Evaluation of Rice Husk Biochar Modified with Magnesium Oxide in Adsorbing Heavy Metals from Tannery Wastewater

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Tannery wastewater contains heavy chemicals that are dangerous to the environment, as well as to human health. Lead, cadmium, and chromium are known to cause nervous disorders, cancer of the lung, skin diseases, and sudden death. The removal of heavy metals from the ecosystem is a global concern, and many methods like electroplating, ultrafiltration, and reverse osmosis, have recorded success in removing heavy metals from wastewater. However, they are expensive and require sophisticated technology, thus necessitating the need to develop cheap and affordable adsorbents for adsorbing heavy metals from the wastewater. Biochar derived from the agrowaste materials and modified with nanoparticles, such as magnesium oxide (MgO) have high surface area, cation exchange capacity, and diverse functional groups, making it suitable for adsorbing heavy metals and other pollutants from wastewater. For this reason, rice husk and MgO were pyrolyzed at 500 ºC, at a residence time of two hours to produce an adsorbent (RHBMgO@500) for adsorbing lead, cadmium, and chromium from the tannery wastewater. An Atomic Absorption Spectroscopy (AAS) was used to determine the removal efficiency of RHBMgO@500 for lead, cadmium, and chromium, and the results revealed the removal efficiency for lead as 43.09% to 93.518%; for cadmium as 19.17 % to 86.30%, and for chromium as 85.793 % to 100.00 %. Its adsorption capacity for lead, cadmium, and chromium were 0.43mg/g to 0.858mg/g, 0.00028mg/g to 0.0027mg/g, and 58.073mg/g to 232.200mg/g respectively. For the optimization, the Ramp graph function from the response surface methodology (RSM) based on the central composite design (CCD) showed that temperature, contact time, pH, and adsorbent dosage are set to 41 ºC, 45 minutes, 6, and 4g, respectively, the removal efficiency for lead, cadmium, and chromium can be 62%, 62%, and 100%, respectively.
Keywords: Chromium, Tannery, Adsorbent, Biochar, Adsorption, nanoparticles

Introduction

The impact of industrial waste effluent on the ecosystem is quite alarming globally. Almost all industrial wastewater contains heavy metals and other micro-pollutants that are capable of causing environmental degradation and loss of biodiversity. Heavy metals are non-biodegradable, and when adsorbed into the body system, living tissues of animals, or plant tissues, undergo bioaccumulation and later reach biomagnification, which are capable of causing stunted growth, nervous disorders, lung and skin cancer, skin irritation, and kidney failure. Incessant discharge of untreated industrial wastewater into the ecosystem system is responsible for the contamination of groundwater and surface water bodies, scarcity of water, and water related illness, such as diarrhoea and cholera (Gwenzi *et al*., 2017; Ullah & Rahman, 2024). Jayakumar *et al*. (2021) reported that 485, 000 die every year as a result of drinking contaminated water or using it for domestic purposes.

Tannery industry requires huge amount of quality water and generates huge amount of wastewater water across the globe annually. To process one kilogram of hides and skin into leather, about 35 to 40 litres of water is needed (Fouda *et al*., 2021). Tannery wastewaters contain heavy metals such lead, manganese, copper, cadmium, chromium, arsen and zinc (Kpega *et al*., 2026). Heavy metals are threat to the environment and human being, which made World Health Organization (WHO) to limit the permissible level of heavy metals in the drinking water to the barest minimum. Heavy metals can enter the body system through the drinking water, or the consumption of agricultural products. Heavy metals, such as lead, chromium, manganese when ingested into the body cause nervous disorder, visual impairment, skin disease, stunted growth, cancer, and sudden death (Kpega *et al*., 2026). One of the common heavy metals in the tannery wastewater is chromium because chromium sulphate is one of the chemicals used in the tannery industry. It is obvious that heavy metals are threat to environmental and public health, therefore, it is important to explore sustainable, cost-effective means of treating tannery wastewater. However, there are different methods of treating wastewater, like reverse osmosis, ultrafiltration, gravity separation, anaerobic digestion, electrodialysis, and ion exchange, but they are expensive and requires high level technical knowhow, thus making it difficult for many people, or industries to have access to it (Naseem & Durrani, 2021). This resulted in disposing untreated industrial wastewater into the environment, thus causing environmental pollution and degradation of the water bodies, and air.

For this reason, many researchers have developed adsorbent using agrowaste materials and nanoparticles (magnesium oxide, iron (iii) chloride, kaolin, zinc oxide) to adsorb heavy metals from the wastewater, and many of the researchers recorded success. An adsorbent (biochar) produced from the agrowaste materials and modified with the nanoparticles have high diverse functional groups, high surface area, cation exchange capacity, porosity which made them suitable for handling micro-pollutants in the wastewater.

