



Removal of contaminant metformin from water by using Ficus benjamina zero-valent iron/copper nanoparticles

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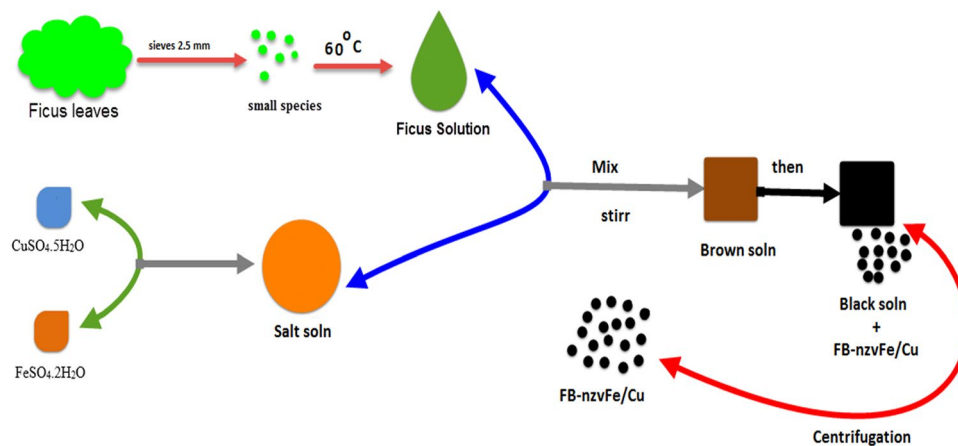
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Abstract

Green synthesis approach using Ficus benjamina leaves extract was successfully used for preparing bimetallic zero-valent iron/copper nanoparticles. The characterization using scanning dispersive X-ray spectroscopy, scanning electron microscopy, and Fourier-transform infrared spectroscopy confirmed the synthesis of bimetallic nanoparticles. The removal efficiency of metformin HCl (MF) concentration (10 mg/L) reached 73% under the (pH 5, dose 0.2 g/L, and time 30 min). Ficus nano-zero-valent Fe/Cu has good durability and stability and possesses excellent reusability for the MF removal even after being reused five times. Langmuir adsorption model is more appropriate with isotherm by the high correlation coefficients ($R^2 = 0.9991$) with $q_{\max} = 42.82 \text{ mg g}^{-1}$. The results of adsorption kinetics indicate that MF uptake on Ficus benjamina ZVFe/Cu nanoparticles is following the pseudo-second-order kinetic model. Overall, green Ficus nZVFe/Cu particle is a committed substance to remove MF. Operational parameters effect was more than 97% on the removal process, which was studied using SPSS linear regression analysis.

Graphic abstract

The formation of FB-nZVFe/Cu.



The formation of FB-nZVFe/Cu

Keywords Bimetallic Fe/Cu · Ficus benjamina · Green synthesis · Metformin HCl · Nanoparticles

Introduction

Metformin HCl (MF) is one of the most prescribed medications in the world for diabetics since 1957 [10]. MF has been considered as one of the newly identified emerging

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contaminants of concern, due to its permanence in the ecosystem and endocrine disruption [20].

Wastewater treatment plants (WWTPs) are not designed to completely remove contaminants, which result in presence of residual in wastewater and surface water [26]. It was concluded that metformin is being discharged in the aquatic environment (0.05 mg L^{-1}), which can affect the food chain [9, 12]. Many removal techniques did not perform to the required level, because they may be expensive or ineffective to remove the contaminants [6, 8].

Green synthesis has become one of the most significant low-cost and friendly alternative procedures for synthesizing nanoparticles by using plant leaf extract [4, 14]. Green leaves contain an enormous amount of both reducing and capping agents [2]. Polyphenols, flavonoids, and other reducing substances can reduce salts to zero valent and protect them from agglomeration [3]. Plants abundance, cost-effectiveness, high efficiency, and non-toxic nature, all these advantages can take advantage of it in preparing green nanoparticles in to be used in water treatment [25]. *Ficus benjamina* leaves extract provides a greener, economic, and fast method for the synthesis of nanoparticles with an appropriate size [3].

Adsorption with nanoparticles of a green-based plant is the easiest and the best technique, and it is also an inexpensive economic one which contains a large variety of functional groups that support it better in water treatment [7]. Also, they are nontoxic, do not release chemicals, and do not affect the quality of treated water.

