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Adsorption of lead ions from aqueous solution using local natural zeolite

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ABSTRACT

The present study investigated the adsorption of lead ions (Pb^{2+}) from aqueous solutions using natural Clinoptilolite zeolite. The variables that were studied in this process and during the examinations include contact time, pH, adsorbent dose, and initial lead ion concentration, and primary concentration of the solution of lead. To determine the chemical composition of zeolite, X-ray fluorescence analysis was applied, Also Adsorption isotherms and kinetics in constant temperature and stirrer speed of 250 rpm has been examined also. The results showed that the optimal dose was 5 mg/g which give high removal efficiency of 83.3%. However, with the increase of metal concentration, While the removal is a significant issue in the field of health community is a significant issue in the field of health community val efficiency decreased with increasing metal concentration, the adsorption capacity was enhanced. Also, the Langmuir model showed better fit for the adsorption of lead ($R^2=0.8658$) in comparison to other isotherms . The findings of this study suggest that natural zeolite Clinoptilolite can be considered as an efficient and inexpensive adsorbent for the removal of a heavy metal like lead from aqueous solutions.

1. Introduction

The lead concentration in raw natural waters ranges from 0.4 to 0.8 mg/L.1, and the lead ions concentration in the wastewaters streams (especially industrial effluents) may approach (200–500) mg/L; but the texts reports of the world Health Organization (WHO) state that the maximum permissible limit (MPL) of lead must not exceed 0.05 mg/L in drinking water [24, 25, 35] .

Therefore, lead removal or controlling lead concentration in the aqueous solutions is a significant public health issue. The health effects of lead ions primarily target the nervous system and cause sever and irreversible damages to the brain [1].

Health effects of lead toxicity on human body are detected based on the level of lead in the blood samples 20. Lead poisoning also can causes hypertension, renal impairment , anaemia, toxicity and immunotoxicity to the reproductive organs, and it is believed that the behavioural and neurological effects of lead are to be irreversible [28].

The various well-known chemical and physicochemical methods of removing heavy metals especially the lead ions, include chemical precipitation, adsorption, solution concentration, ultrafiltration, and ion-exchange [35].

For each method, there are some associated advantages as well as limitations such as being uneconomical and lack of high-efficiency regarding the removal of pollutants existed at extremely low amounts.

To reduce the pollution of lead ions, many methods have been used such as adsorption, ion exchange, coagulation, precipitation, ultrafiltration, ion-exchange , reverse osmosis, activated carbon adsorption [9], electro-dialysis and electro-coagulation [4 , 5, 7]

The using bisulphite, lime, is ineffective technique for precipitation the lead ions at low concentration [10]

The precipitation technique is considered as expensive method because its need adding other chemicals which also lead to generate high percentage of sludge content the matter that raise the total cost of this method [10,24].

Electro-coagulation is an electrochemical method, makes the dissolved ions precipitate by applying an electrical current and neutralized the solution ions by opposite electrical charges that make it precipitate directly, this approach represent good choice for removing nitrate from waste water [26] and dyes from colored effluents [6].

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coupling Electro-coagulation with some physical processes (such as, activated carbon, sand filtration, and sedimentation) gives a feasible method for removing the pollutants from wastewater such as linear alkylbenzene sulfonate,phenol [16],

But unfortunately, this approach is expensive where it requires electrodes to supply the solution with the current where the electrodes exposed to electrodes wearing and electrodes tearing because of a lot of strain on it [27].

Combination adsorption with other treatment methods was subject of many research papers

Edison et al [12] worked on alternative methods for treatment wastewater taken from industrial facility located in Medellín (Colombia), they found that application of Electrocoagulation plus Photo-Fenton (PF) processes were not fully effective for the removal of acute toxicity

Edison et al [12] noticed that adding activated carbon adsorption as additional step will reduces the acute toxicity to 10% and 0% .

Gameel Abdulla et al [3] used limestone to remove iron from iron aqueous solution. They completely removed iron ions from the prepared solution by using activated carbon and limestone (1:1 ratio) [3].

Therefore adsorption method can be represent as the key function for removing pollutants from streams [12].

The adsorption method generally has many advantages and disadvantages [19], but the disadvantages of this method can be less impactful and less effective if compare with the advantages [14]. the low cost, high performance, wide pH range, and easy operation makes the adsorption approach a good choice for removing lead ions from WWTP [19].

The use of natural absorbent materials for reducing the amount of the pollutants in the aqueous solution is prefer as a compare with other treatment methods where The using of natural material doesn't produces negative environmental impacts or not expensive process [2].

