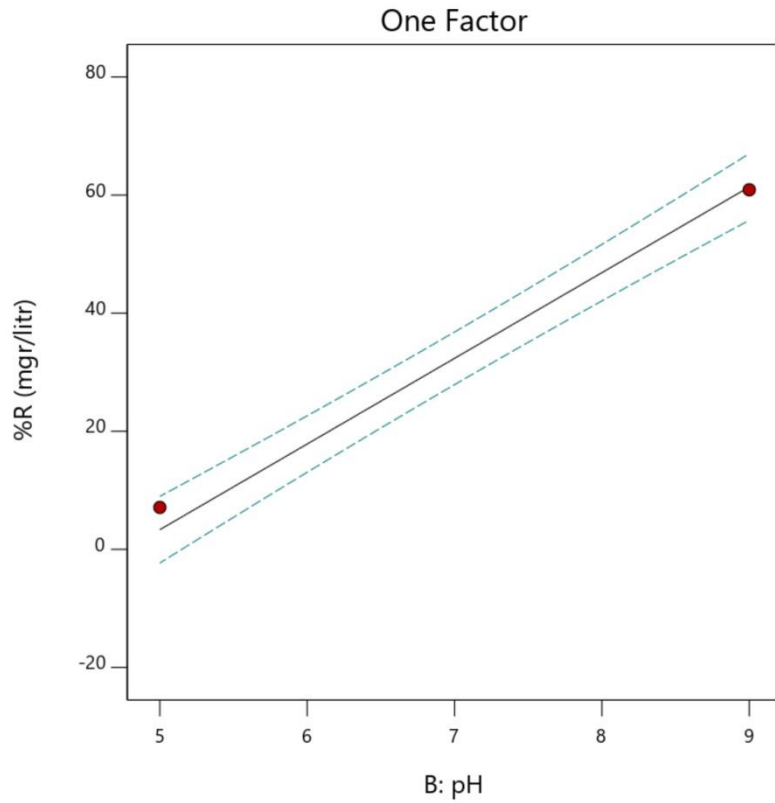
Figure 7. Changes in the adsorption rate based on pH at the contact time of 2.5 hours and the lowest adsorbent

One Factor

Figure 8. Changes in the adsorption rate based on pH at the contact time of 2.5 hours and the maximum adsorbent

