

Nano zero-valent iron supported on activated carbon: Effect of AC/nZVI ratio on removal of nickel ion from water

Ulucan-Altuntas K.*, Debik E. and Gungor S.

Yildiz Technical University, Environmental Engineering Department, Istanbul, Turkey. E-mail:

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*to whom all correspondence should be addressed: e-mail: kulucan@yildiz.edu.tr

Abstract

The present study investigated the possible effect of contact time, initial nickel concentration, and adsorbent concentration on removing Ni (II) ion, which is toxic to living organisms, from the aqueous media using nano zero valent iron (nZVI) and nano zero valent iron coated powdered activated carbon. A large portion of the nickel removal was achieved in a contact time of five minutes. A removal efficiency of barely 24% was able to be achieved only using activated carbon, while the nickel removal was achieved by approximately 80% using 200 mg/L nZVI. On the other hand, the efficiency was observed to increase up to 99% even with the particle containing the lowest nZVI in the experimental studies using the AC-nZVI nanoparticles containing AC at a rate of 25%, 50% and 75%. Adsorption capacity was increased from 125 mg/g and 820 mg/g for activated carbon and nZVI, respectively to 1190 mg/g for 50% AC-nZVI. Based on our study results, the effect of nZVI is promoted when nZVI is synthesized together with activated carbon. The Freundlich model was also found the best to represent the isotherm modeling made for nZVI.

Keywords: Nano Zero Valent Iron, Activated Carbon, Adsorption, Nickel Removal, Isotherm

1. Introduction

Nanotechnology is a popular topic which we come across in our daily life and has been increasingly used in environmental engineering practices. Although the focus has often been on soil remediation regarding the use of nanoparticles for the environmental pollution control, the increasing number of studies on water pollution suggests that nanotechnology would be also commonly used in the future in this respect. Several studies conducted specifically on removing metal contamination and phenol-containing compounds using nanomaterials have demonstrated that zero valent iron is effective in stabilizing and removing pollutants by means of its high reduction feature (Jegadeesan *et al.*, 2005; Zhu *et al.*, 2009; Li *et al.*, 2006b; Pradeep and Anshup, 2009; Boparai *et al.*, 2011; Kurniawan *et al.*, 2012; Huang *et al.*, 2013).

Nanoparticles have a greater surface area than conventional adsorbents by having higher surface area (Li

et al., 2006b; Pradeep and Anshup 2009; Ambashta and Sillanpaa, 2010; Ulucan *et al.*, 2013). They are noticeable with certain advantages such as providing an effective treatment even in very low concentrations on account of their nano-size pores and being associated with a lower amount of waste (Pradeep and Anshup, 2009; Ambashta and Sillanpaa, 2010; Kurniawan *et al.*, 2012).

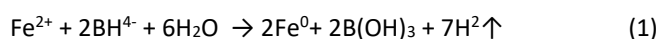
Nickel is a toxic heavy metal causing lung and nasal cancer, which is used in electroplating, silver refineries, coin manufacturing and several industrial products such as stainless steel and magnet (Jegadeesan *et al.*, 2005). It is an important pollutant for the industrial plants where such products are manufactured. In the literature, several methods such as adsorption (Hasar, 2003; Yavuz *et al.*, 2003; Wang *et al.*, 2010), membrane filtration (Gabor and Endre, 2009) and electroflocculation (Sun *et al.*, 2009) have been performed to remove Ni²⁺ ions from the wastewater. Although conventional activated carbon is often used for the removal of heavy metals as well as the natural substances such as kaolinite, bentonite and chitosan, the adsorption capacity of such materials is very low at low pollutant concentrations (Yavuz *et al.*, 2003; Ayari *et al.*, 2007; Vijaya *et al.*, 2008). Many studies have reported that nano zero valent iron (nZVI) with a higher adsorption capacity is effectively used at low concentrations to remove chrome and arsenic as heavy metals (Geng *et al.*, 2009; Zhu *et al.*, 2009; Shi *et al.*, 2011; Kakavandi *et al.*, 2014; Xu *et al.*, 2014).

