Desalination and Water Treatment
www.deswater.com
doi:10.5004/dwt.2017.21671

98 (2017) 244–253
December

Comparative study on adsorptive removal of Cu, Pb, Zn heavy metals by modified perlite composites

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Received 16 March 2017; Accepted 14 November 2017

ABSTRACT

This study examined the removal of heavy metals Cu^{2+}, Pb^{2+}, Zn^{2+} from water solution by using perlite, perlite activated with oxalic acid and perlite modified with chitosan. The adsorption isotherms were also calculated. The conditions that affect the adsorption process were asserted as pH, adsorption dose and operation time. The highest adsorption yield among the four different adsorbent examined was provided by perlite activated with oxalic acid. The metal holding capacities were determined as 16.72 mg/g for Cu, 18.48 mg/g for Pb and 14.184 mg/g for Zn. This capacity was interpreted as the indicator of usability on the heavy metal removal functions in industrial treatment facilities. The experimental metal sorption of perlite activated forms is described best by the Freundlich adsorption isotherm model with determination values over 0.99. The research results prove that perlite and modified perlite are good natural adsorbents for the removal of heavy metal from water.

Keywords: Chitosan; Perlite; Copper (Cu); Lead (Pb); Zinc (Zn); Heavy metals

1. Introduction

Copper, lead and zinc are extremely toxic heavy metals that can be found widely in fresh and waste water. For that reason these metals should be removed from domestic and industrial water. Copper (Cu) is one of the toxic metals. The main sources of copper ions are, essentially, the industrial waste streams of metal cleaning and plating baths, pulp, paper, paperboard and wood preservative WWW employing mills, the fertilizer industry etc. [1]. Lead (Pb) is one of the most important environmental pollutants.

Firstly, lead is emitted from the exhausts of cars into nature. The permissible level of lead in drinking water, according to the US Environmental Pollution Agency (EPA) is 0.05 mg L^{-1}. Therefore, even low amount of lead in water is extremely toxic [2]. Zinc is a material that can be found naturally in air, water and soil. Most zinc is produced by the mining, coal and waste incineration and steel processing industrial activities.

Soil is contaminated where the zinc is extracted, refined or sewage mud is used as fertilizer. As a result of the contamination of the bodies of fish living in zinc contaminated water, zinc may be included in the food chain. When agricultural land is contaminated with zinc, this may affect the health of plants and animals negatively [3].

Using natural materials for the removal of heavy metal is a wide concept. Cheap and easy availability of the materials is the most important element of the process. Perlite is a volcanic glass that can be found widely in Turkey. It can expand and become porous when heated to the correct temperature, and can be found from grey to black naturally. Because of the different characteristics of different perlite types, they have different compounds [4]. Perlite expands its volume almost 20 times when it is heated between 800

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and 1200°C, with individual forms of grain thickness; it expands like corn and becomes very heavy. Expanded perlite is white and its density is 32 kg/m^3 [5,6]. It is possible to increase the yield of concentration removal with adsorption on the surface of perlite with different activations.

Chitosan, which is used in the study, has the chemical structure poli-[b-(1,4)-2-amino-2-deoksi-b-D-glukopiranoz]. Chitin and chitosan polysaccharides look like cellulose chemically, but they differ among themselves. While the hydroxyl (–OH) group bound to the second carbon atom exists in cellulose, acetamide(-$NHCOCH_3$) in the chitin and amine (-NH_2) group exists in chitosan. Molecule heaviness is one of the other important parameters for the natural and synthetic application of chitosan. The molecule heaviness of the chitosan varies according to the resource gathered and especially the de-acetylation conditions (temperature, time and NaOH concentration) [7,8].

In recent years, chitosan has also increasingly been studied as an adsorbent for the removal of heavy metal ions from aqueous solutions because the amino and hydroxyl groups on the chitosan chain act as a chelation or reaction site for the substances to be removed [9]. Irani et al. (2011) used perlite, dolomite and diatomite for lead removal from water solutions. The effects of the variables such as natural perlite, dolomite and diatomite with lead adsorption, pH, waiting time, initial concentration and temperature were examined. The maximum lead adsorption capacity was determined as diatomite (25.01 mg g^{-1}), dolomite (19.69 mg g^{-1}) and perlite (8.906 mg g^{-1}) [10]. Khobragade et al. (2015) researched Cu (II) and Ni (II) removal with single-metal and binary-metal solutions of surfactant-modified alumina (SMA). It was determined that the removal of Cu (II) and Ni (II) was depend on pH, detention period and adsorbent dose, and the adsorption kinetic is suitable with the Freundlich isotherm kinetic [11]. Cu (II) removal from water solutions was examined by using chemically modified chitosan by Kannamba et al. who considered the effect of parameters such as pH, temperature, adsorption dose, initial Cu (II) concentration and detention period on the adsorption capacity. The maximum adsorption capacity from the Langmuir isotherm was found to be 43.47 mg g^{-1} [12].

