The use of natural zeolite to remove heavy metals Cu (II), Pb (II) and Cd (II), from industrial wastewater

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The use of natural zeolite to remove heavy metals Cu (II), Pb (II) and Cd (II), from industrial wastewater

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Abstract: In this study, the adsorption behavior of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ from synthetic metal solutions using natural zeolites was studied in order to investigate the efficiency of adsorbents of heavy metals from industrial wastewater. The kinetic study indicated the suitability of the zeolite for the removal of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions from synthetic wastewater; batch experiments were used to identify the effect of parameters that affect the rate of adsorption such as the adsorbent mass, the initial solution concentration, the initial solution pH, the adsorbent particle size, and the agitation speed and evaluated their impact on the removal efficiency of heavy metals from industrial wastewater using the natural zeolite. Zeolite samples with masses between 1 g and 10 g were contacted with constant volume (100 ml) of multicomponent synthetic solutions containing Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions. They were agitated at agitation speeds in the range of 100–300 rpm for agitation times from 1 hr to 8 hrs in a magnetic stirrer at room temperature, the pH values were monitored and adjusted regularly. Every hour, 15 ml of the samples was analyzed using the Atomic Absorption Spectroscopy. The results show that the removal efficiency of Cu$^{2+}$ increase from 60% for 1 gr to 99% for 10 gr of the mass of absorbent, increase from 62% for 1% to 94% for 7 of the initial solution pH, increase from 90% for 100 rpm to 94% for 300 rpm of the agitation speed, the amount...
adsorbed increase from 0.5 mg/g for 100 mg/l to 2.1% for 400 mg/l of the initial solution concentration. Similar results are obtained for the two other heavy metals (Pb2+, Cd2+) and showed that the capacity of the adsorbents for the removal of heavy metals is directly proportional to the mass of absorbent, initial solution pH, agitation speed, and initial solution concentration. The highest adsorption rate of Cu2+, Pb2+ and Cd2+ ions took place in the first hours followed by a slower adsorption rate later on.

Subjects: Chemical Engineering; Water Engineering; Water Science

Keywords: zeolite; heavy metals; copper; lead; cadmium; wastewater; kinetic

1. Introduction
The presence of accumulated heavy metals in the environment constitutes a serious threat to human life and the environment due to their toxicity. Cu2+, Pb2+ and Cd2+ are the common metals that tend to accumulate in organisms, causing numerous diseases (Ahali Abadeh & Irannajad, 2017; Buashia et al., 2020; Salman et al., 2017). Several techniques such as adsorption, extraction, phytoextraction, ultrafiltration, reverse osmosis, electrodialysis, ion-exchange, and membrane processes are proposed for the handling of wastewater pollution and removing heavy metals from industrial wastewater required high energy or special operational requirements (Ayoub et al., 2016; Ebrahimi et al., 2014; Kwon et al., 2010). The selection of alternative low-cost materials such as sorbents for the removal of heavy metals has emphasized. Recently and the adsorption method using zeolite is considered to be an appropriate method for wastewater handling because of its cost-effectiveness and simplicity (Gupta et al., 2009; Scharnberg et al., 2020).

The efficiency of zeolite for the removal of three heavy metal Cu2+, Pb2+ and Cd2+ ions from a synthetic solution was considered. These three ions were chosen as abundance more pollutants that are present in industrial wastewaters.

The first part discusses the experimental materials and methods used for the adsorption of heavy metals from synthetic wastewaters. The experiments included are the most widely used techniques for the batch system. The second part describes the kinetic studies in order to investigate the behaviour of adsorbent and understand the removal mechanisms involved in the adsorption process, in this part, a number of these factors that affect the efficiency of natural zeolite in removing of Cu2+, Pb2+ and Cd2+ from solution are investigated in detail. In conclusion, the specific aims of this study are reviewed to present an overview of the conclusions of this study and to make a critical evaluation and discusses the recommendations for further work.

2. Materials and methods
The experiments have focused on the efficiency of zeolite for the removal or reduction of Cu2+, Pb2+ and Cd2+ ions from synthetic industrial wastewater to achieve allowable limits. Synthetic multi-component solutions of Cu2+, Pb2+ and Cd2+ ions were prepared.