The present study aimed to reduce the cost by coating activated carbon with nZVI both for increasing the adsorption capacity of nZVI and decreasing the optimum nZVI amount to be used. For this purpose, the effects of different pH values, different adsorption capacities, different initial nickel concentrations and adsorbent concentrations were examined to investigate the adsorption capacities of nZVI and nZVI coated activated carbon (AC-nZVI) and, then, the best alternative was studied by comparing the results of the adsorption studies with AC, nZVI and AC-nZVI.

2. Materials and methods

2.1. Synthesizing of nZVI and AC-nZVI

There are several ways to obtain nano-size zero valent iron (nZVI). The borohydride method is among the most common methods (Li *et al.*, 2006a; Li *et al.*, 2006b; Zhu *et al.*, 2009; Xu *et al.*, 2014). In this method, Fe⁺² and Fe⁺³ ions react with highly reductive sodium borohydride and are reduced to zero valent iron. This method is simple and enables to obtain nZVI with only two common reactants without any other specific chemical requirement (Boparai *et al.*, 2011) nZVI is generally spherically shaped and has a core-shell structure. The reduction process occurs as per the Equation 1 (Huang *et al.*, 2013).



For synthesizing nZVI used in the experimental studies, FeSO₄·5H₂O, NaBH₄ at calculated amounts as per the Equation (1) were used and PEG-2000 was used to prevent aggregation. All chemicals used in the study are of technical quality (Merck). For preparing nZVI, the iron sulfate solution was dissolved in the ultra-pure water:ethanol (30:70 v:v) mixture. The NaBH₄ solution prepared using ultra-pure water is added to the iron sulfate solution at a rate of 30 mL/min. Stirring is continued until the formation of H₂ gas is completed. The resulting mixture is washed with ethanol and pure water and stored in ethanol until it is used.

Activated carbon used to produce nZVI-coated activated carbon (AC-nZVI) is purchased by TARKIM which particle size is 2.0 mm and specific area is 1100m²/g. Before production, activated carbon was grinded to powdered form and was kept in 250 mL 5% nitric acid solution for 1 day to clean it. The activated carbon was washed with ultra-pure water and ethanol for removing the acid and was kept in the oven (105 °C) for one night to lose its water content. In the present study, AC-nZVI containing three different ratios of AC (25%, 50%, 75%) was produced. For this purpose, synthesized amount of nZVI is calculated theoretically as given in Equation (1). Mass amount of activated carbon was calculated in order to contain 25%, 50%, 75% rate in total amount of theoretical nZVI amount and activated carbon mass. Calculated mass amounts of AC added to FeSO₄·7H₂O solution. PEG 2000 was also used to inhibit aggregation. Subsequently, the procedures used in the nZVI production were conducted to produce AC-nZVI.

2.2. Experimental Studies

NiCl₂·6H₂O (MERCK) was used to prepare the synthetic nickel samples used in the experiment sets. Basically, the experiment sets were conducted by shaking 100 ml nickel sample, which were taken into Erlenmeyer flasks, at 200 rpm and constant temperature (20 °C) using GallanKamp brand Incubator & Orbital Shaker after the addition of the specified amounts of adsorbent type. After stirring for a specified amount, the experiment sets were centrifuged at 3000 rpm for 10 minutes using ALLEGRA X-12 brand centrifuge for separating nanoparticles from the solution media. The initial and effluent nickel analyses of the synthetic samples were made using ICP-OES.

2.3. Isotherms

In order to describe adsorption process, adsorption isotherms were applied. For calculation of adsorption capacity, Equation (2) is used. When the temperature is constant, this function is equals to the following equation:

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \quad (2)$$

where, q_e: Adsorption capacity (mg/g), C_i: Initial nickel concentration (mg/l), C_e: Effluent nickel concentration (mg/l), V: Sample volume (l), m: nZVI or AC/nZVI amount (g).

The equations commonly used to assess the experimental isotherm data were developed by Freundlich and Langmuir and named after them. One of the most commonly isotherms, the Freundlich isotherm is expressed as follows:

$$q_e = K_f C_e^{1/n} \quad (3)$$

where, q_e: unit mass substance adsorbed by unit mass adsorbent (mg/g), K_f: Freundlich capacity factor (mg/g), C_e: Effluent nickel concentration, 1/n: Freundlich density parameter.