Nanoparticles (NPS) are significant in the removal of wastewater contaminants [16, 23]. Fe nanoparticles (Fe NPS) have been used for wastewater treatment because of their properties such as a wide dispersion of reactive sites, a very high surface area, and adsorption capacity [17, 22]. Copper nanoparticles (Cu NPS) have considerable chemical/physical properties, high surface area, and less cost [19]. Cu nanoparticles are the most stable in comparison with other zero-valent NPS [27]. The potential difference (0.78 V) between iron and copper enhances the rates of contaminants diminished of Fe/Cu bimetallic particles [24]. So far, bimetallic Fe/Cu nanoscale particles prepared by green chemistry are confirmed to be efficient to remove several pollutants and more than mono-NPS [11].

Ficus benjamina nZVFe/Cu is a new idea used for the removal of metformin HCl. Therefore, the current study is to assess the ability of FB-nZVFe/Cu and applied to contaminated water of metformin HCl removal.

Experimental

Chemicals and reagents

Substances used were of the analytical grade. pH solutions were adjusted using 0.1 M sodium hydroxide and 0.1 M hydrochloric acid liquids.

Methods

Synthesis of *Benjamina* nZVFe/Cu

Ficus benjamina (FB) leaves were washed with tap water to remove dust and then with distilled water and dried in the oven at $50 \text{ }^\circ\text{C}$. Then, the leaves were ground into small pieces and sieved using a 2.5 mm sieve. In Erlenmeyer flask, 20 g of leaves was added to 100 mL distilled water and the solution was boiled at $60 \text{ }^\circ\text{C}$ for 5 min and then filtrated using Whatman no. 1 filter paper; then, the filtrate was stored at $4 \text{ }^\circ\text{C}$ until being used as a capping and reducing agent.

Synthesis of Fb-nZVI/Cu

Dissolve 0.18 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 0.94 g $\text{FeSO}_4 \cdot 2\text{H}_2\text{O}$ in 100 mL with distilled water in a volumetric flask, then add 50 ml of *Ficus benjamina* leaves extract (FBLE) drop by drop, followed by stirring for 20 min. Change of the solution color from yellowish to brown and then to black indicates the synthesis of *Ficus benjamina* zero-valent Fe/Cu nanoparticles. Fb-ZVFe/Cu nanoparticles were separated using centrifugation for 10 min and washed with anhydrous alcohol. *Ficus* ZVFe/Cu nanoparticles were then placed at $65 \text{ }^\circ\text{C}$ in a vacuum dryer oven and then stored at $4 \text{ }^\circ\text{C}$ until use.

Characterization of FB-nZVFe/Cu

The prepared FB-nZVFe/Cu sample was examined using SEM, EDAX, and FTIR spectroscopy. Before field emission, SEM FB-ZVFe/Cu nanoparticles were layered by gold which raises electrons scatter to give high contrast. EDAX is an analytical procedure used for defining the composition of the sample. FTIR spectroscopy is necessary to prove the formation of nanoparticles. It helps in detecting the chemical composition of prepared FB-nZVFe/Cu particles.

Batch adsorption studies

About 0.1 g of MF was dissolved in 100 mL water, and 10 mL of this stock was diluted to 1L with water (10 mg

L^{-1}). About 0.2 g L^{-1} of FB-nZVFe/Cu particles was added to MF solution with concentrations (5, 10, 15, and 20 mg L^{-1}). The removal was calculated using Eq. (1):

$$\text{Sorption}[\%] = [C_0 - C_e]/C_0 \times 100 \quad (1)$$

where C_0 is the initial MF concentration (mg L^{-1}) and C_e is the equilibrium MF concentration (mg L^{-1}).

The amount of MF removed by Ficus nZVFe/Cu particles was calculated using Eq. (2):

$$q_e = [(C_0 - C_e)V]/m \quad (2)$$

where q_e is the equilibrium adsorption capability (mg g^{-1}), V is the volume of aqueous solution (L), and m is the weight of the adsorbent (g).

Adsorption study

Freundlich and Langmuir isotherm models are the two most public isotherm applications [5].

Freundlich adsorption isotherm

Freundlich for dissimilar adsorption surface is given in Eq. (3):

$$\text{Ln } q_e = \frac{1}{n} \text{Ln } C_e + \text{Ln } K_f \quad (3)$$

where n (dimensionless) and K_f [$(\text{mg g}^{-1})(\text{mg L}^{-1})^{-1/n}$] are Freundlich constants related to the adsorption intensity and adsorption capacity, respectively, and K_f and n evaluated by plotting $\text{Ln } q_e$ and $\text{Ln } C_e$.