Several further conventional methods have been used for the removal of heavy metals in the purification methods of environmental engineering for a long time. The chemical precipitation adsorption and ion-exchange method are some of these methods. In recent years, in particular, adsorption studies have concentrate on the removal of heavy metals such as Cu(II), Pb(II) and Zn (II) from fresh and waste water, and various natural adsorbent modification studies were performed [13–20]. However, new adsorbents which are locally available, have a high adsorption capacity and are economic materials are still needed. Therefore, perlite which is available and cheap in our country was used as an adsorbent. The study examined the yield elevation effect on the removal of Pb, Zn and Cu with a new approach in which perlite is activated with oxalic acid and chitosan.

The adsorption capacity of Cu, Pb and Zn by various adsorbents is summarized in Table 1.

2. Material and methods

2.1. Chemicals reagents

Synthetic solutions of Cu(II), Pb(II) and Zn(II) were diluted daily from a 1000 mg L^{-1} stock solutions prepared by solving a required quantity of $Cu(NO_3){\cdot}6H_2O$, $Pb(NO_3)_2$, $Zn(NO_3)_2$ purchased from Merck (Germany). All reagents were analytical grade and used as received without further purification. 99% pure oxalic acid dihydrate and NaOH beads (95–100%) and HCl (35% content, extra pure grade) were purchased from Fisher Scientific Company. Chitosan (molecular weight 1–3 lakh), analytical grade anhydrous, were procured from Aldrich Chemical Corporation.

2.2. Adsorbent characterization

The perlite used in the study was extracted from different regions of our country. The perlite used in this study was provided from the Manisa/Turkey region.

The physical and chemical characteristic of perlite is given in Table 2. Perlite was washed with purified water

Table 1
The adsorption capacity of various adsorbents for Cu, Pb and Zn

Adsorbent	Adsorbate	pH	Adsorption capacity (mg/g)	Max. initial concentration (mg/L)	Ref.
Chitosan (shrimp)	Cu(II)	5.5	152.7	325	[13]
Chitosan	Cu(II)	5	160	288	[14]
Chitosan coated perlite	Cu(II)	5	196	>1000	[15]
Chitosan coated perlite bead	Cu(II)	4.5	104	1000	[5]
Chitosan coated expanded perlite	Cu(II)	7–9	16.72	100	This study
Expanded perlite	Pb(II)	6.5	6.27	5	[1]
Expanded perlite	Pb(II)	5	13.39	400	[10]
Perlite	Pb(II)	6	8.906	100	[16]
Chitosan/magnetite composite bead	Pb(II)	6	63.33	80	[17]
Chitosan coated expanded perlit	Pb(II)	7–9	18.48	100	This study
Chitosan coated expanded perlite	Zn(II)	7–9	14.184	100	This study

Table 2
Chemical composition of perlite

Constituent	Percentage (wt. %)
SiO_2	71
Al_2O_3	12.5–18
Na_2O	2.9–4
K_2O	4–5
CaO	0.5–2
Fe_2O_3	0.1–1.5
MgO	0.02–0.5
TiO_2	0.03–0.2
H_2O	0–0.2
MnO_2	0–0.1
SO_3	0–0.2
FeO	0–0.1
Cr	0–0.1

several times in order to remove dust and other water soluble matters. It was dried at 105°C for 24 h and sieved with 30 mesh.

2.3. Synthesis of chitosan coated perlite and perlite modified with oxalic acid

Expanded perlite (EP) and its activated forms (oxalic acid EP and chitosan coated EP) were used as an adsorbent. Perlite (sieved with 30 mesh) was first soaked with 0.2 M oxalic acid for 4 h then acid treated perlite was washed with distilled water and dried in an oven for 12 h. When this process was complete thirty grams of chitosan flakes were dissolved in 1 L of 0.2 mg L^{-1} oxalic acid solution to prepare a gel. Then, 60 g of perlite prepared with oxalic acid was added to the gel. It was stirred for 4 h while heating at 323 K (50°C) to obtain a homogeneous mixture. The chitosan-coated perlite was set by the drop-wise addition of the mixture into a 0.7 M NaOH precipitation bath. The granules were washed with deionized water to a neutral pH and the grain size increased 235 mesh by drying. The adsorbents were characterized by, Fourier transform infrared spectroscopy and surface area analysis. Also, the effectiveness of perlite modification were estimated using elemental (C,H, N, S) and thermogravimetric analysis.

2.4. Characterization methods

2.4.1. Fourier transforms infrared (FTIR) studies

Pure perlite, perlite washed with oxalic acid and chitosan-coated perlite (CCP) FTIR spectrums were taken between 400 and 4000 cm^{-1}. FTIR measurements were performed with a PERKİN ELMER UATR-TWO diamond ATR device.

2.4.2. Brunauer, Emmett and Teller (BET) studies

The surface area of the pure perlite, perlite with oxalic acid and chitosan-coated perlite was measured by using a Micrometrics (USA) BET (Brunauer, Emmett and Teller) instrument using the nitrogen intrusion technique. The microstructure of the sorbent was characterized using physical adsorption/desorption of nitrogen at −196°C. The samples were analyzed with an elemental analyzer, and on the basis of difference in the nitrogen content. Nitrogen isotherms were measured with an ASAP 2000 micro pore analyzer at 77 K.