The Langmuir isotherm, in turn, is expressed as follows:

$$q_e = \frac{K_L C_e}{1 + a_L C_e} \quad (4)$$

where, q_e: unit mass substance adsorbed by unit mass adsorbent (mg/g), C_e: Nickel concentration remaining in the solution after adsorption, a_L and K_L empirical coefficients.

3. Results and discussion

Within the scope of the present study, the optimum pH and AC-nZVI amount were analyzed and the effect of different initial nickel concentrations on removal efficiency was investigated. The experimental data obtain were used to develop isotherm modeling.

3.1. nZVI and AC-nZVI Characteristics

As mentioned in the nZVI and AC-nZVI production, the TEM images of the produced nanoparticles are presented in Figure 1. Figure 1a shows that the particle size of nZVI varies from 50 to 100 nm with a spherical and chain structure. Figure 1b in turn, presents the TEM image of 50% AC-nZVI obtained as optimum in the present study and shows the chain-structure nZVI nanoparticles adsorbing on activated carbon. The calculated size of nZVI adsorbing on activated carbon was 50 nm.

3.2. Effect of pH on Removal Mechanism

To establish the pH in which the optimum removal was achieved, samples of 50 mg/L nickel solution were prepared with pH of 2 to 12. For each pH value, 100 mL was taken from the samples, and samples were put into Erlenmeyer flasks so that there would be 100 mg/L nZVI and shaken for 30 minutes using a shaker (20 °C) and at 200 rpm which is selected for complete contact with the

adsorbent. Removal efficiencies and the effluent pH values of samples are presented in Figure 2.

At lower and higher pH values, adsorption of heavy metals can be decreased (Zou *et al.*, 2016). At lower pH values, because of the increase in nZVI corrosion, adsorption of

metal ions are low. According to Figure 2, the removal efficiency was 5% at pH 2 and reached to 50% removal efficiency at pH 5. At lower pH values, surface of nZVI will be positively charged and this caused to adsorption of nickel ions unfavorable (Zou *et al.*, 2016)

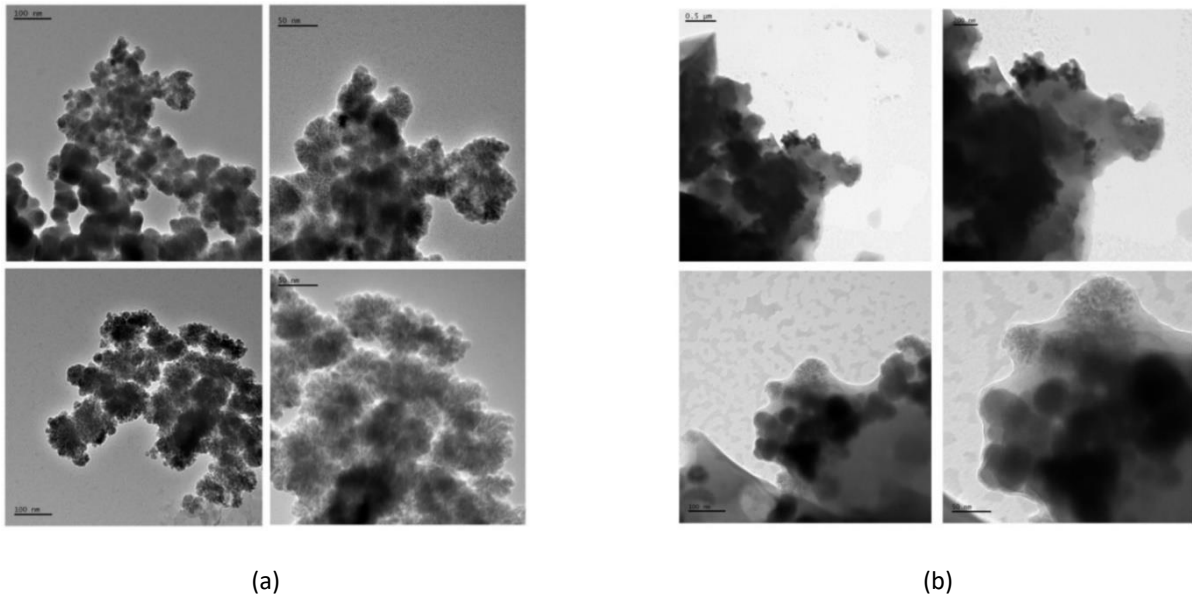
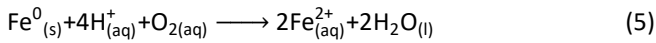


Figure 1. a) The TEM image of nZVI b) The TEM image of 50%AC-nZVI

The effluent pH values were exhibited increment to the isoelectric point and nZVI surface was oxidized due to the reaction of the samples with a pH under isoelectric point (pH≈8 (Sun *et al.*, 2006)) as per the Equation 5 (Sun *et al.*, 2006).