Changes in the adsorption rate based on the adsorbent doseBased on Figure 9, the adsorption rate in different doses of adsorbent was drawn at neutral pH and a residence time of 2.5 hours. The results revealed that as adsorbent increases, the rate of adsorption and removal of arsenic increases. This result is in line with the result of a study by Kihani et al. (2013), who investigated the effectiveness of Kiwi wood ash and sawdust in removing Congo red dye from textile effluents. In Figures 10, 11, 12, and 13, graphs were drawn based on the maximum and minimum dose and residence time.
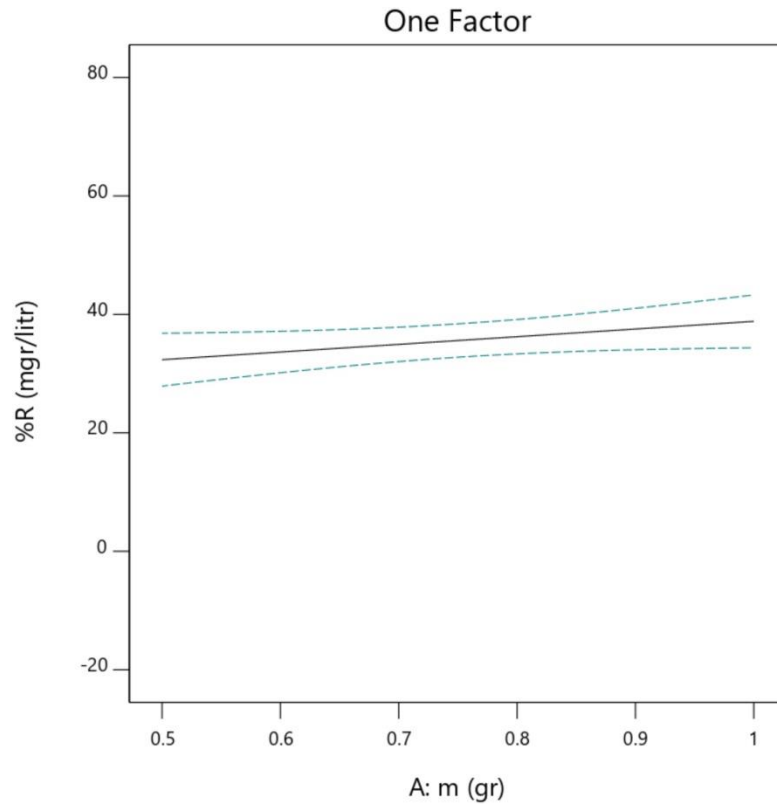
