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Scientific paper

Synthesis of Fe_3O_4 Nanoparticles Modified by Oak Shell for Treatment of Wastewater Containing Ni(II)

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Abstract

In present study, removal of nickel ions (Ni (II)) from synthetic wastewater using Fe_3O_4 nanoparticles modified by oak shell was investigated. The FTIR analysis of the adsorbent suggested the occurrence of interaction between the carboxyl group on oak shell modified magnetic nanoparticles (OSMMN) surface and Ni (II). Also, the morphology and size of the adsorbent were observed by SEM and TEM. Additionally, the effect of different parameters such as contact time, adsorbent dose, solution pH and initial concentration of nickel (II) ions were investigated on the adsorption of nickel. The adsorption experiments showed that the maximum Ni(II) adsorption was obtained as contact time = 15 min, temperature = 25 °C, adsorbent dosage = 2.6 g/L, and pH = 4.5. In these conditions, 93.88% Ni(II) was removed from aqueous solution. Moreover, in order to study equilibrium behavior of adsorption, Langmuir and Freundlich isotherm models were applied. The results showed that the experimental data were fitted well with the Langmuir isotherm model, and the maximum adsorption capacity of the adsorbent using Langmuir model was determined to be 454.54 mg/g which was a considerable amount.

Keywords: Oak shell; magnetic nanoparticles; adsorption; synthetic wastewater; nickel

1. Introduction

Heavy metal contamination of water is a common phenomenon. The effluent of a number of industrial and metallurgical processes like plating, photography, aerospace, atomic energy and petrochemical facilities can result heavy metals pollution in the water resources, if the metal content is not treated.[1,2] So, the discharge of heavy metals into an aquatic ecosystem has become a matter of concern over the last decades because of their extreme toxicity and tendency for bioaccumulation in the food chain even in relatively low concentrations.[3] Pollutants of serious concern include lead, chromium, mercury, uranium, selenium, zinc, arsenic, cadmium, gold, copper and nickel.[4] Nickel is a toxic heavy metal that is widely used in silver refineries, electroplating, zinc base casting and storage battery industries.[5] The chronic toxicity of nickel to humans and the environment has been well documented. For example, high concentration of nickel (II) causes cancer of the lungs, nose and bone. It is essential to remove Ni (II) from industrial wastewater before being discharged.

There are a number of methods for removal of heavy metals from aqueous solutions and industrial wastewater. Common removal techniques of heavy metals from industrial wastewaters are chemical precipitation, ion change, solvent extraction, reverse osmosis, ultrafiltration, electro-dialysis and adsorption. Adsorption technique is an attractive method for water treatment, especially if the adsorbent is costly efficient, convenient to separate and easy to regenerate.[6,7] Nowadays, bio-adsorption is used for heavy metals ions removal and is highly favorable. In such biologic adsorption processes, many biomaterials are employed. Using agricultural residues or industrial by-products having biological activities has received a considerable attention.[8] In recent years, a number of agricultural materials such as moss peat,[5,9] banana peels,[10,11] orange peel,[12] peanut hulls,[13] activated charcoal,[14] almond husk,[15] eggshell[16] and other unimportant agricultural wastes have been used to remove heavy metals contaminants. Recently, magnetic particles have also gained special attention in water treatment.[17] In terms of simplicity, high potential, high surface area and high efficiency to removal heavy metal ions from waste water. Of these particles, Fe_3O_4 is the traditional particle that is extensively used in wastewater treatment; because of their high activities, hydrophilic, chemically stable, non-toxic and magnetic. Also, it's an environment-friendly adsorbent, cheap and available.[18,19,20]

The purpose of this study was to determine the potential and adsorption capacity of Fe_3O_4 nanoparticles modified by oak shell (OSMMN) for the removal of Ni (II) from aqueous solution. The effect of various parameters such as namely contact time, adsorbent dose, pH, and the initial concentration of Ni(II) ions was studied. Also, the equilibrium behavior of adsorbent was investigated. To the best of author's knowledge, this is the first report of the application of OSMMN for attenuation of Ni ions from aqueous solution.