The removal efficiencies at pH 7 and pH 8 were similar as 68% and 66% respectively. Minimal effects on removal rate were observed at higher pH. At high pH values, the

effluent pH values were decreased towards to isoelectric point. At higher pH values than pH 8, passive film of iron hydroxide could be arisen with the iron corrosion, and this could be caused to the release of hydrogen ions (Zou *et al.*, 2016). Iron hydroxide could inhibit the adsorption of nickel on nZVI and this could be caused to decrease in removal at high pH. In light of the data that was obtained, a further study was conducted with pH 7, which is also the original pH of the sample.

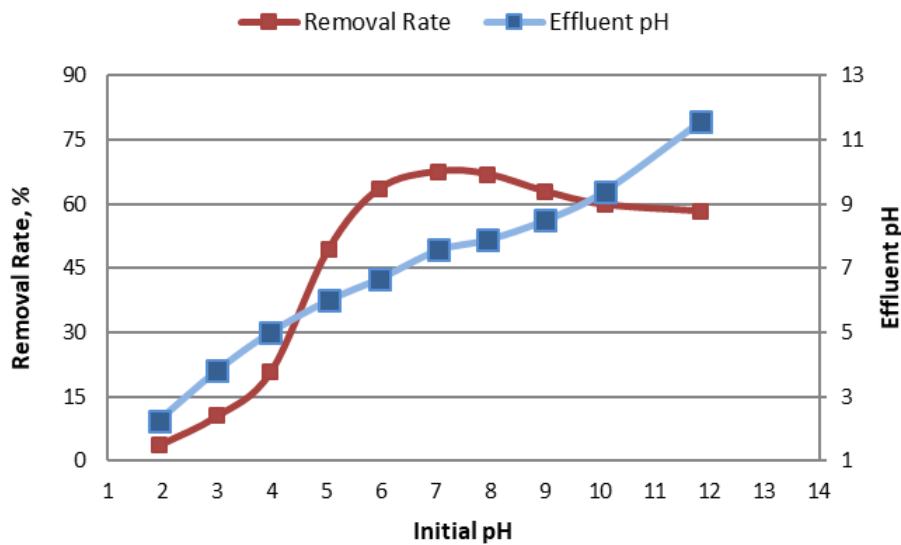


Figure 2. The Effect of Initial pH on Removal Efficiency

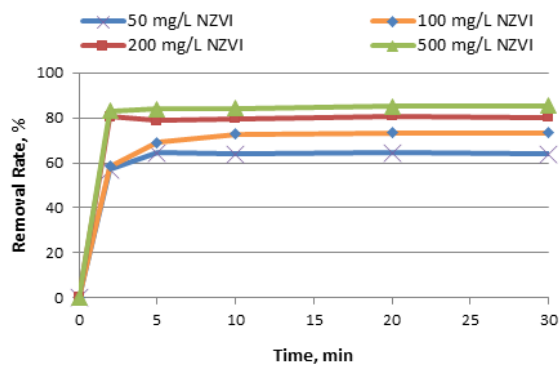
3.3. *The Effect of Adsorbent Amount and Contact Time*

In the present study, 100 mg/L constant nickel concentration was chosen to observe the effect of nanoparticle concentration. Different sets were designed with 50, 100, 200 and 500 mg/l nZVI. The contact time was chosen as 2, 5, 10, 20 and 30 minutes in all sets.