Langmuir adsorption isotherm

Langmuir assumes monolayer coverage of MF over a similar surface of FB-nZVFe/Cu. The Langmuir linearized is given in Eq. (4):

$$C_e / q_e = 1 / (K_L q_{\text{max}}) + C_e / q_{\text{max}} \quad (4)$$

where q_e (mg g^{-1}) is the mass of MF adsorbed per mass of adsorbent used, C_e (mg L^{-1}) is the equilibrium concentration of MF, q_{max} (mg g^{-1}) is the maximum monolayer capability of adsorption, and K_L (L/mg) is the Langmuir constant correlated with binding sites affinity and adsorption energy. The plot of C_e/q_e versus C_e was used to generate the values of q_{max} and K_L .

Kinetic study

Factors from two kinetic models, namely pseudo-first-order and pseudo-second-order, have been used to describe adsorption kinetics in solid–liquid systems.

Pseudo-first-order kinetic model (PFO)

The adsorption rate can be labeled using the PFO and is expressed as in Eq. (5):

$$\ln(q_e - qt) = \ln q_e - K_1 t \quad (5)$$

where q_e and qt (mg/g) represent adsorption values of MF in aqueous mediums at equilibrium and at time (min), respectively, and k_1 is the first-order equilibrium constant which calculates the plot of $\ln(q_e - qt)$ against t .

Pseudo-second-order kinetic model (PSO)

PSO equation is the most simplified and very frequently used kinetic equation, which is shown as Eq. (6):

$$t/qt = 1/K_2 q_e^2 + \frac{t}{q_e} \quad (6)$$

where q_e and qt are the adsorption capability (mg/g), at equilibrium, and at time (min), respectively, and k_2 $g/(\text{mg min})$ represents the rate constant of the adsorption. We can calculate values of k^2 from the plot of t/qt against t .

Reusability of Ficus ZVI/Cu NPs

Reusability of the adsorbent is a significant factor for estimating the cost-effective applicability and satisfying the ecologic and economic thresholds. Metformin HCl adsorption onto FB-nZVFe/Cu was carried out at MF concentration (50 mg L^{-1}). To further examine the reusability, the experiments were reiterated (up to five times) by exposing a reacted FB-nZVFe/Cu to a fresh MF solution. Each time following the reaction, the FB-nZVFe/Cu was collected immediately from the solution by centrifugation (10 min) and washed with ethanol 96% and dried in the oven at 45°C before being used for the next adsorption recycle.

Results and discussion

Characterization and analysis of FB-nZVFe/Cu

SEM and EDAX

SEM image displays the size range around 19–63 nm and a semi-spherical shape of FB-nZVFe/Cu nanoparticles. There are many pores that improve the removal process of MF, as shown in Fig. 1.

EDAX analysis indicates the synthesis of bimetallic nZVFe/Cu particles. Fe and Cu peaks point to the

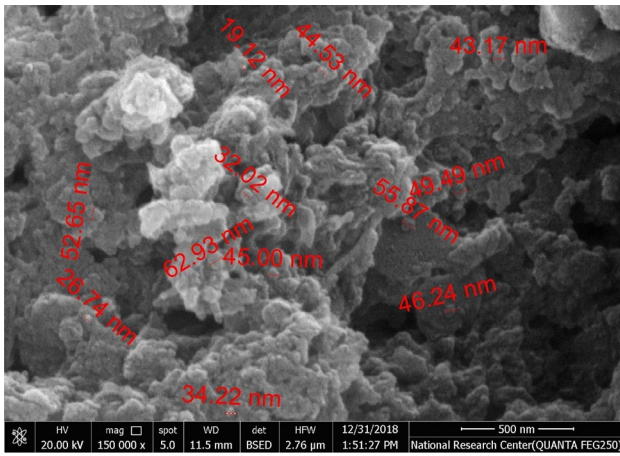


Fig. 1 SEM of FB-nZVFe/Cu nanoparticles

occurrence of bimetallic. Other peaks such as C, O, Si, and S of Ficus extract are shown in Fig. 2.

FTIR measurements

Figure 3 displays the FTIR of FB-nZVFe/Cu before the reaction (range of 400 to 4000 cm^{-1}). The broadband between 3400 and 3000 cm^{-1} was an indicator of O–H vibration, which indicates the presence of polyphenols. The phenolic peaks (existence and strength) can reduce metals and are an indicator for the synthesis of FB-nZVFe/Cu. The band at 1539 cm^{-1} indicates the occurrence of Ficus amide group; the peak at 1362 cm^{-1} signposts the occurrence of polyphenols Ar-ring C=C vibration.