Figure 9. Changes in adsorption rate based on the adsorbent dose in contact time of 2.5 hours and neutral pH
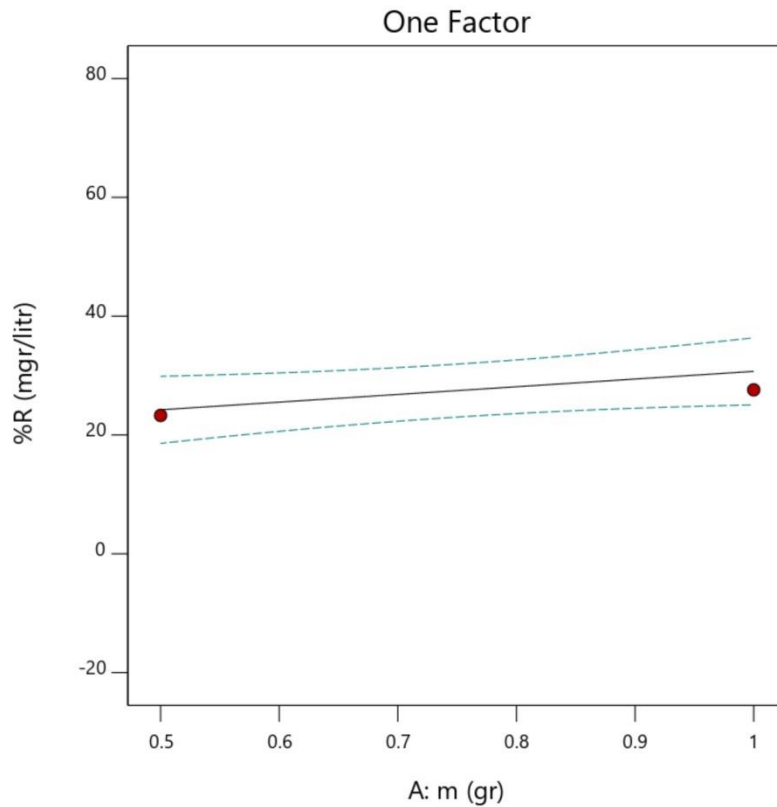
Figure 10. Changes in adsorption rate based on the adsorbent dose in the minimum contact time and neutral pH
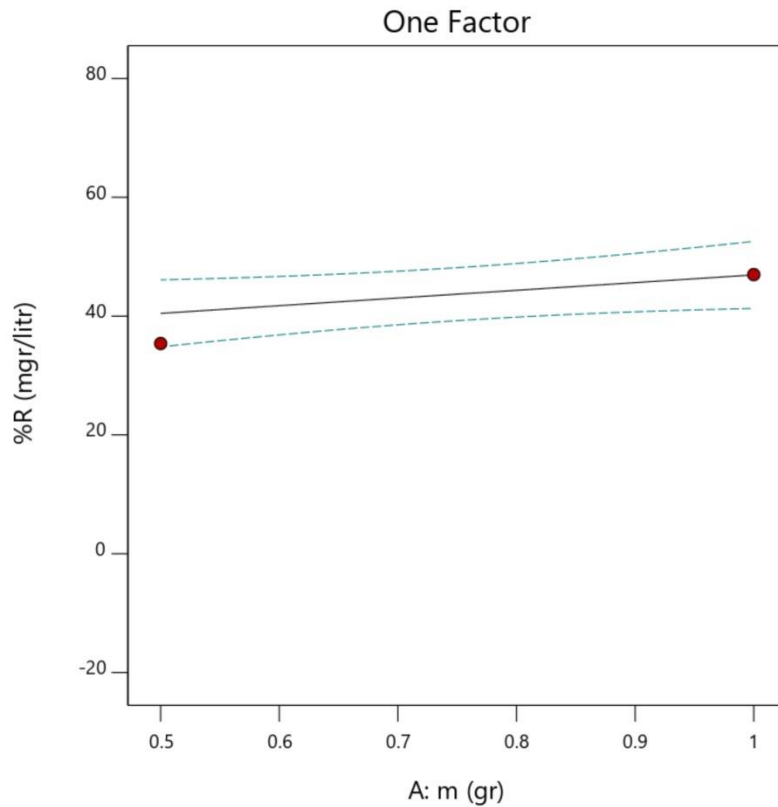
Figure 11. Changes in adsorption rate based on the adsorbent dose in the maximum contact time and neutral pH
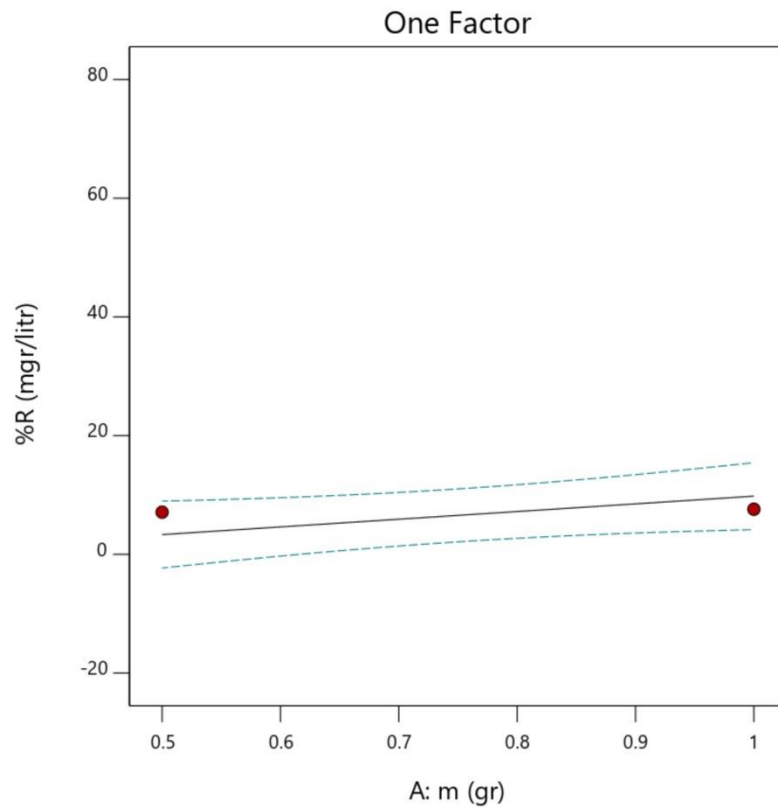
Figure 12. Changes in adsorption rate based on the adsorbent dose in contact time of 2.5 hours and neutral pH
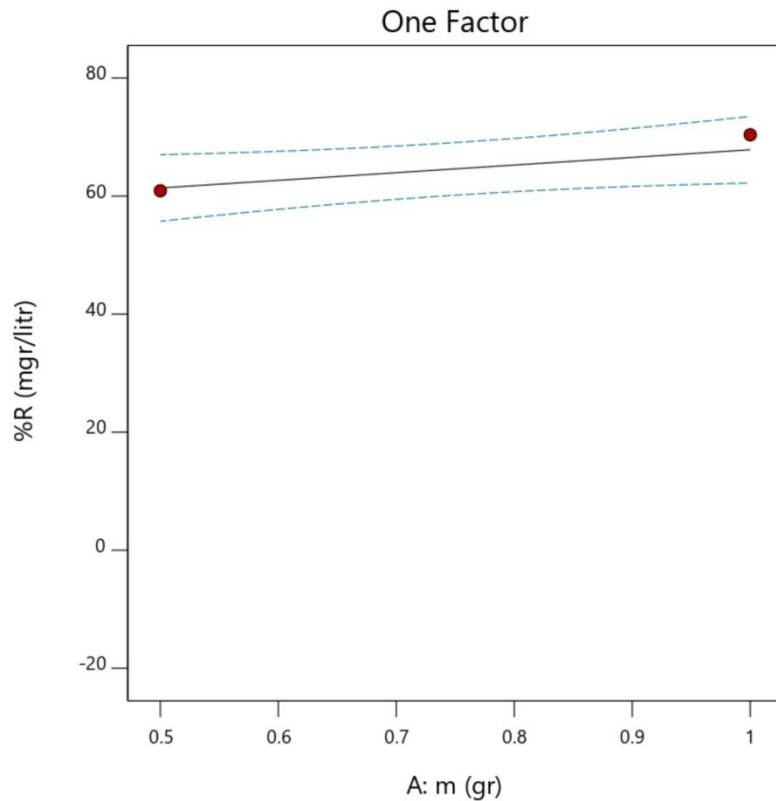
Figure 13. Changes in adsorption rate based on the adsorbent dose in contact time of 2.5 hours and alkaline pH