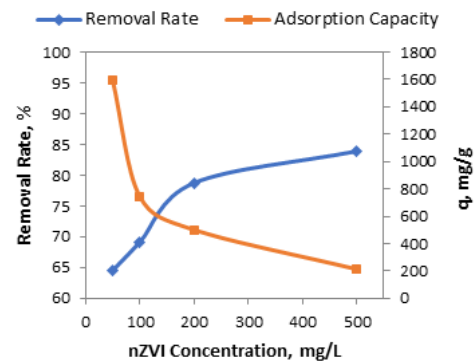
Figure 3a presents different nZVI concentrations and nickel removal efficiencies at different time points from the study conducted with constant 100 mg/L Ni. The graphic shows that the mean efficiency was about 60% in the study with an nZVI concentration of 50 mg/L concentration, while it was 73% at 100 mg/L nZVI concentration, 80% at 200 mg/L nZVI concentration and 85% at 500 mg/L nZVI concentration. It is apparent that the efficiency increases with increasing nZVI concentrations. The efficiency varying between 200 mg/L

nZVI and 500 mg/L nZVI concentration is very low compared to the adsorbent used. In general, it is seen that the efficiency remains constant following a mean contact time of five minutes in all studies. The results were similar with literature (Efecan *et al.*, 2009; Poguberovic *et al.*, 2016; Rathor *et al.*, 2017; Ulucan-Altuntas *et al.*, 2017)

Figure 3b shows varying removal efficiencies and adsorption capacities after a contact time of five minutes in 50, 100, 200 and 500 mg/L initial concentrations. The removal efficiency of the nZVI concentration increasing up to 200 mg/L within the same contact time increases almost linearly, while it continues at a low angle after 200 mg/L nZVI concentration. On the other hand, the adsorption capacity decreases as the adsorbent amount increases.



(a)



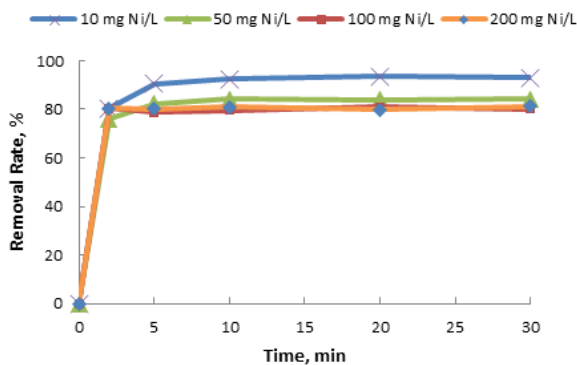
(b)

Figure 3. a) Efficiency graph based on nZVI concentration b) The effect of different nZVI concentrations on the nickel removal efficiency

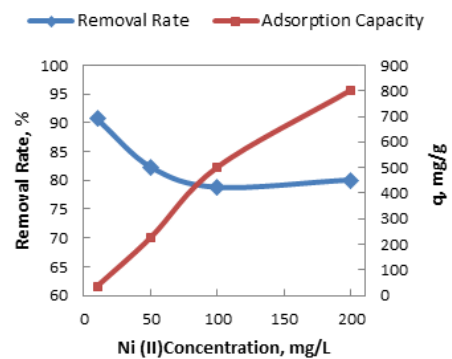
3.4. *The Effect of Different Initial Nickel Concentrations*

To establish the maximum nickel concentration to be adsorbed by the nanoparticle in the study, samples were prepared at different nickel concentrations and contact times were changed as the variable in all sets in similar to the previous study. Figure 4a shows the removal efficiencies for the samples containing 10, 50, 100 and 200

mg/L at a constant working concentration of 200 mg/L nZVI. It is seen in the graphic that the efficiency is about 90% at 10 mg/L, while it is approximately 80% at 50, 100 and 200 mg/L. The graphic also demonstrates decreased efficiency with increasing nickel concentrations. Besides, the efficiency is observed to remain constant after a contact time of about five minutes.



(a)



(b)

Figure 4. a) The effect of different initial nickel concentrations on removal efficiency b) The removal efficiency and adsorption capacities in different initial nickel concentrations