2. Materials and Methods

2. 1. Chemicals and Devices

All chemicals and reagents were analytical grade. $FeCl_2.4H_2O$ (99.9%), $FeCl_3.6H_2O$ (96%), KCl salt, citrate, hydrochloric acid (37%) and ammonia (NH_3OH) solution (25%), were purchased from Merck company (Darmstadt, Germany) and used without further purification. Nickel was also purchased from Sigma-Aldrich (Germany) and oak shell was were collected from local trees. The amount of Ni (II) in aqueous solutions was measured by using Cintra 101 spectrophotometer (GBC Specific Equipment, Australia) at a wavelength of 546 nm before and after adsorption process. Also, the citrate buffer solution with pH 4.5 was prepared using citric acid (0.1 M), NaOH and HCL (0.1 M). In addition, a transmission electron microscope (TEM, 906E, LEO, Germany), Scanning electron microscope (SEM, VEGA, TESCAN), pH-meter (632Metrohm, Herisau, Switzerland) and a super magnet (1.2 T, 10 cm × 5 cm × 2 cm) were used in the experiments. Moreover, Dynamic Light Scattering (HORIBA Jobin Yvon, SZ-100) is applied for measuring the particle size distribution of the adsorbent.

2.2 Preparation of Fe_3O_4 Nanoparticles Modified by Oak Shell

Magnetic oak shell nanoparticles was prepared by co-precipitation method. To do so, Fe_3O_4 nanoparticles were synthesized by co-precipitation of 4 mmol ferric ($FeCl_3$ $6H_2O$) and 2 mmol ferrous salts ($FeCl_2$ $4H_2O$) in distilled water. After stirring for 1 h, chemical precipitation was achieved at 80 °C under vigorous stirring by adding 40 ml of NH_4OH solution. During the reaction process, the pH was maintained approximately at 10. After adding ammonia solution, it was stirred for 1 h. Afterwards, the precipitate was washed with distilled water for removal of all existing in the effluents. Next, using a magnet, the magnetic adsorbent was collected at the bottom of the balloon and the solution was discarded. After preparing Fe_3O_4, one gram of oak shell was added to the solution and stirred for 20 min. Then, in order to oxidize the mixture, NH_4OH solution added as dropwise and stirring continued for 50 min, and stirring was then stopped. After, the product was separated from the solution using a magnetic field and washed with distilled water. The solution was then heated in an oven at 105 °C for 24 h until dry. After cooling, the adsorbent was pulverized by a mill.

2. 3. Adsorption Experiments

The adsorption experiments were done using batch method. 0.03 g of magnetic oak shell nanoparticles were equilibrated with 50 ml of solution containing nickel. The pH value of the samples was adjusted by using diluted solutions of NaOH and HCl (0.1 M). After addition of magnetic oak shell nanoparticles, the resulting solution was stirred for 5 min. Then, the suspension was allowed to settle by a magnet and the supernatant was analyzed to measure the remaining nickel by atomic absorption apparatus.

In all experiments, bio-adsorption percentage of Ni ions (R%) was calculated with equation 1:

$$R\,(\%) = \left(\frac{C_o - C_f}{C_o}\right) * 100 \qquad (1)$$

Where C_0 and C_f represent the initial and final ion concentrations, respectively. Also, all experiments were done duplicate.

2. 4. Desorption Process

For the desorption test, the adsorbed nickel ions on the adsorbent were transferred to a flask containing 100 mL of desorbing agent such as HNO_3. The mixture was stirred at 200 rpm using a magnetic stirrer at room temperature for 2 h and the desorbed nickel(II) concentration in the solution were determined by spectrophotometer. The desorption process was done consecutively six cycles.