Rate of adsorption changes based on contact time
Based on Figure 14, the arsenic adsorption rate has been drawn at different contact times at neutral pH and the dose of 0.75 g. As the contact time increased, the adsorption rate also increased. In other words, due to the high porosity of the prepared adsorbent, more effluent pollution will be adsorbed on the surface of the adsorbent over time. This result is in line with the result of a study by Heibati et al. (2010), who evaluated the activated carbon prepared from walnut wood in the removal of azo dyes in textile effluents and determined Acid Red 18 isotherms. Based on Figures 15, 16, 17, and 18, it can be concluded that the maximum level of adsorption was obtained by increasing the adsorbent dose and changing the state from acidic to basic.

Figure 14. Changes in adsorption rate based on residence time in 0.75 g adsorbent dose and neutral pH

Figure 15. Changes in adsorption rate based on residence time in 0.75 g adsorbent dose and acidic pH
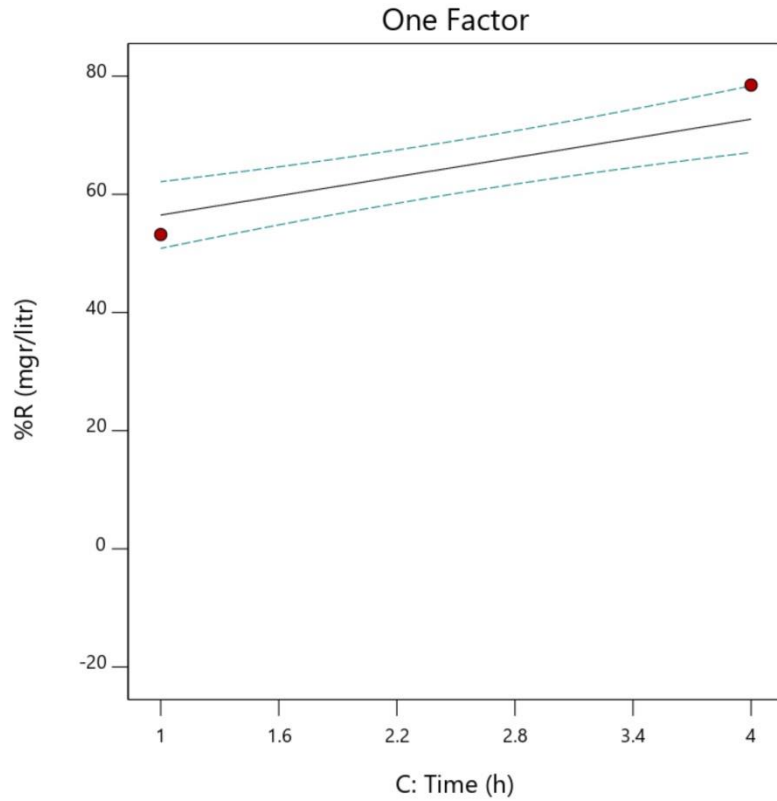
Figure 16. Changes in adsorption rate based on residence time in 0.75 g adsorbent dose and alkaline pH