Fatemah S. Abdulraheem et al used straw and reed to produce natural absorber cell for removing iron ions from water, this cell was absorbed 33.1% of iron [2].

Therefore the using of natural absorption having ion-exchange and sorption properties can be considered very good choice for removing the Lead ions from solutions [21].

The clinoptilolite is a natural zeolite [8] , and appears to be more appropriate for lead removal because of it is natural material and its low-cost [21], Furthermore, the applied zeolite can be removed or recycled and the process is cost-effective [21].

Natural zeolite clinoptilolite has a three-dimensional structure and its typical molecular structure is $[(Na, K, Ca)2Al3(Al, Si)2Si13O36\cdot 12H2O]$, [13, 20]. Clinoptilolite has wide uses due to its structural and adsorbent properties, where it utilized as an environmental removing agent, also it was used in many industrial applications and it was adopted for some therapeutic purposes such as antitumor, natural intestinal cleanse, and antioxidant.

Due to the existence of massive zeolite resources in Iraq and the exploitation of these resources in regions such as Akasshat and Sinjar [20], they can be widely used to remove environmental pollutants like heavy metals [8, 18, 25].

The current study explores the efficiency of using natural zeolite clinoptilolite in order to remove the heavy metals, especially lead, from water and wastewater.

2. Material and Methods

Systems Clinoptilolite was purchased from Afrand Toska company. It was crushed, sieved, washed to remove dust and dirt and finally, it was dried at a temperature of 100°C for 24 h until further use.

Chemical properties of the zeolite were determined by the Standard Test Method for Silica content (ASTM D859–16), Standard wet chemical analysis and titrimetric Methods for determine: Fe_2O_3, Al_2O_3, Mgo, CaO, TiO_2, MnO and P_2O_5, and gravimetric methods was adopted for SiO_2 analyzing. Flame photometry was used to determine Na_2O_3 and K_2O. all these results are presented in Table 1.

The preparation of lead stock solution was based on the methods presented by the standard references of the examination of water and wastewater 10. For this purpose, 2.744 g of lead nitrate powder (Pb(NO3)2 with 99% purity) was dissolved by adding about 1000 mL of distilled. To prepare other concentrations such as 10 mg/L up to 100 mg/L, this solution was diluted by different dilution factors.

In this study all the experiments of adsorption tests were carried out in a batch system with 100 mL samples containing lead with concentrations of 10 mg/L, 20 mg/L, 40 mg/L, 50 mg/L, 60 mg/L, 80 mg/L,90 and 100 mg/L. also a zero percent of lead as a blank sample was used to compare with other results .

The adsorption percent was calculated by using the following equation:

$$\%Adsoption = \frac{C_i - C_f}{C_i} * 100\% \tag{1}$$

Where: C_i - is the initial concentration and Cf is the final concentration of the metal ion in initial and final solutions respectively.

The variables that were studied in this process and during the examinations include contact time, pH, adsorbent dose, and initial lead concentration (heavy metal). All experiments were repeated three times to ensure the accuracy and precision. The findings resulted from studying the effect of pH, contact time, primary concentration of metals, also the effect of the adsorbent amount on the adsorption rate are presented in advance alongside the graphs which are created in Excel.

To determine the chemical composition of zeolite, X-ray fluorescence analysis was applied (Table 1 and 2) .

Table 1 . Chemical properties of the zeolite.

Component s	Percent % in 100 g
SiO2	50.74
Al2O3	11.54
Fe2O3	12.72
CaO	1.84
Na2O3	0.64
K2O	2.36
Mgo	3.98
TiO2	1.385
MnO	0.033
P2O5	0.120
S	181 ppm

Table 2 Physical properties of the natural zeolite.

Properties	Value
Color	Light green
Cation-exchange capacity (CEC)	(165-185) meq /100g
Selectivity	Pb^{2+} > Cs^{+} >Cd^{2+} > >k > Ba > Sr3+ > Cr3+ > Cu^{2+} > Co^{2+} > > Zn^{2+} > Ni^{2+}
Mineral %	(80- 90) percent
Specific gravity	1 g/cm3

3. Results and Discussion

The effect of the adsorbent dose on lead adsorption was determined by varying the dose and monitoring the final concentration, adsorption capacity, and lead removal efficiency at different adsorbent doses, pH of 7.0, contact time of 60 minutes, and inlet concentration level of 50 mg/L where all these changes were studied. According to **Figure 1**, by increasing the adsorbent dose, the lead removal efficiency increases with increasing adsorbent dose, stabilizing at a dose of 5 g/L. Therefore, this dose is considered as optimum dose. The removal efficiency and adsorption capacity at this dose are 83.3% and 5 mg/g, respectively. Lead removal efficiency increases at the 5 g/L dose of adsorbent.