Effect of operating parameters

Effect of pH

pH system plays the main role in the MF removal efficiency process, where adsorbent surface charge and capability and

Fig. 2 EDAX of prepared FB-nZVFe/Cu sample

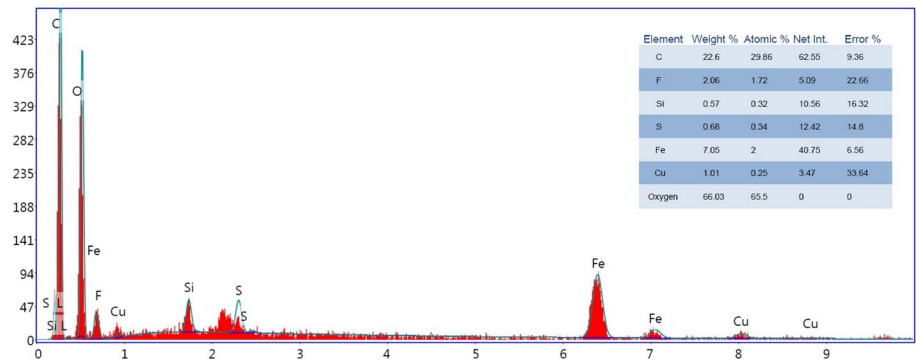
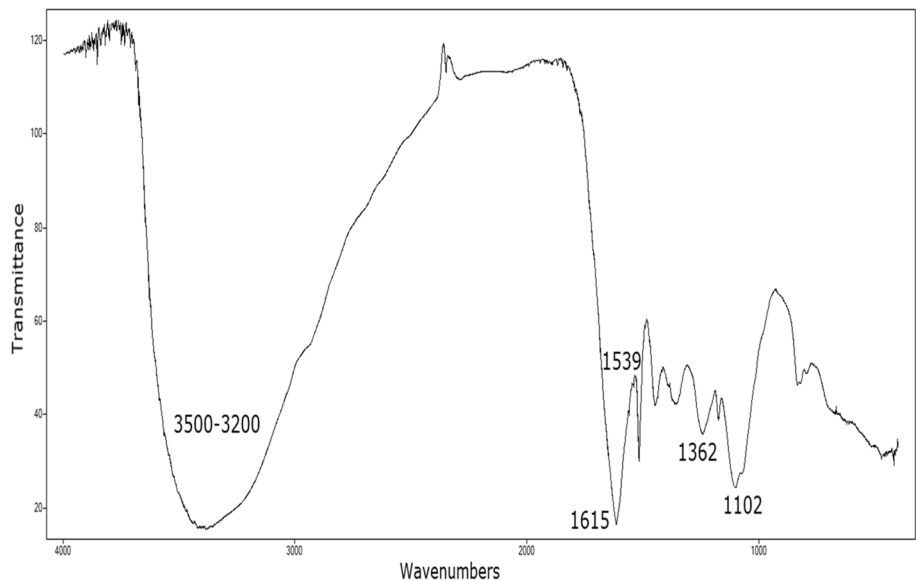