Figure 17. Changes in adsorption rate based on residence time in the minimum adsorbent dose and neutral pH
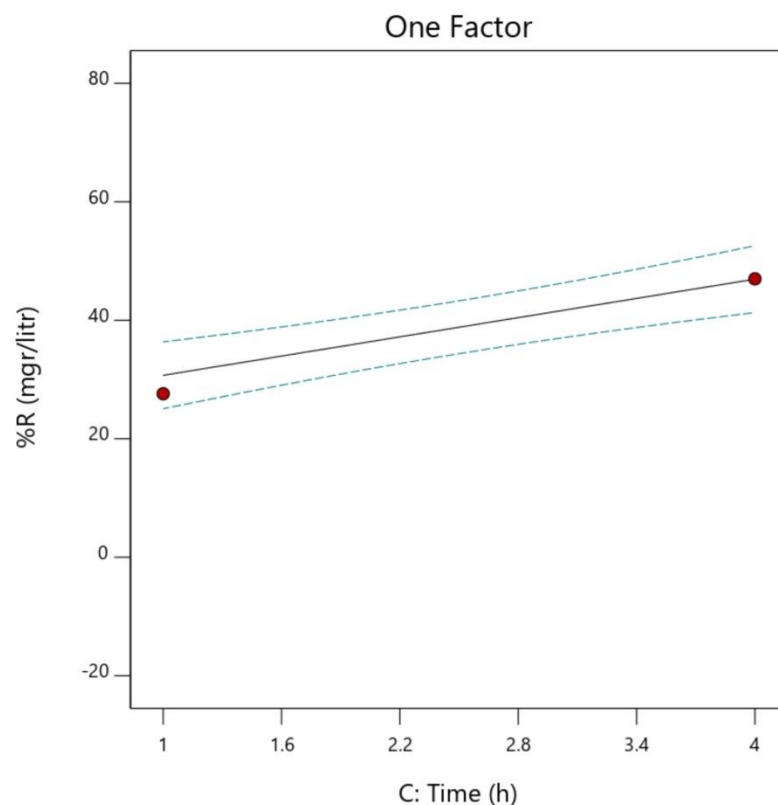

Figure 18. Changes in adsorption rate based on residence time in the maximum adsorbent dose and neutral pH

General representation of changes in adsorption with all three independent variables