In all experiment stages deionised water was used, which had almost all of its mineral ions removed. The pH of the starting solution was pH 5 to get the best adoration rate. The pH values were monitored and adjusted using a pH meter (Thermo Scientific Orion Star A111 Benchtop). The pH was adjusted to as 1, 5, and 7, respectively, by adding hydrochloric acid or bases sodium hydroxide. To observe the effect of agitation speed; agitation in a beaker was obtained by using a magnetic stirrer (IKA, Germany, RH basic 2, 3,339,000) at a speed of 100 rpm, 200 rpm and 300 rpm. The mesh sizes of the zeolite particle used in this study was 100 µm < dp < 200 µm, unless stated. A Thermo Scientific model PR305220G, Gravity Convection stainless steel universal oven was used to dry and heat the samples.
Zeolite samples with masses 1 g, 5 g and 10 g were contacted with constant volume (100 ml) of multicomponent synthetic solutions containing Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions. They were agitated at agitation speeds of 100, 200 and 300 rpm for agitation times from 1 hr to 8 hrs in a magnetic stirrer at room temperature, the pH values were monitored and adjusted regularly. Every hour, 15 ml of the samples was filtered and taken for metal ion concentration analysis using the Atomic Absorption Spectroscopy (AA6800Shimadzu) with oxygen air-acetylene flame analysis technique was used to analyse the digested solutions, the wavelength range was between 190 and 900 nm. A flame temperature is continuously adjustable between 2300°C and 2950°C, which makes it possible to choose the best atomization temperature for different elements. Integrated flame/graphite furnace atomization system, changeable with flame emission burner, was involved. Hollow cathode lamps were used as sources of radiation and the background correction was provided by a deuterium lamp.

The experiments were duplicated four times in order to examine the reproducibility of the results, while the mean value was used for all taken data, the deviation between the duplicate samples in analysing the cations was ± 5.1%, 4.5% and 5.1% for Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions, respectively.

3. Results and discussion
The adsorption of heavy metal cations from solution using zeolite materials was affected by several factors; examples of such factors were the adsorbent mass, initial solution concentration, initial solution pH, agitation speed and adsorbent particle size. The effect of these factors on the efficiency of zeolite in removing Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ from solution will be investigated in order to determine the behaviour of adsorbents and understand the removal mechanisms involved in the any adsorption experiment in wastewater treatment (Amarasinghe & Williams, 2004; Ennigrou et al., 2015; Myroslav et al., 2006; Inglezakis et al., 2004).

3.1. Kinetic study results
Multicomponent solutions were mixed with zeolite and agitated for an hour at room temperature and the initial concentrations of the mixed component solutions were 100 mg/l for the Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions.

The data obtained from the kinetic adsorption tests were used to determine the removal capacity, $q_e$ (mg/g) of the different adsorbents using the following Equation (1) and (2):

\[ q_e = (C_0 - C_e) \times \frac{V}{m} \]  

Equation (1)

The percentage removal of metal ions from solution was also determined using the equation below:

\[ \text{Removal efficiency(%) } = \frac{(C_0 - C_e)}{C_0} \times 100 \]  

Equation (2)

3.2. Factors influencing the rate of adsorption
A number of parameters influencing the rate of adsorption were studied and described, these parameters include the effect of adsorbent mass, effect of initial solution concentration, effect of initial solution pH, effect of adsorbent particle size, and effect of agitation speed.

3.2.1. Effect of adsorbent mass
The effect of adsorbent mass on the adsorption capacities from solutions was investigated. Three different masses were used in this study, 1 g, 5 g and 10 g of zeolite in 100 ml solutions. The particle size of zeolite used was 200 μm, the pH of the solution was adjusted to 5, the agitation speed was 200 rpm at room temperature; the samples were collected every hour and analysed
using the Atomic Absorption Spectroscopy (AA6800Shimadzu) with flame and graphite techniques. The experiment was carried out at fixed initial metal concentrations.