Fig. 3 FTIR spectrum of FB-nZVFe/Cu



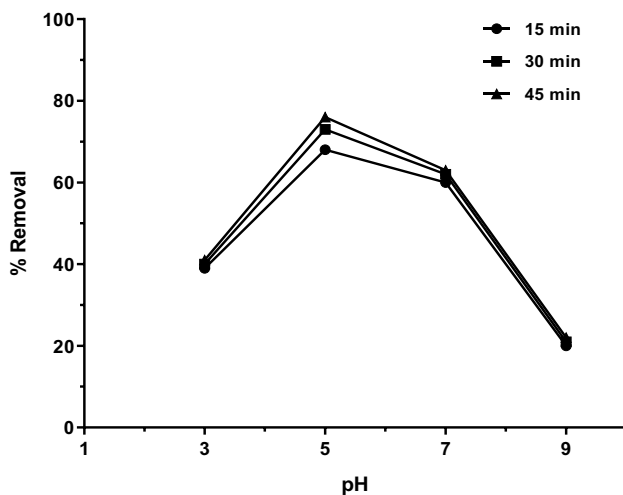


Fig. 4 Effect of pH on Metformin HCl removal

adsorbate solubility properties are controlled by acidity or alkalinity. Figure 4 shows the MF removal efficiency at different pH 3, 5, 7, and 9, and at different times 15, 30, and 45 min, respectively. When the dose of FB-nZVI/Cu was 0.2 g L^{-1} , concentration 10 mg L^{-1} , and stirring rate 100 rpm, the MF removal were (39, 68, 60, and 20%), (40, 73, 62, and 21%), and (41, 76, 63, and 22%), respectively. It was observed that pH 5 is the most appropriate for the MF removal. Ficus benjamina has a low zero-charge point of 4.85 due to the high acidity of the solution of leaf extract [2]. In contrast, MF is neutral in the range of pH 3 to 11 [28]. At pH of PZC, the surface of the nanoparticles is not charged and there is a potential difference between iron and copper, which leads to increase in the number of free e^- , which enhances the degradation of the CAF compound; also there are a lot of available vacant adsorption sites, which allow the CAF mass to be transported better and the spread to the inner FB-nZVFe/Cu; all this led to a high removal efficiency. At $\text{pH} < \text{pH}_{\text{pzc}}$, [in acidic medium], Low in the removal capacity due to the presence of an excess of H^+ ions that occupy available vacant adsorption sites, the free e^- which generated from the adsorbent neutralize the excess of H^+ , and portion of nanoparticles dissolve in the medium [18]. At $\text{pH} > \text{Pzc}$ [in alkaline medium], the repulsion between hydroxide (OH^-) ions and the negatively charged surface of FB-nZVFe/Cu leads to a low removal efficiency [21]; or maybe the low availability of nanoparticles which precipitates in the solution [15]. Based on the above, pH5 is the optimal MF removal.

Effect of contact time

Effect of different times of 15, 30, 45, 60, 90, and 120 min was studied on the metformin HCl (10 mg L^{-1}) removal

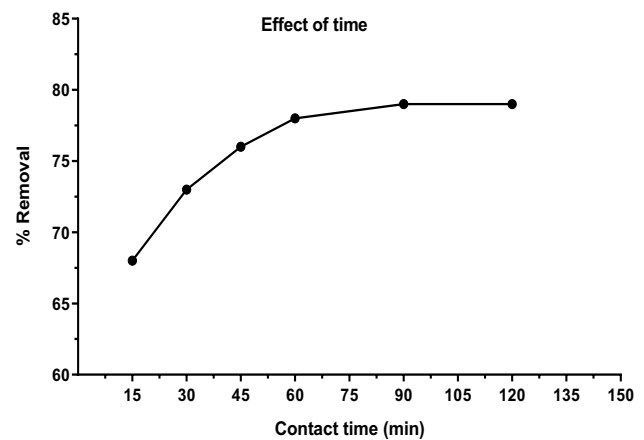


Fig. 5 Effect of contact time on metformin HCl removal

using 0.2 g L^{-1} of bimetallic Ficus nano-Fe/Cu at pH 5, and the removal rates were 68, 73, 76, 78, 79, and 79%, as shown in Fig. 5; the increase in connection time gradually leads to increase in the interference of contaminant in the unfilled sites of the nanoparticles and increase in the amount of electrically attracted molecules between the negatively Ficus ZVFe/Cu surface and MF positively charged; in total, all this has led to increase in the removal.

Effect of adsorbent dose

Figure 6 depicts MF removal efficiency as a function of the adsorbents dose. FB-nZVI/Cu doses were varied between 0.1 and 0.3 g L^{-1} . Other operational factors such as pH, contact time, and stirring rate were 5, 30 min, and 100 rpm, respectively. Metformin concentration was 10 mg L^{-1} ; the removal percentages were 44, 73, and 98%. Optimal dose for MF removal was found to be about 0.2 g L^{-1} , as shown in Fig. 7. As expected, increase in the dose of Ficus nZVFe/Cu