Figure 19 shows the schematic of the changes in the adsorption rate at neutral pH and the residence time of 2.5 hours and 0.75 grams of adsorbent dose. The adsorption rate increased with the increase of independent variables, and with the decrease of all three variables, the adsorption rate of arsenic decreased, as shown in Graphs 20 and 21.
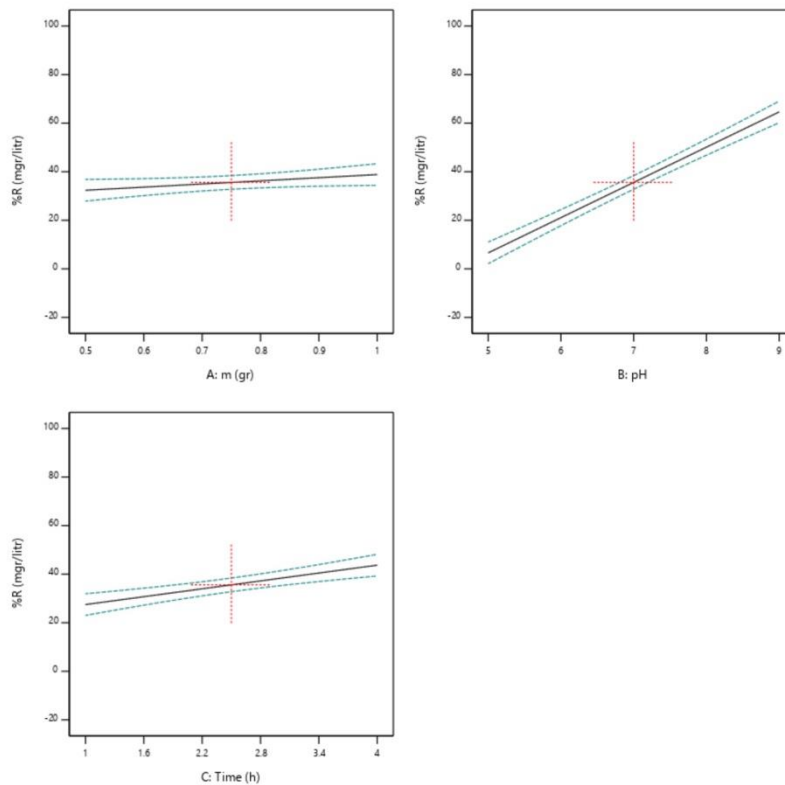
Figure 19- General changes in adsorption rate based on the residence time of 2.5 hours, adsorbent dose, and pH
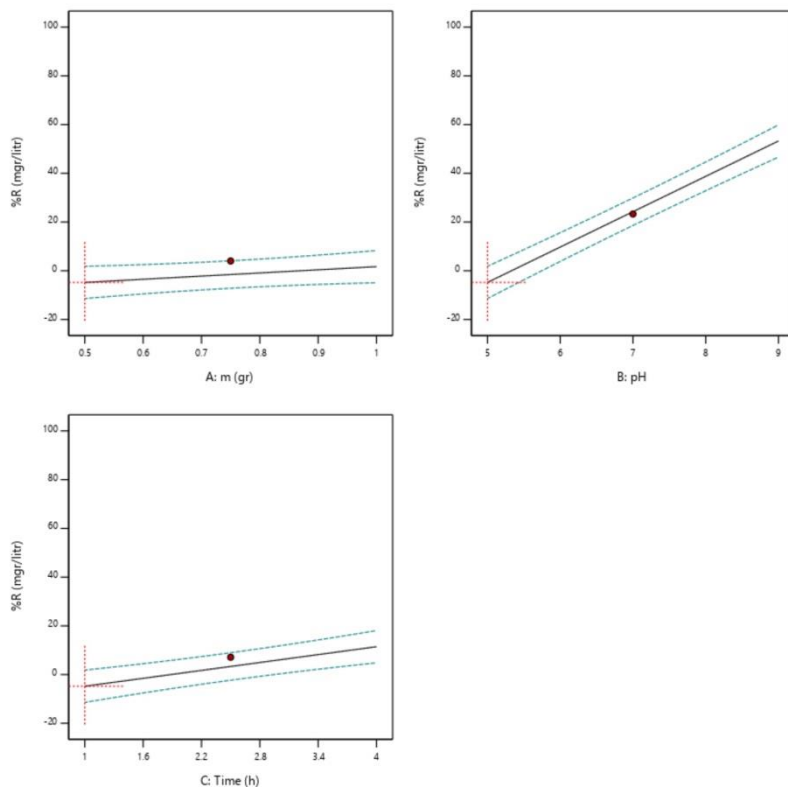
Figure 20- General changes in adsorption rate based on minimum residence time, adsorbent dose, and pH
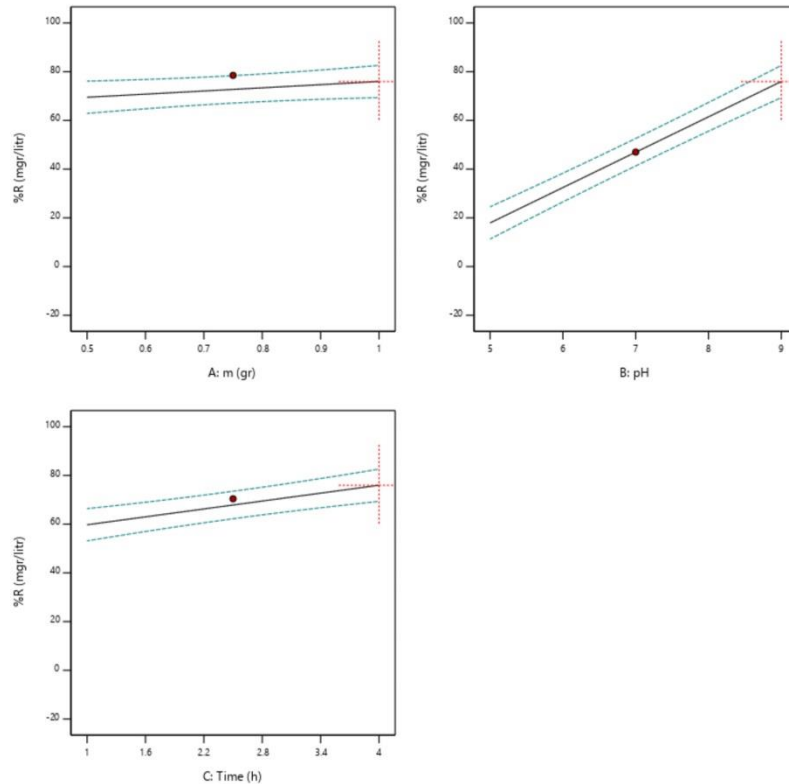
Figure 21- General changes in adsorption rate based on maximum residence time, adsorbent dose, and pH