3.Results and Discussion

3. 1. Characterization of Biosorbent

To characterize the functional groups in bio-adsorbent, FTIR analysis was applied. The FT-IR spectra of

Fe_3O_4 nanoparticles in the range of 400–4000 cm^{-1} is represented in Fig. 1-a. An adsorption band at 593 cm^{-1} is belonged to the vibrations of the Fe-O functional group. The bands appearing at 3447 cm^{-1} can be attributed to O-H group that cover iron oxide surfaces in an aqueous environment. Modification of oak shell onto the Fe_3O_4 MNPs was also ascertained by FTIR. As shown in Fig. 1-b, the sharp peak at 612 cm^{-1} corresponds to Fe-O vibration in magnetite. The peak at 3412 cm^{-1} belongs to the N-H bond of oak shell that indicates the presence of oak shell onto magnetic iron oxide nanoparticles. Also, several peaks have been viewed in this Figure which can be related to the groups of C=C, N-H and etc.

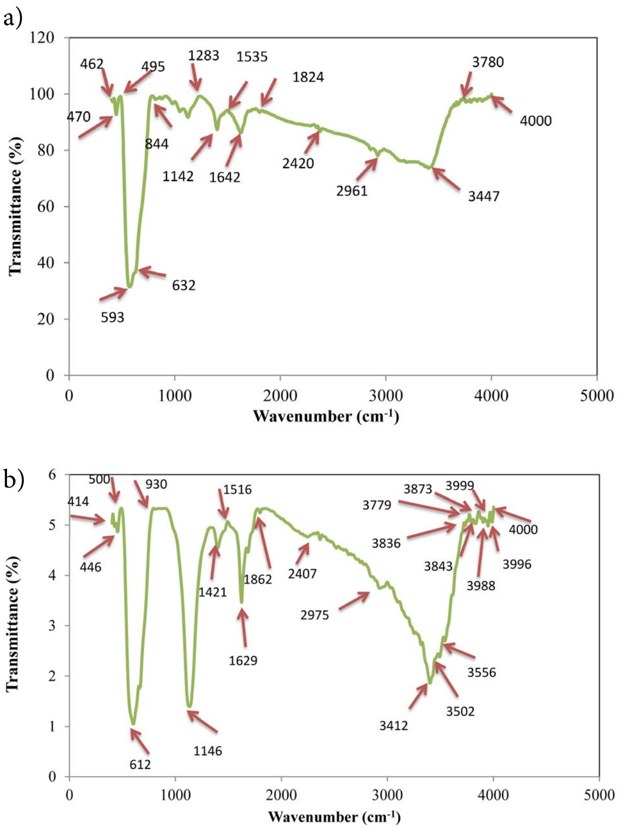

Fig. 1: (a) FT-IR spectra of Fe_3O_4 nanoparticles and (b) FT-IR spectra of OSMMN

Furthermore, SEM and TEM images provide information about morphology and size of oak shell magnetic nanoparticles. Figs. 2 and 3 are shown SEM and TEM images, respectively. It can be observed that the magnetic nano-particles with the average size of 40 nm have a high surface area and abundant pore for adsorption of ions.

Also, particle size distribution of a material can be important to understand the average particle size. Particle size distribution of adsorbent is displayed in Fig. 4. As shown in this Fig., the average size of particles is about 10 nm which shows the particle size of the adsorbent is on a nano scale.