In this research work, rice husk, and magnesium oxide were used to produce an adsorbent at a pyrolytic temperature of 500 ^oC. The experimental design was conducted using response surface methodology based on the central composite design (CCD), where four factors (contact time, pH, adsorbent dosage, and temperature) were simultaneously investigated on the ability of adsorbent (RHBMgO@500) to adsorb lead, cadmium, and chromium.

Materials and Methods

Collection and characterization of tannery wastewater

Tannery wastewater was collected from the Nigerian Institute of Leather and Science Technology (NILEST), Zaria, Kaduna State. The wastewater was analysed with Atomic Absorption Spectroscopy (AAS) to determine the heavy metals, such as lead, chromium, cadmium, copper, and manganese.

Collection, preparation and production of biochar

The rice husk used to prepare the biochar was collected from the National Agricultural Extension and Research Liaison Services, Ahmadu Bello University, Zaria, Rice Mill. After the collection, the rice husk was cleaned and washed with distilled water to remove impurities. Also, a magnet was used to inspect the biomass to remove the magnetic materials. The rice husk was dried, crushed with a hammer mill, and further reduced to finer particles with a ball mill. The adsorbent, RHBMgO@500, was produced by mixing 300 g of rice husk with 60 g of magnesium oxide and 400 ml of distilled water and thoroughly mixed with a mechanical mixer. Thereafter, the mixture was placed in a pyrolyzer and pyrolyzed at 500 ^oC at a residence time of 2 hours. Rice husk has a high content of lignin, silicate, hemicellulose, cellulose, high pore and surface area, and active sites, which make it suitable for producing biochar for adsorbing heavy metals. A temperature above 500 ^oC caused biochar to lose functional groups through the process of decarboxylation, deoxygenation, and dehydrogenation (Che Pa *et al*., 2024).

Fourier Transform Infrared (FTIR) was used to characterize the biochar prepared at different temperatures to ascertain the functional groups in each biochar. The FTIR helped to determine the level of functional groups on the adsorbent.

Experimental design

The batch adsorption experiment was designed with Design of Expert Full Version13.0.1.0, based on the Response Surface Methodology (RSM), and Central Composite Design. Four independent factors: pH of the solution with five levels (2 to 12), temperature with five levels (30 to 45), contact time with five levels (20 to 120 minutes), and adsorbent dosage with five levels (2 to 8) were used to conduct the removal efficiency, and adsorption capacity of adsorbent (RHBMgO2500) for lead, cadmium and chromium. The pH of the wastewater was adjusted with the aid of sodium hydroxide, hydrogen chloride, and pH meter. The experiment was randomized, with 30 runs and no blocking. The data were subjected to analysis of variance (ANOVA), and it indicated that quadratic model is suitable for analysing the results.

Batch adsorption experiment

In the batch adsorption experiment, the following input variables, such as pH solution, adsorbent dosage, contact time, and temperature, were varied to determine their effect on the adsorption capacity and removal efficiency of the adsorbent on lead, cadmium, and chromium. A 100 ml of wastewater was used throughout the experiment, while the adsorbent dosage was varied. The volume of wastewater was measured using a cylindrical glass and poured into a conical flask, and a measured sample of an adsorbent was introduced into the wastewater. Thereafter, the conical flask was placed on the magnetic stirrer/heater. The heating temperature was adjusted, and the stirrer speed was adjusted and allowed to reach the specified time considered for the experiment. After that, the wastewater was filtered through filter papers, followed by digestion. The digestion involved the addition of nitric acid and perchloric acid into the extracted wastewater, followed by heating using digestion box. The heating continues until whitish fumes started coming out from the conical flask, then the samples were removed from the digestion box. After the samples were removed from the digestion box, distilled water was added to the samples and thoroughly shook.

After digestion, the samples were taken to the AAS machine to determine the concentration of the remaining heavy metals (lead, cadmium, and chromium). The final reading from the AAS machine, and the initial concentrations of each heavy metals were used to calculate the removal efficiency, and adsorption capacity of the adsorbent for lead, cadmium, and chromium.