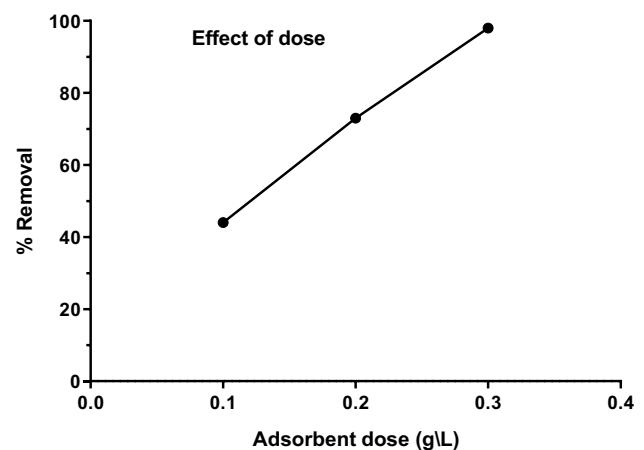


Fig. 6 Effect of adsorbent dose on metformin HCl removal

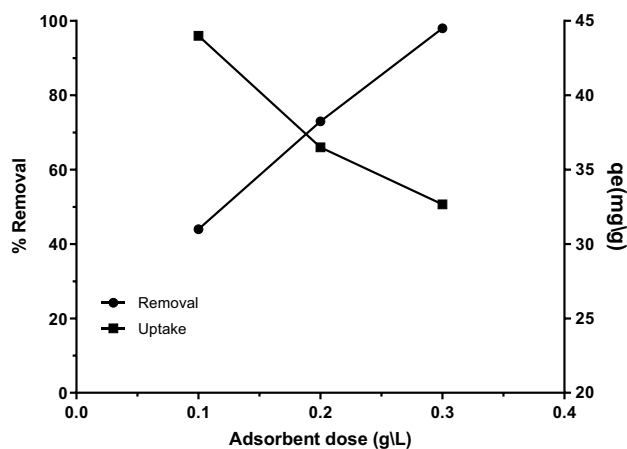


Fig. 7 Optimum effective dose for metformin HCl removal

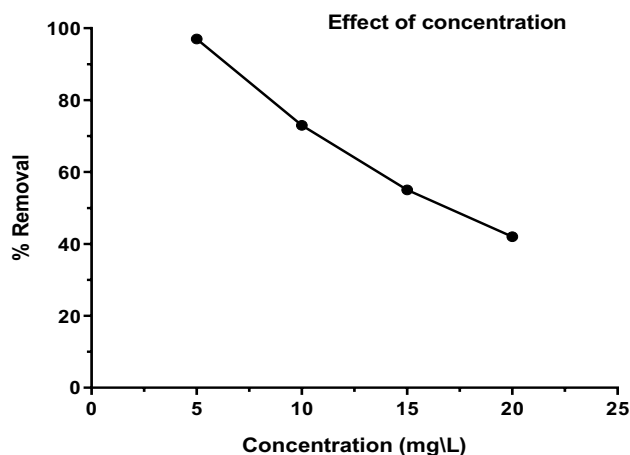


Fig. 9 Effect of concentration on metformin HCl removal

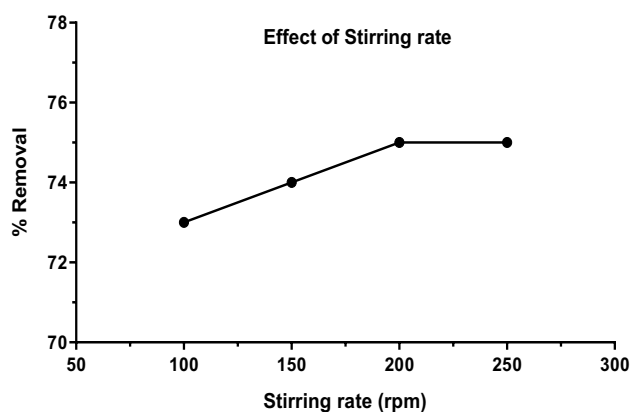


Fig. 8 Effect of stirring rate on metformin HCl removal

Cu leads to an increase in the unoccupied sites number; then, the removal increased.

Effect of stirring rate

Figure 8 displays metformin HCl removal by Ficus nZVFe/Cu at varied stirring rates (100 to 250 rpm) and other influences as pH 5 and time 30 min. Metformin HCl concentration was 10 mg L^{-1} , and the removal ratios were 73, 74, 75, and 75%. The optimal stirring rate for MF removal was found to be 100 rpm.

Effect of the concentration

The removal experiments using Ficus ZVFe/Cu particles were carried out on metformin HCl solutions having a several concentrations of 5, 10, 15, and 20 mg L^{-1} , at pH 5, time 30 min, and dose was 0.2 g L^{-1} , and the removal percentages were 97, 73, 55, and 42%, as shown in Fig. 9. At the beginning of the adsorption process, the removal efficiency

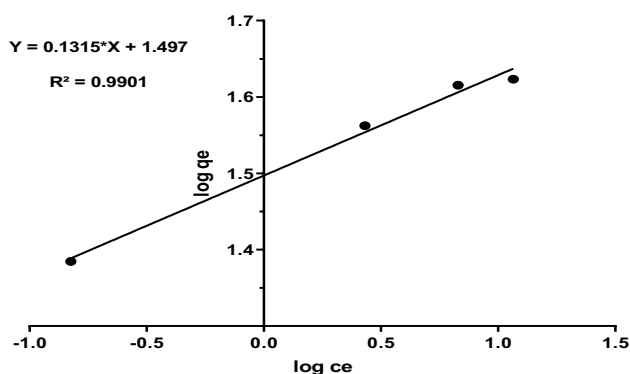


Fig. 10 Freundlich for metformin HCl contributing component

was higher because of the excessive number of available adsorption sites of FB-nZVFe/Cu particles, but it decreased with time gradually, due to saturation of and decrease in the number of these sites.