The results of examining the interaction of variables on adsorption rate

In Figure 22, the interaction effect of pH, residence time, and the adsorbent dose on the adsorption of the pollutant concentration was observed. The residence time is 2.5 hours. As observed, with the increase in pH and the adsorbent dose, the pollutant concentration decreased and the adsorption rate increased. Figures 23 and 24, respectively, show the time it took to reach the minimum and maximum values, and the changes in the adsorption rate were observed. With the increase of time, the adsorption rate increases.
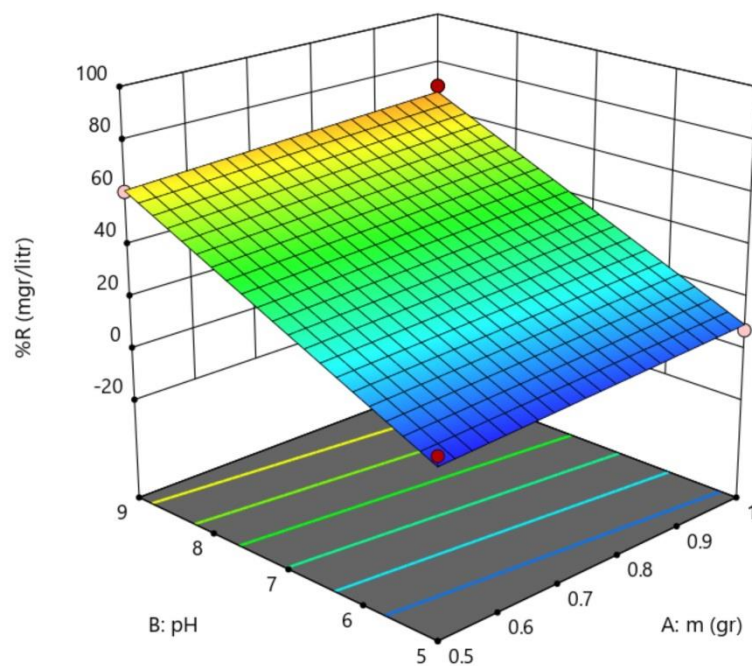
Figure 22. Changes in the adsorption rate based on pH and adsorbent dose at the residence time of 2.5 hours
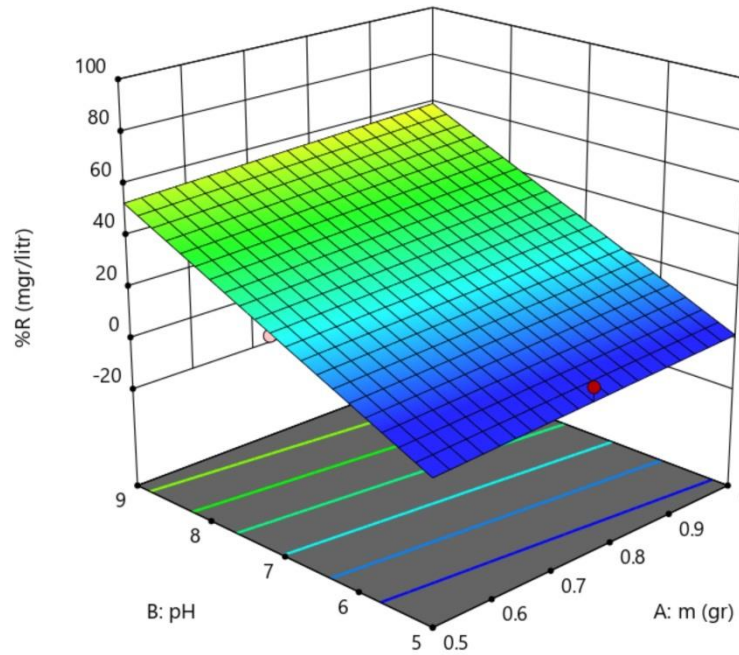
Figure 23. Changes in the adsorption rate based on pH and adsorbent dose at the minimum residence time
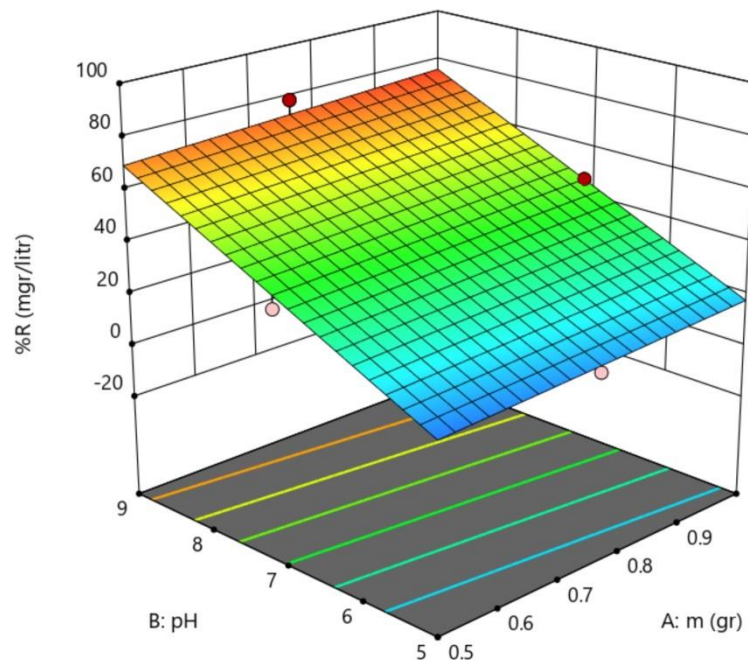
Figure 24. Changes in the adsorption rate based on pH and adsorbent dose at the maximum residence time