Computation of removal efficiency, and adsorption capacity of RHBMgO@500

The computation of removal efficiency, and adsorption efficiency of adsorbent for chromium, lead, and cadmium was determined using equations 1 and 2, respectively.

$$\text{Removal efficiency (\%)}, R = \frac{C_o - C_t}{C_o} \times 100 \ldots\ldots 1$$

$$\text{Adsorption capacity } \left(\frac{mg}{g}\right), q_t = \frac{(C_o - C_t) \times V}{m} \ldots .2$$

C_o (mg/L) = initial concentration of adsorbate
C_t (mg/L) = adsorbate concentration at the equilibrium
m (g) = mass of biochar added to the solution
R (%) = percentage of removal of adsorbate by the adsorbent
q_e(mg/g) = adsorption capacity of biochar at equilibrium
V (L) = Volume of wastewater

Results and Discussion

Characterization of tannery wastewater

The results of heavy obtained from the characterization tannery wastewater presented in Table 1. From the results presented in Table 1**,** the concentrations of heavy metals: lead, chromium, copper, manganese, and cadmium are beyond the permissible limit stipulated by the WHO, and Food for Agriculture Organization (FAO). The WHO permissible limit of lead, chromium, copper, manganese, and cadmium were 0.05 ppm, 0.05 ppm, 2.00 ppm, 0.08 ppm, and 0.003 ppm, respectively. Based on the WHO standard, the concentration of heavy metals in the tannery wastewater are far beyond the permissible limit specified by the WHO (Geremew & Tekalign, 2017).

Table 1: The chemical properties of tannery wastewater

S/No	Heavy metals	Concentrations (ppm or mg/L)
1	Lead	3.934
2	Chromium	4645.76 (29.036 x160)
3	Copper	1.311
4	Manganese	0.883
5	Cadmium	0.073

Functional groups on the adsorbent (RHBMgO@) produced at 500 °C

The functional groups in the biochar (Rice Husk + MgO) produced at 500 °C is presented in Figure 1. The ability of biochar to adsorb micro-pollutants in the wastewater is due to the presence of diverse functional groups on its surface. The biochar has functional groups such as hydroxyl groups, carboxylic groups, aromatic groups, C=O stretching of hemicellulose, C-OH, N-H binding, and O-H stretching, which in agreement with findings of (Kuang *et al*., 2023). (Kuang *et al*. (2023) stated that adsorption intensities of biochar produced within the range of 300 °C to 500 °C declined from 2800 to 3000 cm^{-1}. Studies revealed that an increase in the pyrolysis temperature resulted in a decrease or disappearance of -C-O-C stretching in lignocellulose, and -CH stretching associated with lignin, and cellulose (Wang *et al*., 2021).

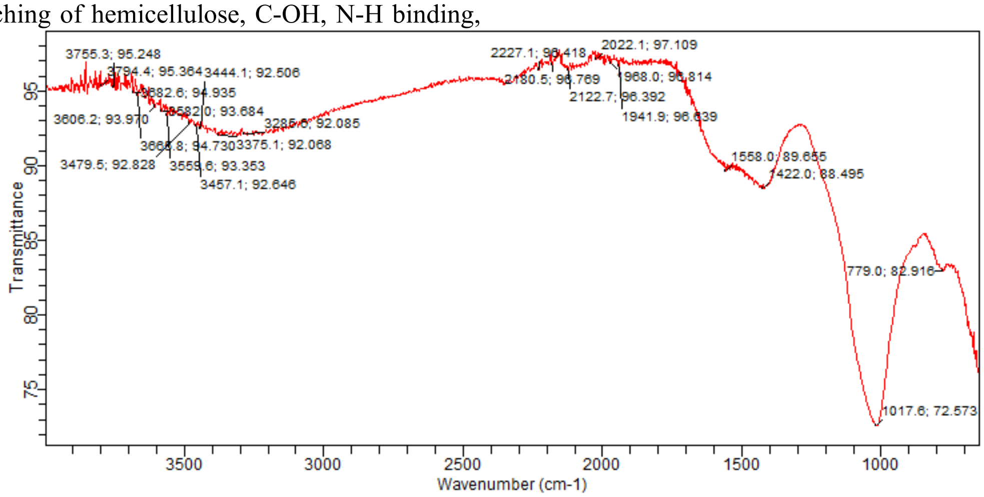

Figure 1: The availability of functional groups in the biochar produced at 500 °C

Removal efficiency of RHBMgO@500 for lead, cadmium, and chromium

The biochar was produced from the pyrolysis of rice husk and magnesium oxide at 500 ºC, at a residence time of 2 hours. A response surface methodology (RSM) based on the Central Composite Design (CCD) was used to investigate the effect of input variables: temperature, contact time (B), pH (C), and adsorbent dosage (D) on the removal efficiency of RHBMgO@500 on the lead, cadmium, and chromium.