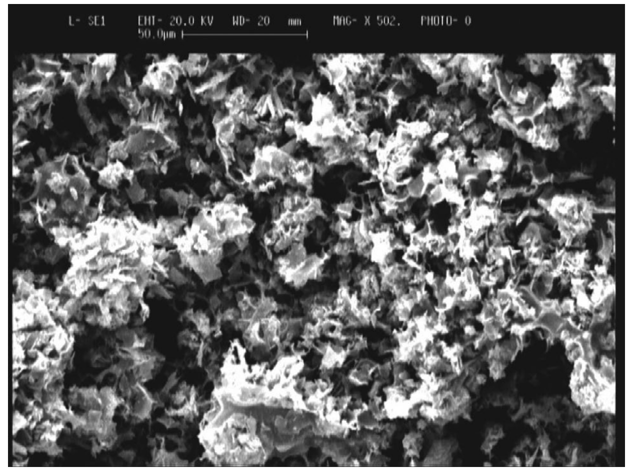

Fig. 2: SEM image of OSMMN

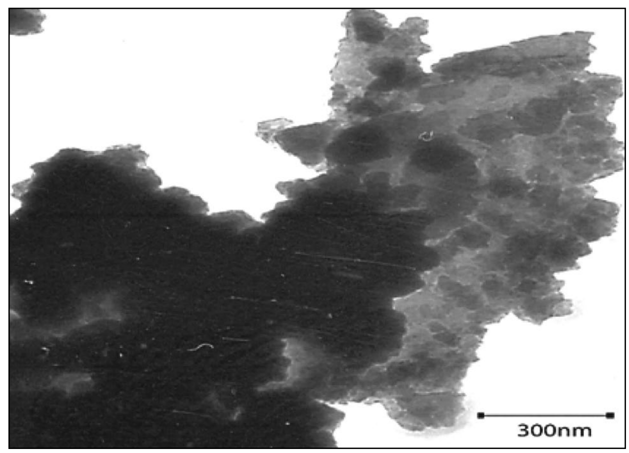

Fig. 3: TEM image of OSMMN

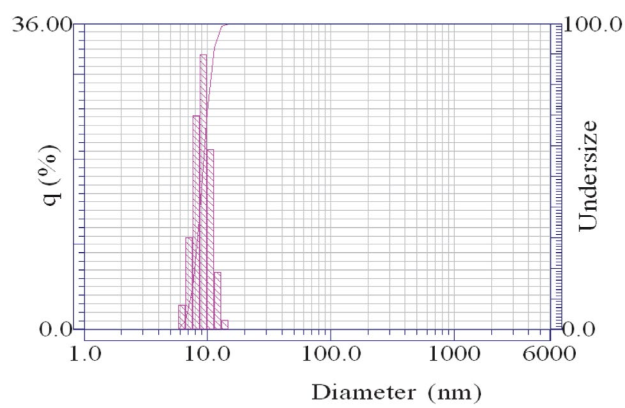

Fig. 4: Particle size distribution of Fe_3O_4 nanoparticles modified by oak shell

3. 2. Effect of Solution pH and Buffer Volume

Initial solution pH is one of the effective parameters in adsorption of metal ions, because hydrogen ion competes with metal ion to relocate active sites of the adsorbent.[21] The effect of pH was conducted by mixing 0.03 g (0.6 g/L) of adsorbent in 50 ml Ni (II) solution with the concentration of 20 mg/L. HCl and NaOH was used for the purpose of keeping pH in the range of 4-7 throughout the experiments. The effect of pH on the removal of Ni^{2+} from aqueous solution is presented in Fig. 5. It can be observed that the removal of nickel ions increased with increasing pH and reached a maximum value at pH 4.5. At lower pH values, Ni (II) ion removal was inhibited, because at low pHs the medium contains a high concentration of hydrogen ions, therefore competition between H^+ and Ni^{2+} ions for the available adsorption sites could be possible. The percentage removal of Ni was observed to be sharp between pH value of 4.5 to 7 (from the percentage removal of 92.2% to 66.38%). At pH values greater than 4.5, the adsorption of Ni (II) ions decreases because of the precipitation of nickel hydroxide resulting from Ni (II) ions reacting with hydroxide ions. In further works, the pH of the solutions was adjusted by using citrate buffer volume. The effect of buffer volume on the removal efficiency is displayed in Fig. 6. As shown in this Fig., the removal yield was increased with increasing buffer volume and the maximum efficiency was obtained at 3 ml of buffer solution with the adsorption of 92.8%.