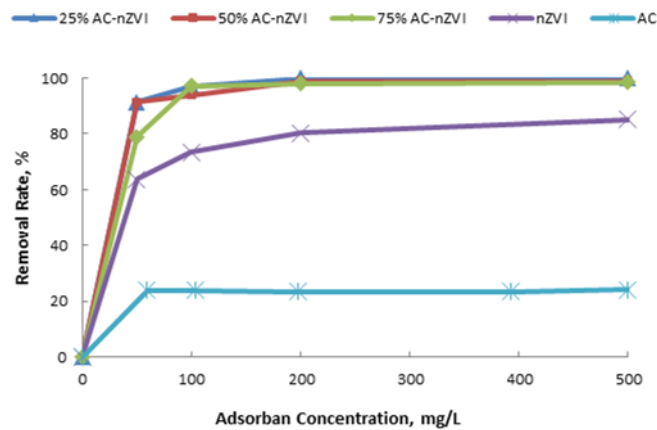
Figure 4b shows varying removal efficiencies after a contact time of five minutes in 10, 50, 100, and 200 mg/L initial concentrations. It is also clear that the efficiency decreases at increasing nickel concentrations within the same contact time. Obtained results were similar with literature (Efecan *et al.*, 2009; Poguberovic *et al.*, 2016; Rathor *et al.*, 2017; Ulucan-Altuntas *et al.*, 2017).

3.5. Experimental Studies with AC-nZVI Nanoparticle

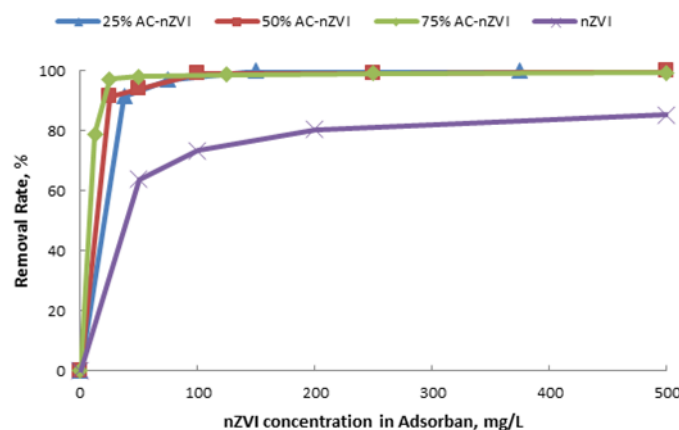
Sets were established to examine the extent to which the AC-nZVI nanoparticles synthesized at different ratios would increase the Ni (II) removal efficiency and to compare the effects of activated carbon alone and nZVI on removal. Figure 5a shows the removal efficiencies with 25%, 50% and 75% AC-nZVI adsorbents, nZVI and activated carbon alone within a 30-minute contact time by using different adsorbent concentrations at a constant working concentration of 50 mg/L. The graphic shows that each nanoparticle type has a removal efficiency of above 99% as at 100 mg/L adsorbent concentration. An efficiency of 80% was achieved even at 50 mg/L adsorbent concentration containing 75% AC-nZVI.

The literature review of the studies on nickel removal using activated carbon reveals that the adsorbent concentration should be 3000 to 5000 mg/L to increase the removal efficiencies above 80% and the removal efficiencies of 50 mg/L adsorbent concentration can only reach 20% (Kadirvelu *et al.*, 2001; Yavuz *et al.*, 2003; Onundi *et al.*, 2010; Wang *et al.*, 2010). In the present study, the removal efficiency was able to reach only 24% for 500 mg/L, whereas only nZVI reached 85%. The adsorbents obtained by the co-synthesis of activated carbon and nZVI produced an efficiency of 99%. The effect of nZVIs produced with activated carbon on removal efficiency is apparent.

In a similar vein, a comparison based on the nZVI amount contained by the nanoparticles containing 25%, 50% and 75% AC-nZVI is presented in Figure 5b. Bases on these data, 50% AC-nZVI adsorbent is sufficient to achieve nickel removal at a rate of 90% with a minimum 50 mg/L adsorbent and a 25 mg/L nZVI amount.



(a)



(b)

Figure 5. a) The effect of AC-nZVI nanoparticles with different ratios of nZVI on removal efficiency b) The varying removal efficiencies in different nZVI concentrations within the adsorbent

Figure 6 shows the adsorption capacities of AC-nZVI particles in comparison with activated carbon and nZVI. For 50 g/L adsorbent concentration, activated carbon adsorption capacity is about 125 mg/g, while it is 820, 1000, 1190 and 1170 mg/g for nZVI and 25%, 50% and 75% AC-nZVI, respectively. It is seen that the adsorption

capacities increase in direct proportion to the nZVI content. It is observed a small portion of decreased at 75% AC-nZVI. Based on highest adsorption capacity belong to 50% AC-nZVI, optimum activated carbon ratio of 50% can be regarded as optimum ratio.