The results in Figure 1 clearly show that when the adsorbent mass was increased, this resulted in an increase in the adsorption of the heavy metal ions. The main reason for this is that as the adsorbent mass increases, more adsorption sites are available per mass of adsorbent surface and thus the total amount of metal that is removed increases. This result indicates that the removal of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions mostly occurs in the early stage and the mineral mass in the solution can affect the adsorption capacity for the removal of heavy metals as it determines the availability of adsorption sites.

3.2.2. Effect of initial solution concentration

The initial concentration of the solution significantly impacts on the heavy metal removal process, several studies have reported that an increase in the initial concentration resulted in an improvement in the adsorption capacity at the initial stage and a decrease in the overall heavy metal removal efficiency, which depends on the availability of active sites (Coruh & Ergun, 2009; Ören & Kaya, 2006).

The effect of the initial metal concentration on the efficiency of zeolite in the removal of heavy metals from the solution was determined using multicomponent solution concentrations ranging from 100 to 400 mg/l. 100 ml solutions at pH 5 were contacted with 5 g of zeolite samples of size 200 μm, agitation speed of 200 rpm was used at room temperature.

The results in Figure 2 show that generally any increase in the initial solution concentration results in an increase in the heavy metal removal efficiency and the rate of adsorption. As the initial concentration increases, the driving force increases, which in turn leads to an improvement in the efficiency of the heavy metal removal process. Then, after the system reaches equilibrium, the initial solution concentration does not show any significant change due to a decrease in the number of active sites (Ibrahim & Mutawie, 2013; Ibrahimi & Sayyadi, 2015; Taamneh & Sharadqah, 2017; Visa & Popa, 2015).

3.2.3. Effect of initial solution pH

The initial solution pH parameter has a significant impact on heavy metal removal processes since it can impact on adsorbent ability to remove metals with the competition of hydrogen ions with heavy metal cations for active sites on the adsorbent surface (Dimirkou, 2007; Inglezakis et al., 2002). An acidic solution can impact on both the character of the exchanging ions and the character of the adsorbent.

Three different pH values of initial solution were preferred 1, 5 and 7 for the multicomponent solutions, the pH of the solution was adjusted using sodium hydroxide solution and hydrochloric acid solution. Thus, 100 ml of the solution samples were in contact with 5 g of zeolite for 1 hour, the particle size of zeolite used was 200 μm, the agitation speed was 200 rpm at room temperature, samples using fixed metal concentrations of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ cations were collected every hour and analysed using the Atomic Absorption Spectroscopy (AA6800Shimadzu) with flame and graphite techniques.

The results are presented in Figure 3, shown that the solution pH increases, the heavy metal removal efficiency also increases. This is due to the competition between the hydrogen ions and heavy metal cations for the same exchange sites and electrostatic repulsion between the heavy metal cations in solution; as more hydrogen ions are adsorbed, the number of protonated zeolite surfaces increases (Alvarez-Ayuso et al., 2003; Hamedreza et al., 2015; Moreno et al., 2001; Wingenfelder et al., 2005).
Also, the results show that the ion exchange process increases with an increase in pH up to a maximum value. It can be concluded that the best heavy metal removal efficiency value was obtained between pH values of 5 and 7, while pH values below pH = 5 or above pH = 7 was found to decrease the heavy metal removal efficiency. These results are in agreement with outcomes obtained from the literature review (Argun, 2008; DalBosco et al., 2005; Mier et al., 2001).
3.2.4. Effect of adsorbent particle size

The effect of adsorbent particle size on adsorption process was investigated: two different sizes were used, 100 and 200 μm. 5 g of zeolite was mixed with 100 ml solution of the appropriate multicomponent solution at an agitation speed of 200 rpm, the pH of the solution was adjusted to 5 and samples were collected every hour, then filtered to remove solids and analysed using the Atomic Absorption Spectroscopy (AA6800 Shimadzu) with flame and graphite techniques. This experiment was carried using fixed initial metal concentrations.