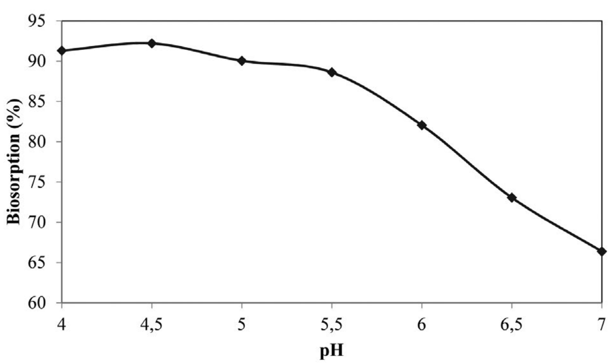

Fig. 5: Effect of solution pH on Ni (II) removal (Conditions: temperature of 25 °C, initial ion concentration of 20 mg/L, contact time 5 min, adsorbent dose of 0.6 g/L).

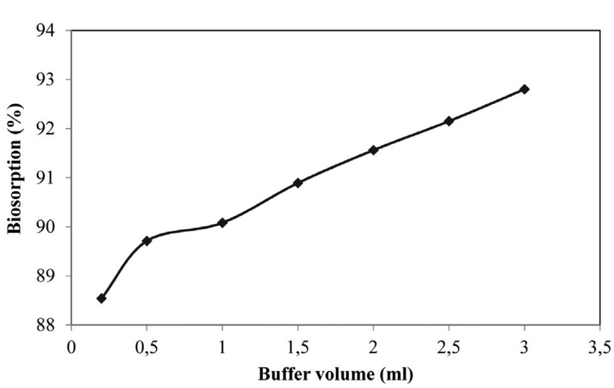

Fig. 6: Effect of buffer volume (ml) of solution on Ni (II) removal.

3. 3. Effect of Electrolyte

The effect of electrolyte concentration (adjusted by KCl) on the adsorption of Ni (II) is illustrated in Fig. 7. As shown in this Figure, the adsorption efficiency of Ni (II) decreased within the concentration range of 0–1 mol/L of KCl in the test solution. At higher concentration, the Ni ion removal efficiency was decreased. So, it is concluded that the presence of KCl electrolyte has negative effect on bio-adsorption of Ni (II) ions using oak shell magnetic nanoparticles and a concentration of 0.0 mol/L (93.52% removal) was used for further works.

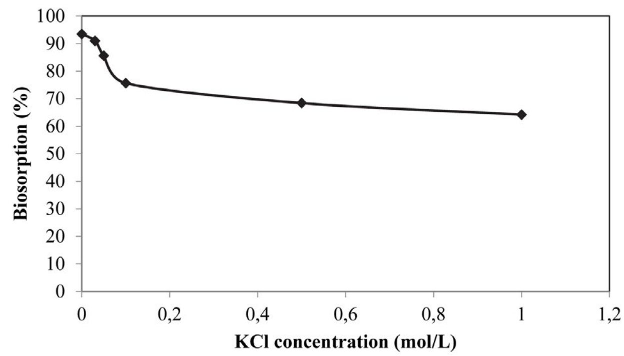

Fig. 7: Effect of electrolyte concentration (adjusted by KCl) on the removal of Ni (II)

3. 4. Effect of Temperature

The effect of temperature on the adsorption of Ni (II) from synthetic wastewater was examined within the temperature range of 5–25 °C. The effect of temperature on nickel (II) removal using OSMMN is illustrated in Fig. 8. The results showed that the adsorption of Ni (II) using 0.6 g/L of OSMMN at the pH of 4.5 is increased versus variation of temperature. At 25 °C, the adsorption efficiency was 93.6% that the highest adsorption rate was obtained.

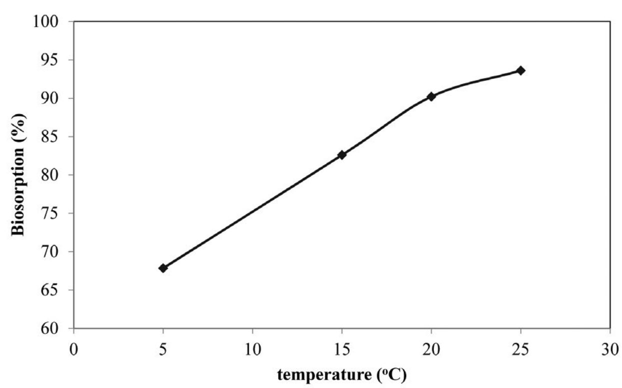

Fig. 8: Effect of temperature on Ni (II) removal from aqueous solution using OSMMN (Conditions: initial ion concentration 20 mg/L, contact time 5 min, adsorbent dosage 0.6 g/L and pH of 4.5).