The removal efficiency of RHBMgO@500 for the lead ranged from 43.09% to 93.518%; for cadmium it ranged from 19.178% to 86.301%, and for the chromium it ranged from 85.79% to 100%. The contour plots of the removal efficiency of the adsorbent for lead, cadmium and chromium are shown in Figures 2, 3, and 4, respectively.

In Figure 2, the colour changed from blue to light yellow which signified tat as the temperature increased, and contact time increased, the removal efficiency of RHBMgO@500 for lead increased, though slowly. The contour lines are widely spaced, and this indicates that the changes in the removal efficiency are not rapid.

The removal efficiency of RHBMgO@500 for lead varied from 43.0923% to 93.518%. The adsorbent dosage and the pH of the solution had a significant effect on the adsorption efficiency of RHBMgO500@500 for lead. The variation of pH solution affected the surface charge of the adsorbent and the degree of ionization of the adsorbing heavy metal ions (Am *et al*., 2017). The removal efficiency of banana biochar for lead, as reported by Amin *et al*. (2017) ranged from 50 to 70%. The pH of the tannery wastewater was varied from 2 to 12, but the range of pH values that gave maximum removal of lead falls within 5 to 7.8. This finding agreed with the findings of (Amin *et al*., 2018), in which they stated that at acidic conditions, the removal of lead was constant at an acid pH range of 3.5 to 5, and after pH 7, the removal of lead started decreasing. Equally, Cucumis melon peel biochar removal efficiency for lead is 98.55% (Manjuladevi *et al*., 2018), Sanka (2020) recorded removal efficiency of 90% for lead using rice husk carbonized at 600 ºC, and 35% using corn husk carbonized at 600 ºC.

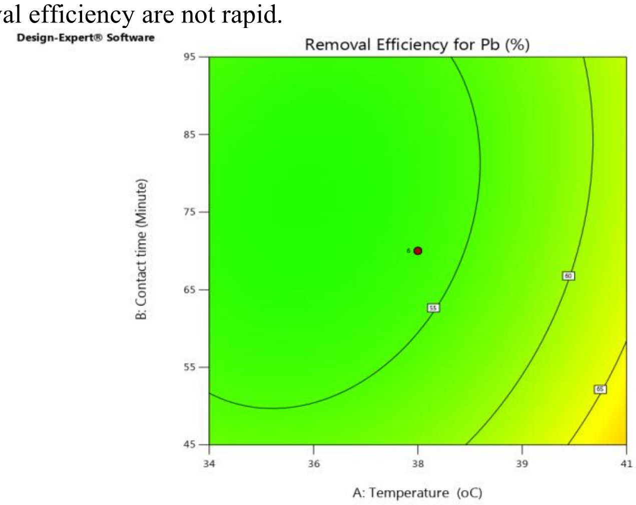

Figure 2: The contour plot showing the removal efficiency of RHBMgO@500 for lead under the effect of temperature and contact time

In Figure 3, the removal efficiency RHBMgO@500 for cadmium varied from 19.1781% to 86.3014%. The four input factors integrated to find the efficiency of RHBMgO@500 for removing cadmium from the tannery wastewater were temperature, contact time, pH, and adsorbent dosage, but pH and adsorbent dosage played a significant role in the removal efficiency of RHBMgO@500 for cadmium. At high acidic solution, the adsorption of cadmium was low, but as the pH value increased, the removal of cadmium increased. The favourable pH value for removing cadmium is between 5 to 7.5, and the adsorbent dosage falls within 4 to 7g. Saeed *et al*. (2021)used kenaf biochar modified with magnesium oxide, and pyrolyzed at a temperature of 550 ºC, and recorded an adsorption efficiency of 69.82% for cadmium. Also, Cucumis melon peel biochar removal efficiency of 97.96 % for cadmium (Manjuladevi *et al*., 2018).

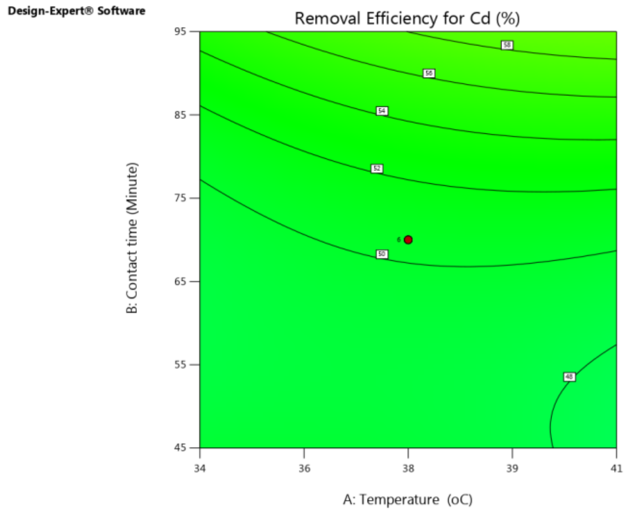

Figure 3: The contour plot showing the removal efficiency of RHBMgO@500 for cadmium under the effect of temperature and contact time