According to **Figure 2**, we notice by increasing the pH of the solution, lead removal efficiency increases, but the rate of removal percentage at the pH higher than 8.0 increases with a slower pace. The removal efficiency and adsorption capacity at this pH are 80.1% and 5.22 mg/g, respectively. Therefore, in this study an optimum pH of 8.0 was achieved.

In order to determination the effect of adsorbent dose on the adsorption of lead

Samples were prepared at different adsorbent doses, pH solution of 7.0, contact time of 60 minutes, and inlet concentration level of 50 mg/L, where according to **Figure 1**, As shown in **Figure 1** by increasing the lead primary concentration the removal efficiency increases as it reaches 87.3% at the concentration of 10 mg/L,

Where According to **Figure 1**, it noticed that by increasing the lead primary concentration the removal efficiency increases as it reaches 87.3% at the concentration of 10 mg/L.

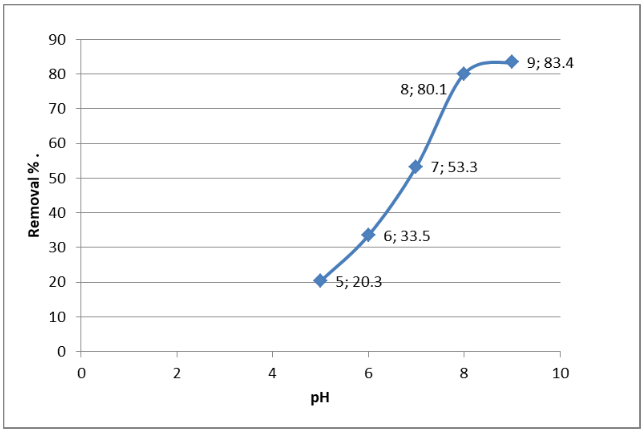

Figure 1: Pb removal Efficiency versus doses of adsorbent, with 60 minutes contact time , pH=7 and feed concentration of 150 mg/L.

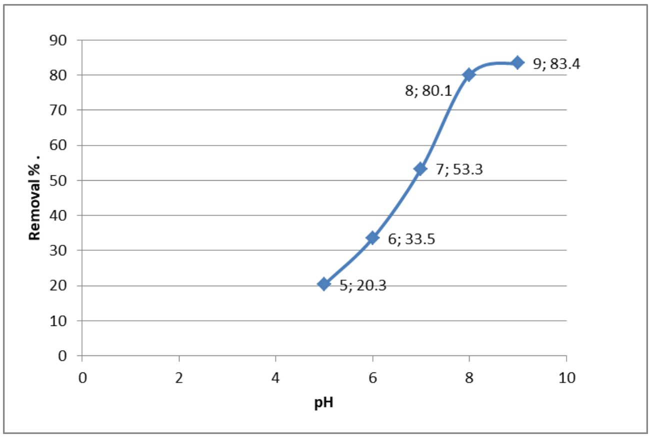

Figure 2: Lead removal Efficiency in different pH, with 60 minutes contact time, feed concentration 150 mg/L, and dosage of adsorbent 5 g/L

Given the results of this analysis, it can be seen that the zeolite is made of 50.74% SiO_2 for each 100 g of the zeolite with a specific gravity of 1 g/cm3 , However to investigate the effect on the process kinetics a three different sizes of the adsorbent molecule volume were used was as follow: 2mm <dp <4mm, 1mm <dp <2mm, 0.5mm <dp <1mm.

Where it was mixed 0.5 gr of one of the above adsorbent molecule volumes with a dose of 50 ml solution has an initial concentration of 100 ppm, after 60 minutes of contact time the samples analyzed to determine the adsorption rate as shown in Fig. 3.

Determination of the effect of contact time on the adsorption of lead was done by increasing the contact time, where according to Fig. 4, lead removal efficiency increases with increasing the contact time. At the contact time of 45 minutes, the removal efficiency almost reaches a balance. Therefore, this time is considered as the optimum time. The removal efficiency and adsorption capacity at this time are 80.3% and 5.19 mg/g, respectively.