3. 5. Bio-Adsorbent Dosage Effect

Adsorbent dosage is considered as an important parameter, because this parameter defines the capacity of adsorbent.[22] The effect of bioadsorbent dose on the removal of Ni (II) from aqueous solution was investigated in a batch system by adding various amounts of adsorbent (0.6–3 g/L) into a flask containing 50 mL of Ni (II) solution. The initial ion concentration and pH value of the solutions were fixed at 20 mg/L and 4.5 for all batch experiments, respectively. Also, the suspension was stirred for 5 min. After that time, the solution was coagulated and settled and the supernatant was analyzed for the remaining Ni (II). The results are shown in Fig. 9. The results revealed that as adsorbent dosage went up, the bio-adsorption also increased owing to rise in the number of active sites of bio-adsorbent. The optimum dosage of oak shell magnetic nanoparticles for removing Ni (II) was obtained 2.6 g/L with the maximum removal of 93.66%.

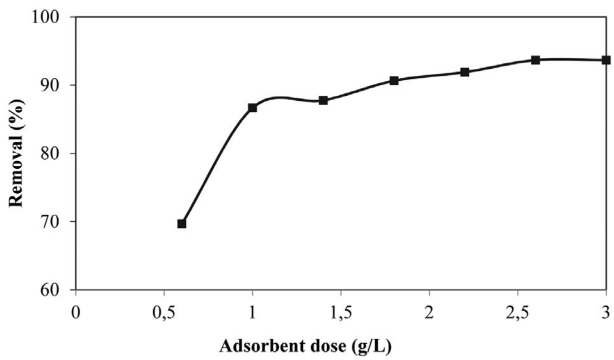

Fig. 9: Effect of OSMMN dosage on the removal of Ni (Conditions: temperature of 25 °C, initial ion concentration of 20 mg/L, contact time 5 min and pH of 4.5)

3. 6. Effect of Contact Time

Contact time is one of the important parameters in bio-adsorption process.[23,24] The effect of stirring time on the performance of OSMMN in adsorbing Ni (II) was investigated. The solution pH and oak shell magnetic nanoparticles dosage were fixed at their obtained optimum values. Fig. 10 shows removal efficiencies for Ni (II) as a function of stirring times (1–20 min). These data elucidate that adsorption started immediately upon adding the magnetic oak shell particles to nickel solution. The removal efficiency of Ni (II) was rapidly increased from 56.81 % to 93.88 % as the stirring time was increased from 1 to 15 min. After 15 min, no change in biosorption was observed. So, according to these results, the optimum stirring time for removing nickel obtained 15 min.

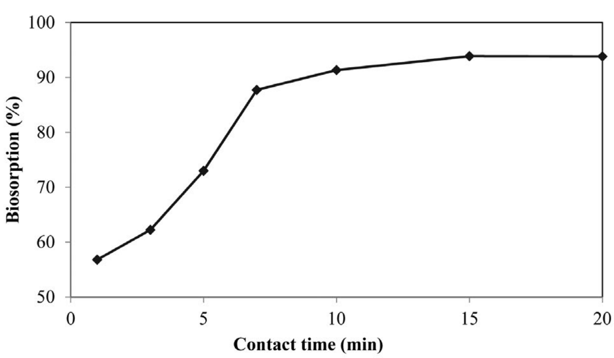

Fig. 10: Effect of contact time on Ni (II) removal (Conditions: temperature of 25 °C, initial ion concentration of 20 mg/L, pH of 4.5 and adsorbent dose of 2.6 g/L)