In Figure 4, there was no colour variation, which signified that the removal efficiency of RHBMgO@500 for chromium is maximum. The colour is uniform and warmer, which implies that the removal efficiency falls within 98% to 100%. The removal efficiency of RHBMgO@500 for chromium is between 85.793 to 100%. The contact time favourable for the high removal efficiency is between 55.8 minutes to 85 minutes, the adsorbent dosage is between 4 to 6 g, and the pH values were between 6 to 8. This finding agreed with the findings of (Peng *et al*., 2021) who reported 100% removal of chromium from the wastewater by using biochar under the following reaction conditions: reaction time of 60 minutes, reaction temperature of 90 ^{o}C and adsorbent dosage of 3g. Sanka (2020) achieved 65% removal efficiency for chromium using carbonized rice husk at 600 ^{o}C and 20% removal efficiency for chromium using carbonized corn husk at 600 ^{o}C.

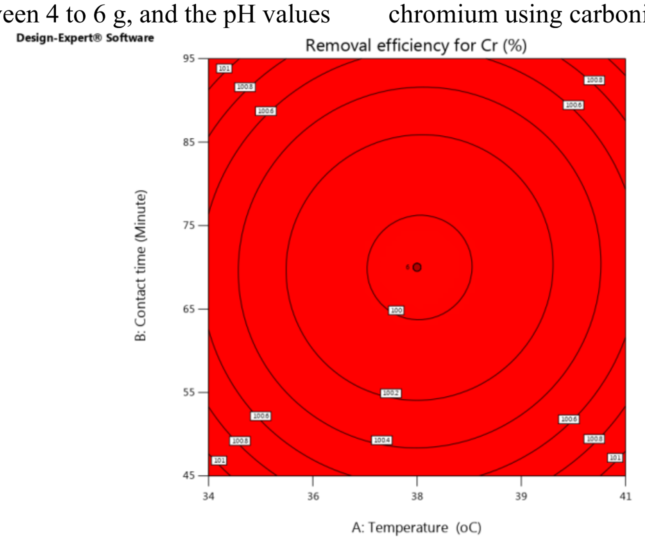

Figure 4: The contour plot showing the removal efficiency of RHBMgO@500 for chromium under the effect of temperature and contact time

Adsorption capacity of RHBMgO@500 for the lead, cadmium and chromium

The adsorption capacity of the adsorbent, RHBMgO@500 for lead, cadmium, and chromium are shown in Figures 5, 6 and 7.

By examining Figure 5, it is obvious that the colour changes as the temeprature and contact time increased, but the wider spaces existed between the the contour lines suggests that the adsorption capacity of the RHBMgO@500 for lead does change rapidly as the independent variables increased. The adsorption capacity of RHBMgO@500 for lead ranged from 43.007 to 85.8031 mg/g. The actual factors that affected the adsorption capacity of RHBMgO@500 for lead are the adsorbent dosage, and the pH solution of the tannery wastewater. The adsorption capacity of RHBMgO@500 for lead ranged from 0.01436 to 0.10715 mg/g.

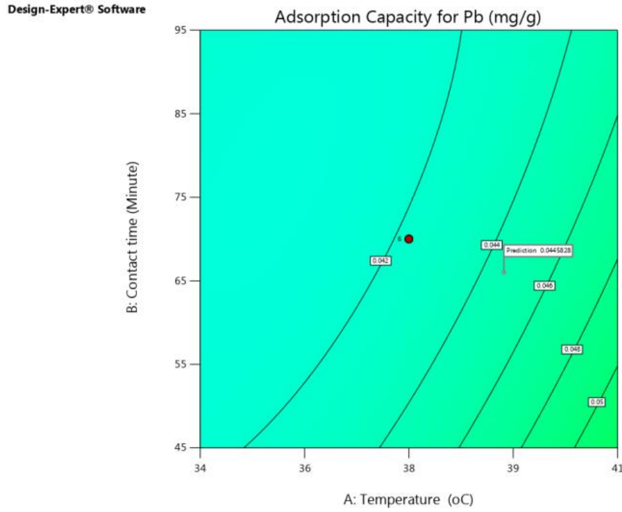

Figure 5: The contour plot showing the effect of temperature and contact time on the adsorption capacity of RHBMgO@500 for lead