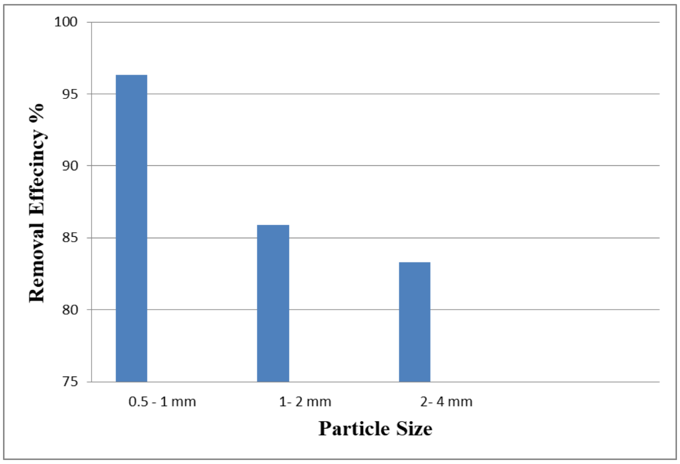

Figure 3: The effect of particles size on the adsorption of lead ions from the solution (T = 60 min, V = 50ml, W = 0.5gr, pH = 6, $C_{initial}$ = 100ppm)

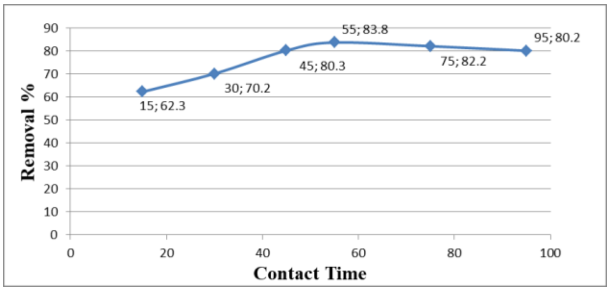

Figure 4: Figure 4. The effect of contact time on the adsorption of lead with 150 mg/L input concentration of, solution pH equal to 8 and of 5 g/L adsorbent dosage.

Investigation of lead adsorption using Langmuir and Freundlich adsorption isotherm models is accomplished according to the data derived from figures 5 and 6 which demonstrate Langmuir and Freundlich isotherm curves for lead adsorption using natural zeolite [11, 22], the Langmuir isotherm (**R^2= 0.8658**) has more conformity in comparison with Freundlich isotherm. Table. 3 presents the parameters of isotherm and capacity of lead adsorption using natural zeolite clinoptilolite.

Table 3 . Lead adsorption isotherm parameters

Isotherm Type		**Langmuir**			**Freundlich**	
Parameter	qmax (mg/g)	K_L (l/mg)	R^2	N	Kf (mg/)	R^2
Value	8.51	0.45	0.8658	2.46	1.37	0.7852

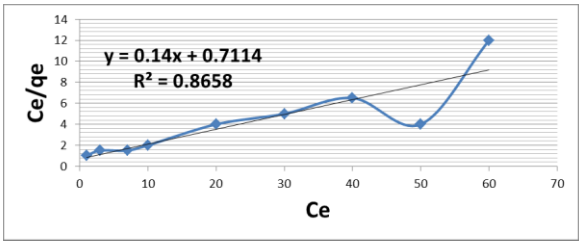

Figure 5: Curve of Adsorption Langmuir isotherm using Clinoptilolite, with adsorbent dosage of 5 g/L, pH=8 and contact time of 45 min.

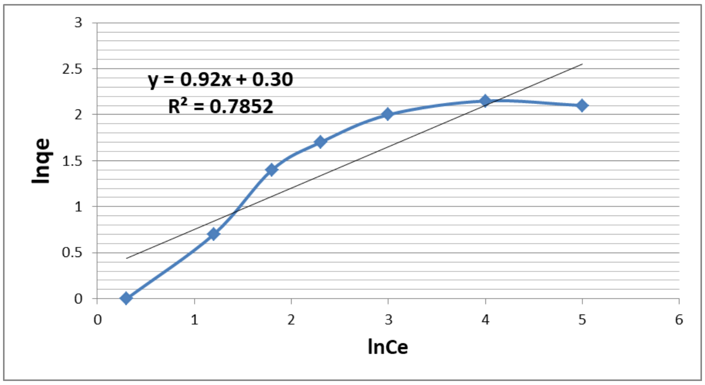

Figure 6: Curve of Adsorption Langmuir isotherm using Clinoptilolite, with adsorbent dosage of 5 g/L, pH=8 and contact time of 45 min.

However , **Figure**. 1 shows the effect of adsorbent dose parameter on the lead removal efficiency. Given the changes of the graph, one can see that lead adsorption efficiency increases rapidly with an adsorbent dose of 5 g/L, but it finds a slower increase after reaching the dose of 7 g/L. Therefore, the optimum adsorption rate is obtained at adsorption dose of 5 g/L with a removal efficiency of 83.3% , where By increasing the adsorbent concentration rate, the adsorption contact surface and consequently the adsorption rate increase [30 , 31].