3. 7. Modeling of Isothermal Adsorption

Adsorption isotherms are useful for the description of adsorption process and its mechanisms and also adsorption isotherms provide the basic requirements for designing adsorption process. Two important isotherm models were selected in this study, Langmuir and Freundlich isotherm models.[20,23,25]

Freundlich model is an empirical model and can describe adsorption capability of adsorbents. This model is applied for non-ideal adsorption on heterogeneous surfaces. The linear form of this model is expressed by Equation 2.[23]

$$Lnq_e = LnK_F + \frac{1}{n} LnC_e \quad (2)$$

In Eq. (2) K_F (Lg^{-1}) and n (dimensionless) are Freundlich isotherm constants and the degree of nonlinearity between solution concentration and adsorption, respectively. The plot of $Ln\ q_e$ versus $Ln\ C_e$ for the adsorption was employed to generate K_F and n from the intercept and the slope values, respectively (Fig. 11-a).

Also, the Langmuir model describes the monolayer bio-sorption process onto the adsorbent surface with specific binding sites. The linear form of Longmuir model is written as follows:[23]

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (3)$$

In Eq. (3), q_m is the monolayer adsorption capacity (mg/g); and K_L is the Langmuir constant (L/mg), and is related to the free energy of adsorption. A plot of C_e/q_e versus C_e for the adsorption of Ni onto bio-adsorbent shows a straight line of slope, $1/q_m$, and intercept, $1/K_L q_m$ (Fig. 11 (b). In order to determine the variability of adsorption, a dimensionless constant called as separation parameter 'R_L' was used and defined as in the Equation (4).

$$R_L = \frac{1}{1 + K_L C_o} \quad (4)$$

Where C_o is the highest initial Ni concentration (mg/L). The value of separation parameter indicates the shape of isotherm to be either favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$).[23,24]

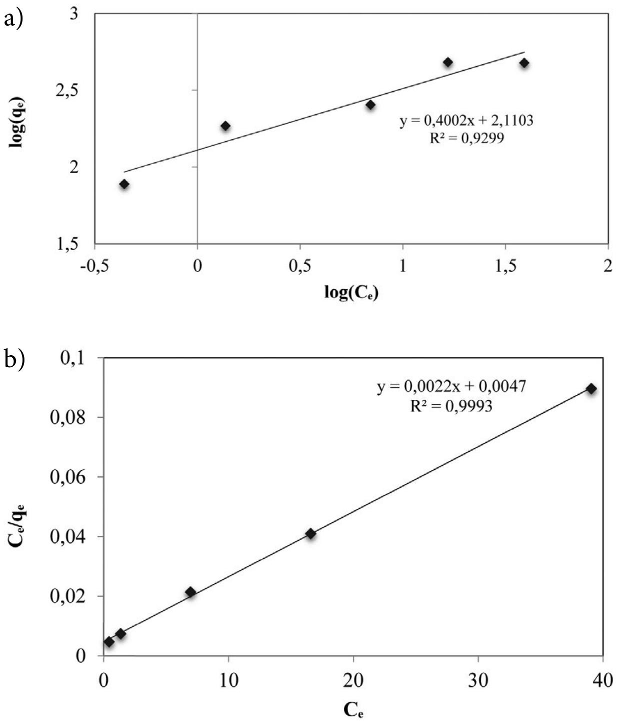

Fig. 11: The adsorption isotherm curves related to a) Freundlich and b) Langmuir models for adsorption of Ni (II) on oak shell modified by magnetic nanoparticle.

The capacities of magnetic oak shell nanoparticles to adsorb Ni (II) were examined by measuring the initial and the final concentration of Ni (II) at the pH of 4.5 and the temperature of 25 °C in a batch system. Both Langmuir and Freundlich adsorption isotherms were used to normalize the adsorption data. The correlation of ion adsorption data with the Langmuir isotherm model was higher (with R2 values of 0.9993) than the Freundlich model (R^2 = 0.9299). Summarizes the models, constants, and coefficients are listed in Table 1. According to this table, the maximum predictable adsorption capacity of Ni (II) is 454.54 mg ion/g adsorbent. K_L represents the equilibrium biosorption constant and therefore, higher values of K_L led to an optimal adsorption process.