In Figure 6, there was no colour variation in the contour plots showing the effect of temperature and pH on the adsorption capacity of RHBMgO@500 for chromium. The contour lines are elliptical, which indicates that the independent variable had a significant interaction on the adsorption capacity of RHBMgo@500 for chromium, and the center of the ellipse represents the optimum adsorption capacity for chromium. The adsorption capacity of RHBMgO@500 for cadmium varied from 0.00028 mg/g to 0.0027 mg/g. The optimum pH values varied from 5 to 8, and the actual factors that influence the adsorption capacity of RHBMgO500 for cadmium were the pH of the solution and the adsorbent dosage. According to Chen *et al*. (2018), a biochar loaded with magnesium oxide (MgO) achieved an adsorption capacity of 178.2 mg/g for cadmium. Xu *et al*. (2022) reported that biochar loaded with MgO had an adsorption capacity of 649.79 mg for cadmium, at pH =7. At a higher pH value, the competition between the hydrogen ions and the heavy metal ions is reduced, thus making biochar space available for the binding of heavy metals. Equally, Du *et al*. (2026) used crab shell biochar loaded with MgO and iron oxide to adsorb cadmium, and reported the adsorption capacity of 301.06 mg/g. This proved that MgO helped to improve the functional groups, such as carbonyls, carboxyls, and hydroxyls on the surface of biochar, which helps to adsorb heavy metals from the wastewater through complexation, ion exchange, and electrostatic attraction. Diverse functional groups and silicate on the surface of RHBMgO@500 were responsible for its great affinity for chromium, lead, and cadmium (Li *et al*., 2022).

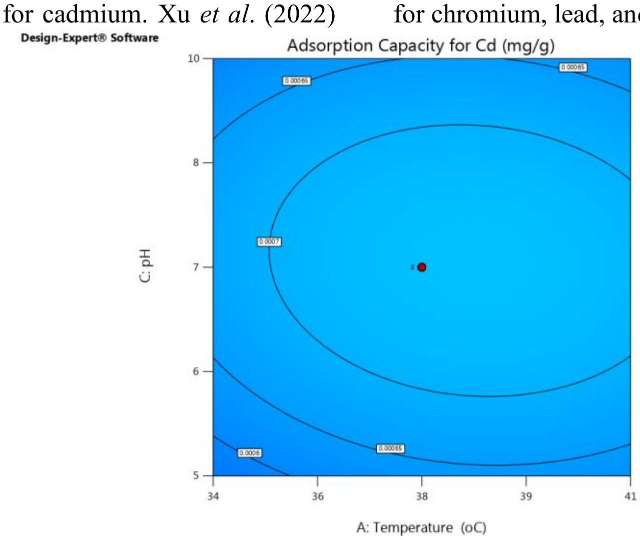

Figure 6: The contour plot showing the effect of temperature and contact time on the adsorption capacity of RHBMgO@500 for cadmium

In Figure 7, the contour lines are circular, and it indicates temperature and contact act independently, and there was little or no interaction between the temperature and contact on the adsorption capacity of RHBMgO@500 for chromium. The circular contour lines indicate that the model has equal predictive power at equal distances from the center point. The adsorption capacity of RHBMgO@500 for chromium ranged from 58.0732 mg/g to 232.218 mg/g. The adsorbent dosage and pH played significant roles in the adsorption capacity of RHBMgO@500 for chromium. A biochar derived from the peanut hulls and modified with ferric chloride hexahydrate and magnesium chloride hexahydrate showed an adsorption capacity of 12 mg/g for chromium (Tabana *et al*., 2025). Likewise, a biochar derived from the Vitex doniana recorded an adsorption capacity of 328.0 mg/g (Anakhu *et al*., 2023).