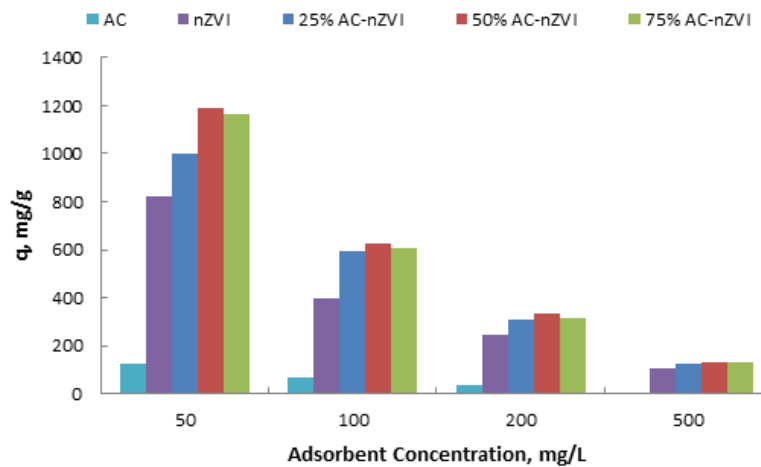


Figure 6. Adsorption Capacity Difference between AC, nZVI and AC-nZVI with different ratios of nZVI

3.6 Isotherm Modelling

The adequacy of the data on nZVI from the studies for the Langmuir Isotherm and the Freundlich Isotherm, which are the most common in the literature (Ajmal *et al.*, 1998; Hasar, 2003; Onundi *et al.*, 2010), was evaluated. Table 1 presents the coefficients obtained in the study conducted to find the Langmuir and Freundlich Isotherm coefficients. Based on these data, the correlation coefficient for nZVI was found to be more adequate for the Freundlich Isotherm and the equation produced is given in the Equation 6.

$$q_e = 52.044 C_e^{0.721} \quad (6)$$

The Freundlich isotherm indicates that the surface of the adsorbent is heterogeneous, while the Langmuir isotherm shows that the surface of the adsorbent is homogeneous (Ajmal *et al.*, 1998). The obtained results show that the surface of synthesized nZVI is heterogeneous.

Table 1. Isotherm Coefficients for NZVI

Langmuir Model		Freundlich Model	
α_L	0.0342	K_f	52.044
K_L	42.735	n	1.387
R^2	0.7446	R^2	0.9963

4. Conclusions

According to the results of the nickel removal study conducted with nZVI, there is an increased efficiency in response to increasing nZVI concentrations; however, there were no significant changes for the removal efficiency after five minutes. As expected, efficiency reduces with increasing nickel concentration; however, a removal efficiency of 80% is achieved even at 200mg/L nickel concentration.

In the study conducted with AC-nZVI particles, which were produced to contain AC at the rates of 25%, 50% and 75% and used to determine the removal efficiency of the nanoparticles obtained by coating activated carbon with nZVI nanoparticle to increase the adsorption capacity, the removal efficiencies were found to be above 99%. An efficiency of approximately 80% was achieved even at 50 mg/L adsorbent concentration containing 25% AC-nZVI, while the efficiency was above 90% in all other studies.

In the present study, the Ni (II) removal efficiency could only reach 24% for activated carbon at 500 mg/L adsorbent concentration. Likewise, only nZVI was able to reach 85%, whereas the adsorbents obtained by the co-synthesis of activated carbon and nZVI produced an efficiency of 99%. It suggests that the effect of nZVI produced with activated carbon on removal efficiency is apparent.

In conclusion, in the study conducted with 25%, 50% and 75% AC-nZVI particles, nZVI nanoparticle and AC, the removal efficiency was 84% with nZVI, while it was 99% with nZVI coated activated-carbon adsorbents. The activated carbon was demonstrated to increase the effect of nanoparticle in the Ni(II) removal. Based on our study results, 50% AC-nZVI adsorbent at 50 mg/L concentration is sufficient to achieve the nickel removal at a rate above 90% with minimum adsorbent and adsorption capacity of 1190 mg/g.

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