Table 1: Parameters and constants of Langmuir and Freundlich isotherm models for the removal of Ni(II) by means of OSMMN.

Langmuir	
Parameters	**Values**
q_{max} (mg/g)	454.54
K_L (L/mg)	0.46
R^2	0.9993
Freundlich	
K_F (mg/g)	8.24
n	5.75
R^2	0.9299

3. 8. Recycling of the Adsorbent

The ability of recovering and reusing of the adsorbent was tested in several steps of adsorption and desorption. The result is shown in Figure 12. As shown in Figure 12, 78% of nickel was desorbed from the adsorbent after first cycle and after 6 cycles, there were slight changes in nickel desorption. So, it was concluded that the desired removal of 90% can be achieved after 6 cycles.

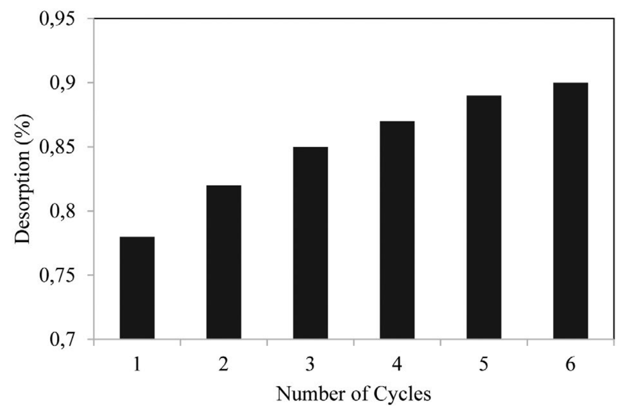

Fig. 12: Desorption of Ni(II) from the adsorbent after six cycles

4. Conclusions

Fe_3O_4 nanoparticles modified by oak shell was used as an applicatory bio-adsorbent for Ni (II) removal from aqueous solution. The effect of pH, adsorbent dosage, temperature, buffer volume, electrolyte and time were studied. The maximum removal of Ni (II) is found to be 93.88% at pH 4.5, temperature of 25 °C, adsorbent dose of 2.6 g/L and contact time of 15 min. The experimental data fitted well with the Langmuir model. Based on Langmuir model, the maximum adsorption capacity of Ni (II) was determined 454.54 mg/g. The capability of adsorbent to remove Ni (II) from aqueous solution efficiently after long cycles (e.g. 6 cycles) was acceptable. In General, resulting tests on the nickel ion removal from effluent using the OSMMN showed the potential applicability of this adsorbent in industrial wastewater treatment.

Conflict of Interests Statement

The authors declare that there is no conflict of interests.

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Povzetek

Študija preučuje odstranjevanje Ni (II) ionov iz odpadnih voda s pomočjo nanodelcev modificiranih z zmletimi želodovimi lupinami (OSMMN). FTIR analiza je pokazala interakcije med karboksilnimi skupinami prisotnimi na OSMMN in Ni (II) ioni. Morfologija magnetnih nanodelcev prekritih s prahom želodovih lupin je bila analizirane s pomočjo TEM. Preučevan je bil tudi vpliv kontaktnega časa, količine adsorbenta, pH vrednosti raztopine in začetne koncentracije Ni (II) ionov na učinkovitost adsorpcije. Eksperimenti so pokazali, da je maksimalna, 93.88 % adsorpcija dosežena pri pH vrednosti 4.5, koncentraciji adsorbenta 2.6 g/L, temperaturi 25 °C in kontaktnem času 15 min. Ravnotežne vrednosti adsorpcije so poskusili opisati tako z Langmuirjevo kot tudi s Freundlichovo izotermo, pri čemer se je izkazalo, da je boljše ujemanje doseženo z Langmuirjevo izotermo. Maksimalna ocenjena kapaciteta OSMMN s pomočjo Langmuirjeve izoterme je znašala 454.54 mg/g, kar je znatna količina.