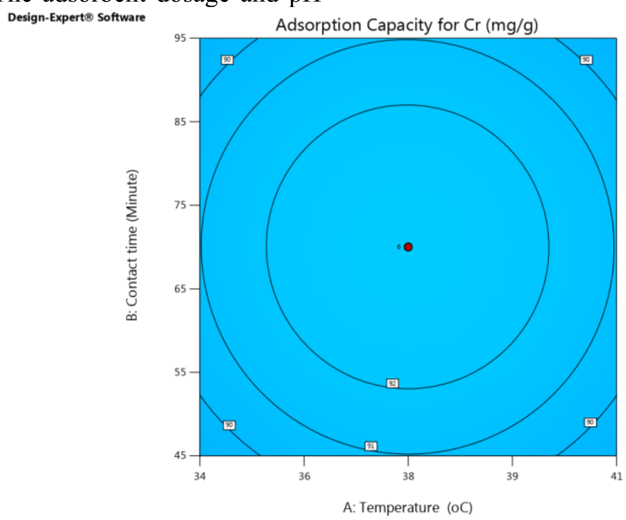

Figure 7: The contour plot showing the effect of temperature and contact time on the adsorption capacity of RHBMgO@500 for chromium

Predictive modelling of removal efficiency, and adsorption capacity of RHBMgO@500 for lead, cadmium, and chromium

The predictive modelling equations of removal efficiency, and adsorption capacity of RHBMgO500 for lead, cadmium, and chromium are presented in equations 3, 4, 5, 6, 7 and 8 respectively.

$$\text{Removal efficiency for lead }(\%) = 77.90 + 2.12A - 1.02B + 1.50C + 8.45D \quad \dots.3$$

$$\textbf{Removal efficiency for Cd} = 50.46 + 0.6849A + 4.45B + 1.83C - 3.20D + 1.71AB - 0.5AC - 4.28AD + 4.28BC + 2.57BD + 7.19CD - 0.83A^2 + 3.11B^2 - 4.42C^2 + 2.08D^2 \quad \dots 4$$

$$\textbf{Removal efficiency for Cr} = 99.98 - 0.0053A + 0.0022B + 1.18C + 0.0023D - 0.0090AB - 0.0092AC - 0.0067AD - 0.0001BC - 0.0070BD - 0.0032CD + 0.2902A^2 + 0.2936B^2 - 1.48C^2 + 0.2939C^2 \quad \dots 5$$

$$\textbf{Adsorption capacity for lead}, \frac{mg}{g} = 60.72 + 1.46A - 0.9022B + 0.7621C - 11.09D \quad \dots 6$$

$$\textbf{Adsorption capacity for cadmium}, \frac{mg}{g} = 0.0007 + 0.000A + 0.0001B + 8.18X10^{-6}C - 0.0003D + 0.00001AB - 8.7X10^{-6}AC - 0.0001AD + 0.0001BC + 0.00001BD + 0.0000CD - 0.0001A^2 + 0.00001B^2 - 0.0001C^2 + 0.0002D^2 \quad \dots\dots 7$$

$$\textbf{Adsorption capacity for chromium}, \frac{mg}{g} = 928.98 - 3.78A + 3.21B + 109.413C - 310.97D - 6.73AB - 7.31AC - 4.51AD + 1.81BC - 6.03BD - 1.12CD - 218.36A^2 - 218.04B^2 - 382.86C^2 + 108.79D^2 \quad \dots..8$$

Optimization of removal efficiency, and adsorption capacity of RHBMgO@500 for lead, cadmium, and chromium

The use of response surface methodology based on the central composite design (CCD) helped to optimize the engineering and experimental process of removal efficiency and adsorption capacity of RHBMgO@500. Figure 8 is a ramp graph which shows the optimum combination of input factors to enhance the optimum performance of RHB500 for removing and adsorbing chromium, lead, and cadmium.

Based on the results presented in Figure 8, to achieve a removal efficiency of 100 % for chromium, the values of temperature, contact time, pH, and adsorbent dosage need to be set at 41 ºC, 45 minutes, 6, 4g, respectively. This set of conditions is required to achieve 80.4748% for lead and 60.4177% for cadmium. For the adsorption capacity of RHBMgO@500 for chromium (129.99 mg/g), lead (0.068 mg/g), and cadmium (0.0013 mg/g), the input factors need to be set at: (Temperature = 34 ºC), (contact time = 45 minutes), (pH = 6), and (adsorbent dosage = 4 g).