The results depicted in Figure 4 show that the smaller particle-sized samples adsorbed more of the Cu\(^{2+}\), Pb\(^{2+}\) and Cd\(^{2+}\) ions and indicate that any decrease in adsorbent particle size causes an increase in the adsorption of the heavy metals ions. This is because as the adsorbent particles get smaller more adsorption sites are available for metal uptake and more contacts are taking place. On the other hand, smaller particle sizes result in the shortening of the diffusion distance that ions have to travel in order to get to an active site. This is in agreement with the results obtained from literature review (Erdem et al., 2004; Malliou et al., 1994; Myroslav et al., 2006), they concluded that the larger particle size adsorbent had lower adsorption capacities than the smaller particle sizes.

The particle size can affect the adsorption capacity mostly at the initial stage. As the contact time increases, in the level of the effect of particle size on adsorption decreases, and as a result, the adsorption process gets slower and the use of very fine particles can also cause some operational problems such as difficulty in the filtration of the zeolite from solution in batch studies (Erdem et al., 2004; Malliou et al., 1994; Inglezakis et al., 2001).

3.2.5. Effect of agitation speed

The effect of agitation speed on the removal of the cations heavy metal from the solution by zeolite was determined using a magnetic stirrer at a speed of 100, 200 and 300 rpm, the mixture was contacted with 5 g of zeolite samples and agitated, the particle size of the zeolite samples used was 200 μm at room temperature, the pH was adjusted to 5. The samples were collected every hour and analysed using the Atomic Absorption Spectroscopy (AA6800Shimadzu) with flame and graphite techniques and the results are presented in Figure 5.

The results show that the metal removal efficiency increased as the speed of agitation increased, Agitation of the mixture results in abrasion and the production of more broken zeolite particles. This means that fresh smaller size zeolite particles are produced and more activate sites are available on the surface (Khachatryan, 2014; Mehdizadeh et al., 2014). Thus, this phenomena leads to an increase in the surface area, which significantly improves the efficiency of the heavy
metal removal from solution. This is in agreement with the results obtained by Ören and Kaya (2006), they concluded that an increase in the speed of agitation resulted in higher adsorption capacities and the agitation helps in overcoming the external mass transfer resistance, which controls the rate of adsorption.
4. Conclusion
Kinetic studies and experiments were carried out in order to investigate the impact of several parameters on the efficiency of zeolite for the removal of heavy metals from industrial wastewater, the results obtained may be summarized as follows:
The highest adsorption rate of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions took place in the first hours followed by a slower adsorption rate later on. The first hour is an initial stage of adsorption when higher rates of adsorption take place; this may be due to the availability of more adsorption sites and the fact that the metal ions exchange easily on the surface of the zeolite grains. After that, a slower adsorption rate follows due to slower diffusion of the metal ions into the interior channels.
The study indicated the suitability of the zeolite used for the removal of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions from synthetic wastewater, while considering the economic aspects of wastewater treatment.

The adsorbent mass, adsorbent particle size, initial solution pH, initial solution concentration and agitation speed are usually the most influential parameters.

The efficiency of heavy metal removal was enhanced with increased initial solution pH, increased agitation speed, increased solution concentration, decreased particle size and greater mass of absorbent.

The adsorption is a heterogeneous process as the removal rate of Cu$^{2+}$, Pb$^{2+}$ and Cd$^{2+}$ ions mostly occurred early on, but as the contact time increased, there was a decrease in the level of the effect of the parameters on adsorption and the adsorption process became slower.

The recent studies show that precipitation affects the rate of removal and the quantities of heavy metals in solution and the adsorption capacity of zeolite that has been regenerated using NaCl is either the same or it changed slightly.

The natural zeolite is promising sorbent due to its availability and its low cost and can substitute effectively the use of activated carbon as adsorbent for the removal of metal cations from wastewater.

Nomenclature and units

- m: The mass of the zeolite used (mg),
- V: The aqueous volume (L),
- $C_i$: The initial metal ion concentrations in the samples (mg/L),
- $C_e$: The equilibrium metal ion concentrations in the samples (mg/L),
- $q_e$: The mass of heavy metal uptake per unit of natural zeolite (mg/g).

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Disclosure statement

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