Optimization

Numerical optimization of Design Expert software was used to determine the desirable values in the tested range. The desirable conditions obtained by the software to maximize the rate of adsorption by the adsorbent prepared based on the factors of pH, contact time, and the adsorbent rate included 73 different conditions. The best of them (based on the highest desirability value) are listed in Table (3). The value of each response in the desirable conditions is predicted by the software. To examine and confirm the correctness of the model and the obtained desirable values, the validation test in Table 4 was prepared with

the desirable conditions predicted by the software. The values obtained in the experiment are very close to the values predicted by the software.

Table 3. Desirable results of effective parameters

Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:m	is in range	0.5	1	1	1	3
B:pH	is in range	5	9	1	1	3
C:Time	is in range	1	4	1	1	3
%R	maximize	4	78.5	1	1	3

Solutions ▼ 📍 Starting Points

Solutions

73 Solutions found

Number	m	pH	Time	%R	Desirability	
1	1.000	9.000	4.000	75.967	0.966	Selected
2	1.000	9.000	3.984	75.881	0.965	
3	0.993	9.000	4.000	75.872	0.965	
4	0.982	9.000	4.000	75.733	0.963	
5	0.999	9.000	3.951	75.696	0.962	
6	0.976	8.999	4.000	75.646	0.962	
7	1.000	8.972	4.000	75.566	0.961	
8	1.000	9.000	3.906	75.460	0.959	
9	0.960	9.000	4.000	75.448	0.959	
10	0.956	9.000	4.000	75.386	0.958	
11	1.000	8.980	3.940	75.350	0.958	
12	0.951	9.000	4.000	75.333	0.957	

Table 4. The responses related to the desirable conditions predicted by the software and the actual result of the experiment

Response	Software value	Experiment value	Error percentage
result			
Adsorption rate	75.967	78.5	3.4%

CONCLUSION

The effects of pH, contact time, and adsorbent dose on pollutant separation were studied in arsenic pollutant

adsorption experiments. The maximum rate of pollutant adsorption was obtained in desirable conditions in 4 hours, alkaline medium, and with one gram of adsorbent. Also, the error value between the experimental and numerical methods was about 3.4%, indicating the accuracy of the test. In this study, based on the obtained results and by examining the morphological and structural properties, it is concluded that the activated carbon prepared from the sawdust of Juniperus excelsa and phosphoric acid can be used as an effective adsorbent in the removal of effluents containing metals such as arsenic.

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