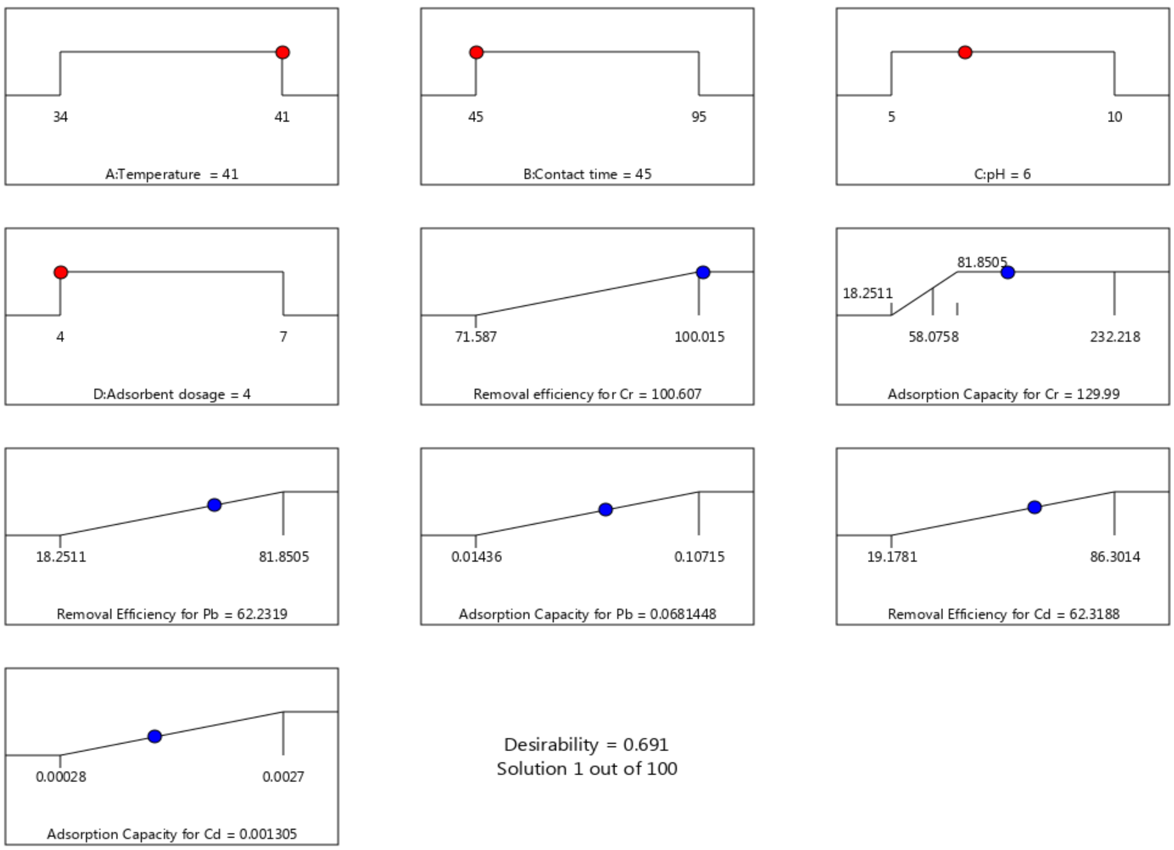

Figure 8: The ramp function graph showing the best combination of input factors to increase the adsorption capacity and removal efficiency of RHBMgO@500 for lead, cadmium, and chromium

Conclusion

An adsorbent (RHBMgO@500) was produced by combining rice husk and magnesium oxide and pyrolyzed at 500 ºC. The adsorbent was inspected with Fourier Transform Infrared (TFIR), and it revealed it had diverse functional groups that are capable of adsorbing heavy metals and other micro-pollutants in the wastewater. The tannery wastewater was analysed using Atomic Absorption Spectroscopy (AAS), and it revealed the presence of heavy metals, such as lead, cadmium, chromium, manganese, and copper. The concentration of chromium in the tannery wastewater exceeded the concentration of other heavy metals therein. In the batch adsorption experiment, the adsorbent had more affinity for chromium because the initial concentration of chromium (4645.76 ppm) is far higher than the initial concentrations of lead (3.934 ppm), and cadmium (0.073 ppm). The use of rice husk as a feedstock for biochar production was good for producing an adsorbent for water remediation and deserves more investigation.

The production of biochar by using response surface methodology (RSM) based on the central composite design (CCD), which can integrate many factors that can enhance the physical, chemical, and thermal properties of biochar, and make it more efficient for removing micro-pollutants from the tannery wastewater is recommended. Incorporating nanoparticles, such as silicates, in the production of biochar from rice husk is advisable, since silicates helps to increase the functional groups on the surface of biochar. Other nanoparticles, such as zinc oxide (ZnO), and iron trichloride ($FeCl_3$), need to be incorporated into the production of biochar from the rice husk, since it will be produced to enhance its functional groups, porosity, and cation exchange capacity, and surface area. In the batch adsorption experiment, it is advisable to design the experiment using RSM based on the CCD, which can enable a researcher to simultaneously investigate the effect of multiple independent variables (pH, contact time, temperature, and adsorbent) on the removal efficiency and adsorption capacity of an adsorbent.

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