Adsorption isotherm studies for metformin HCl removal

The capacity of the Ficus nZVFe/Cu was evaluated by Freundlich and Langmuir adsorption isotherms models, which are the two common public applications under confirmed conditions [1, 13]. Langmuir model was fitted well with isotherm by the high correlation coefficients (R^2) compared to Freundlich, as shown in Figs. 10 and 11, maximum adsorption capability of 42.82 mg/g , and n value > 1 from Freundlich, as shown in Table 1.

Kinetic studies

Table 2 shows that PSO model data fitted well than PFO with higher correlation coefficients R^2 , as shown in

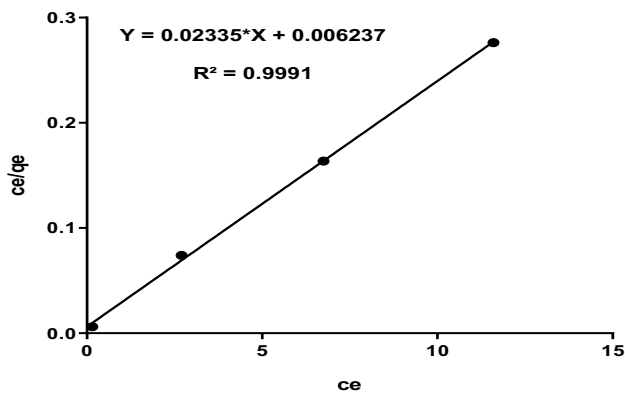


Fig. 11 Langmuir for metformin HCl contributing component

Table 1 Isotherm parameters for the adsorption of metformin HCl

Isotherm name	Parameters	Values
Freundlich	n	7.604
	R ²	0.9901
Langmuir	qmax (mg g ⁻¹)	42.82
	R ²	0.9991

Table 2 Kinetic parameters for the adsorption of metformin HCl

Kinetic name	Parameters	Values
Pseudo-first-order	K ₁	0.053
	q _e (cal)	5.989
	R ²	0.9793
Pseudo-second-order	K ₂	0.007
	q _e (cal)	41.05
	R ²	0.9997

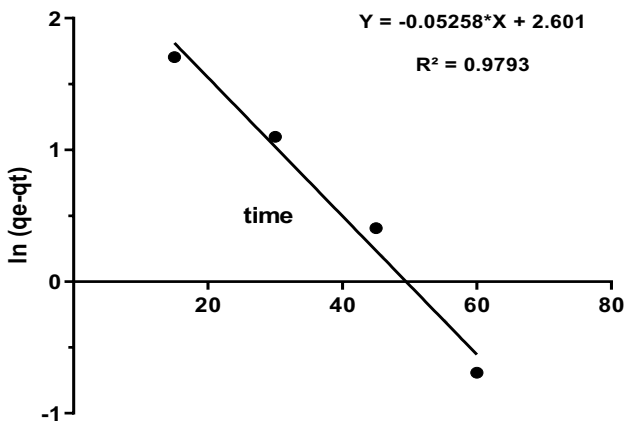


Fig. 12 PFO kinetics model data for metformin HCl

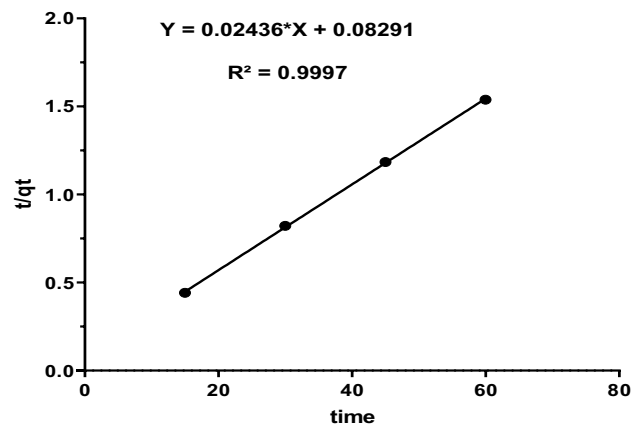


Fig. 13 PSO kinetics model data for metformin HCl

Table 3 Coefficients^a

Model	Unstandardized coefficients		Standardized T coefficients		Sig.
	B	Std. error	Beta		
1 (constant)	78.205	6.741		11.601	0.000
pH	-5.726	0.801	-0.300	-7.148	0.000
Time	0.094	0.023	0.171	4.088	0.001
Dose	270.000	17.855	0.619	15.122	0.000
Stirring	0.015	0.015	0.042	1.003	0.330
Concentration	-3.475	0.211	-0.680	-16.477	0.000