Figure. 2. clearly shows that at pH 5.0 to 8.0, the rate of removal efficiency increases rapidly and after pH 8.0 this increase continues with a slower pace. In this pH, the highest removal rate is 80.1% and adsorption capacity rate of 5.22 mg/g is obtained with 5 gr of the adsorbent. This is due to activity of the adsorbent functional groups, where the activity of the adsorbent functional groups is intensely influenced by the pH solution. At lower pHs, the increase of hydrogen ions leads to a more competition between H^+ and dissolved cations, and zeolite adsorbs H^+ rather than metal ions, thus the adsorption rate decreases. At higher pHs, with the decrease of H^+ the rate of metal ions adsorption increases and leads to an increase in adsorption rate [23, 9].

It's noticed that according to **Figure**. 3. the removal efficiency decreased with increasing the particles size this effect is due to the contact surface area of absorbent where the contacting surface area with 1-2 mm samples be more than 1-2 mm and the last has an effective contacting area more than 2-4 mm.

Figure. 4. shows that by increasing the contact time, lead removal efficiency increases. According to the obtained results, the removal efficiency first increases with increasing the contact time and then in a time of about 55 minutes it comes to a state of balance until the concentration of 83.8 mg/L.

Which represent the highest removal efficiency achieved at a time of 55 minutes.

Later the decrease of lead removal using clinoptilolite which appears alongside with the increase of contact time and, particularly, at the end of the testing, is for the presence of a layer of lead ions on the outer surface, the clinoptilolite pores, and pores distributed on the inner surface of the clinoptilolite particles, also for the long contact time while sustaining the mixing conditions till the end of the testing .

In this study, Langmuir and Freundlich isotherms were investigated in order to investigate the process of metal removal using zeolite. Also to determine the isotherm models, the concentration of 10, 20, 30, 40, 50, 60, 70, 80, and 100 mg/L at the contact time of 90 minutes was decided.

The findings resulted from isotherm studies, revealed that the process of lead removal follows the Langmuir isotherm equation of R^2= 0.8658. Freundlich isotherm conformity demonstrates the presence of a layer of adsorbent molecules on the surface of adsorbent particles, while the lead adsorption conformity with Langmuir model is related to the monolayer adsorption and suggests that the surface of adsorbent has some sites with an equal energy, in which each molecule is dedicated only to one site. Therefore, perhaps it shows the heterogeneous distribution of active sites of the adsorbent surface.

According to the results obtained from Langmuir model regarding the lead metal, maximum adsorption capacity which demonstrates the maximum monolayer adsorption coverage, is 5.3 mg/g. The results of isotherm studies suggest that the constant value of n for Freundlich model is >1 which shows the proper condition for lead adsorption on the surface of zeolite [17,29].

5. Discussion

5.1. Controller Performance Evaluation

A digital sliding mode controller keeps the system stable under changing wind speed conditions according to simulation results. The system reveals several operational issues during performance analysis.

1. The controller shows an incapability to cancel steady-state error because output voltage sets at an improper value beneath reference levels. The strength of integral action in the sliding surface needs further evaluation because of insufficient performance.
2. Changes in output voltage create periods of deep declines which deteriorate the regulatory capabilities. The output voltage experiences these voltage fluctuations at the same times that the controller changes states which could result from implementation methods or chosen parameters.
3. The output voltage shows inadequate tracking of reference values due to problems related to current controller parameters or their respective settings. Even though the controller exhibits certain drawbacks it maintains several important assets in its operation:

- Stability maintenance despite disturbances;
- The system shows quick reaction upon modifications in its input;
- Bounded control action.

5.2 Comparison with Conventional Control Techniques

The article demonstrated that sliding mode control provides better resistance against parameter variations and disturbances compared to standard PI or PID controllers even though a direct comparison was not conducted in this simulation. Some advantages of sliding mode control are reduced by implementation difficulties which appear during digital realization. [17].

6. Conclusion

The core objective of this research is to investigate digital sliding mode control approaches for DC/DC Buck converters in wind turbine applications. A detailed mathematical model of the system was constructed, and the proposed controller was implemented and validated using digital simulation tools. The simulated results verify that the system maintains stability during changing wind speeds but controller performance must be improved for achieving better reference tracking and voltage regulation. The phase portrait evaluation demonstrates both system stability and time-dependent behaviour of the system.

This research defines the benefits and drawbacks of this control technique when operating on wind energy systems with their non-linear characteristics.

Declaration of Competing Interest

The authors declare no conflicts of interest.

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This study didn't receive any specific funds.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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