^aDependent variable: removal

Figs. 12 and 13. The value q_e (cal) = 41.05 is approximately similar to q_e (exp) 39.5 for MF removal.

Statistical analysis

The effect of the following variables on the removal technique has been calculated, where it was found that R² = 0.971. This means that the studied variables occupied more than 97% of the total of the factors affecting the procedure as the error of the estimate is very low (2.52508).

ANOVA program was applied, and the data given showed the sum of squares and effect of the model. It was observed that the P value < 0.001, where the model is considered successful if P value is less than 0.05.

The data given in Table 3 [coefficients^a] showed that all variables had an effect on the removal technique but the effect of the stirring rate was considered to be not significant where the P value (0.330) is more than 0.05, which means that it can be neglected during the removal process.

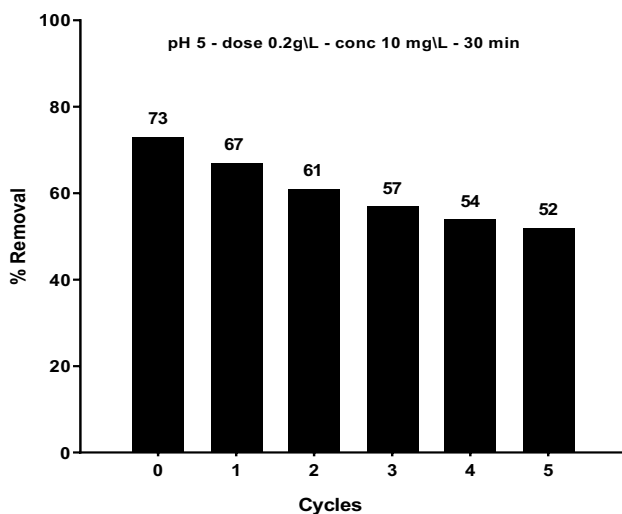


Fig. 14 Reuse performance of the synthesized FB-nZVFe/Cu

Response surface methodologies (RSM)

IBM SPSS Statistics results support the practical results. By applying the B values shown in Table 3, the removal equation can be deduced as shown in Eq. (7):

$$R\% = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_5X_5 \quad (7)$$

where R is the removal percent, B is constant, X_1 is the effect of pH, X_2 is the effect of contact time, X_3 is the effect of adsorbent dose, X_4 is the effect of stirring rate, and X_5 is the effect of concentration.

Reusability of FB-ZVI/Cu NPs

Figure 14 signifies the removal efficiencies decreased with each reuse series. Thus, removal efficiency was 67, 61, 57, 54, and 52% after being used in the first, second, third, fourth, and fifth recycles, respectively. Yet the removal efficiency of MF was still high even in the fifth recycle. Reusability of the adsorbent still retained good adsorption capability after the fifth rounds of recycles. These results reveal that green-nZVFe/Cu has a high potential to be used repeatedly in removing MF without significant decrease in the removal suitability.

Conclusions

In this study, the green nanoscale zero-valent Fe/Cu (FB-nZVFe/Cu) accomplishes MF removal under various operational factors. Maximum MF removal was observed at pH 5. Metformin HCl removal between 97 and 42% was achieved after using different MF concentrations of 5, 10, 15, and 20 mg L⁻¹ with 0.2 g L⁻¹ of FB-nZVFe/Cu, at pH

5, and when the dose increased from 0.1 to 0.3 mg L⁻¹, the removal efficiency increased by 54% ($C_0 = 10 \text{ mg L}^{-1}$). Langmuir adsorption model is more appropriate with isotherm by the high correlation coefficients ($R^2 = 0.9991$). PSO kinetic model is a better fit to the experimental data than PFO kinetic model. The removal efficiency of MF was still over 50% after reusing the material five times. FB-ZVFe/Cu nanoparticles are an eco-friendly technique and can lead to success in wastewater treatment and produce high-quality treated effluent.

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