2.4.3. Scanning electron microscope (SEM) studies

Scanning electron micrographs were taken for the CCP beads, using a software controlled digital scanning electron microscope JEOLJSM 5410, Japan (Eucentric Gonimeter State type). In this microscope, very fine pencils of electrons are used for illumination, which scans the surface of the beads, much as a screen is scanned in a television tube. The wavelength of the electron beam is very short (0.05–0.005 Å), and is produced by a filament heated by high voltage.

2.4.4. Elemental and thermogravimetric analysis

Elemental analysis measurements, which were applied in order to determine the amounts of nitrogen, carbon, hydrogen and oxygen that modified perlite forms (oxalic acid perlite and chitosan coated perlite) consist, were got performed with a LECO CHNS 628 model device by burning at high temperature (1000–1100°C).

To examine the weight variations depends on the temperature of modified perlite forms, were heated from room temperature to 600°C in nitrogen gas atmosphere with a heating rate 10°C/min by NETZSCH-STA 449 F1 instrument. Furthermore, differential thermogravimetric analysis, which is closely related to thermogravimetric analysis, were examined for modified perlite forms. Thus, the first derivative according to time of weight variation was investigated for both forms.

2.5. Batch adsorption studies

The dose amount, pH and mixing time were controlled in the discontinuous experimental studies for adsorption preparatory work. They were performed in water samples containing at different concentrations in 1000 mg L^{-1} stock solutions of Cu (II), Zn (II) and Pb (II) in 250 mL containers. The concentration varied between 20 mg L^{-1} and 200 mg/L mg L^{-1}. Contrary to the other studies this concentration range was preferred since heavy metals in domestic and industrial wastewaters were in this range [10]. For pH adjustment a sodium phosphate buffer (0.1 mol L^{-1}) was prepared by adding an appropriate amount of phosphoric acid to a sodium dihydrogen phosphate solution to result in a solution of pH 2. Ammonium chloride buffer solutions (0.1 mol L^{-1}) were prepared by adding an appropriate amount of ammonia to ammonium chloride solutions to produce in solutions of pH 8–9. The experimental study used different amounts (between 0.1–10 g L^{-1}) and mixing time between 5–120 min. The pH in the experiment ranged between 3 and 9. The required amount of adsorbent material was then added and the contents in the flask were shaken for the desired contact time in an electrically thermostatic reciprocating shaker at 200 rpm. The contents of the flask were filtered through filter paper and the filtrate was analyzed for metal concentration by using ICP-OES (inductively coupled plasma-optical emission spectrometer, Spectra Analytical Instruments, Kleve, Germany).

The percentage of adsorption of metal ion was calculated as follows:

$$Adsorption\ (\%) = \frac{C_o - C_e}{C_0} \times 100 \quad (1)$$

Initial and final metal ion concentrations are exemplified with the C_o and C_e in adsorption formula. Adsorption experiments for the effect of pH were conducted by using a solution having 100 mg L^{-1} of Pb (II), 100 mg L^{-1} of Zn (II) and 100 mg L^{-1} of Cu (II) concentrations with an adsorbent dosage of 2 g L^{-1}. Perlite, chitosan and modified types of these used as adsorbent are depicted in graphics were as a, b, c, d status. a: perlite with oxalic acid, b: chitosan coated perlite with oxalic acid, c: pure perlite, d: pure chitosan. While calculating adsorption isotherms, the calculations were performed over a form which had the best yield.

3. Results and discussion

3.1. FTIR results

The perlite washed with pure water and perlite washed with oxalic acid. FTIR spectrums are almost same in each graph. Washing with oxalic acid has not caused any alteration. There is a weak peak on the graphic between 1000 and 700 cm^{-1}. This peak shows the Si-H peak. According to the graphic there is a sharp peak 1056 cm^{-1}. This peak demonstrates –CO stretching vibration in –COH. When Fig. 1c was considered major changes were observed.

These changes were caused by the connection of chitosan to perlite. Peak around the 2900 cm^{-1} FTIR spectrum demonstrates CH_2 stretching vibrations. The FTIR spectrum of CCP in Fig. 1c indicates the presence of predominant peaks at 3360 cm^{-1} (–OH and –NH stretching vibrations), 1641 cm^{-1} (–NH bending vibration in $–NH_2$) and 1395 cm^{-1} (–NH deformation vibration in $–NH_2$).

3.2. BET results

Surface area, density, pore volume, pore diameter and porosity of the composite perlite were determined with a BET (Brunauer, Emmett and Teller) instrument (Model No: Micrometrics, Turkey) to using nitrogen intrusion technique. The isotherm plots calculated the specific surface area (N_2/BET method) and average pore diameter of pure perlite, while micro pore volume was calculated from the volume of nitrogen adsorbed at p/p_0 0.5702 m² g^{-1}. The sorbent material shows that an average surface area is 0.5982 m² g^{-1}. When perlite was washed with oxalic acid p/p_0 0.7386 m² g^{-1} and the average surface area was calculated as 0.8593 m² g^{-1}. When perlite with oxalic acid coated with

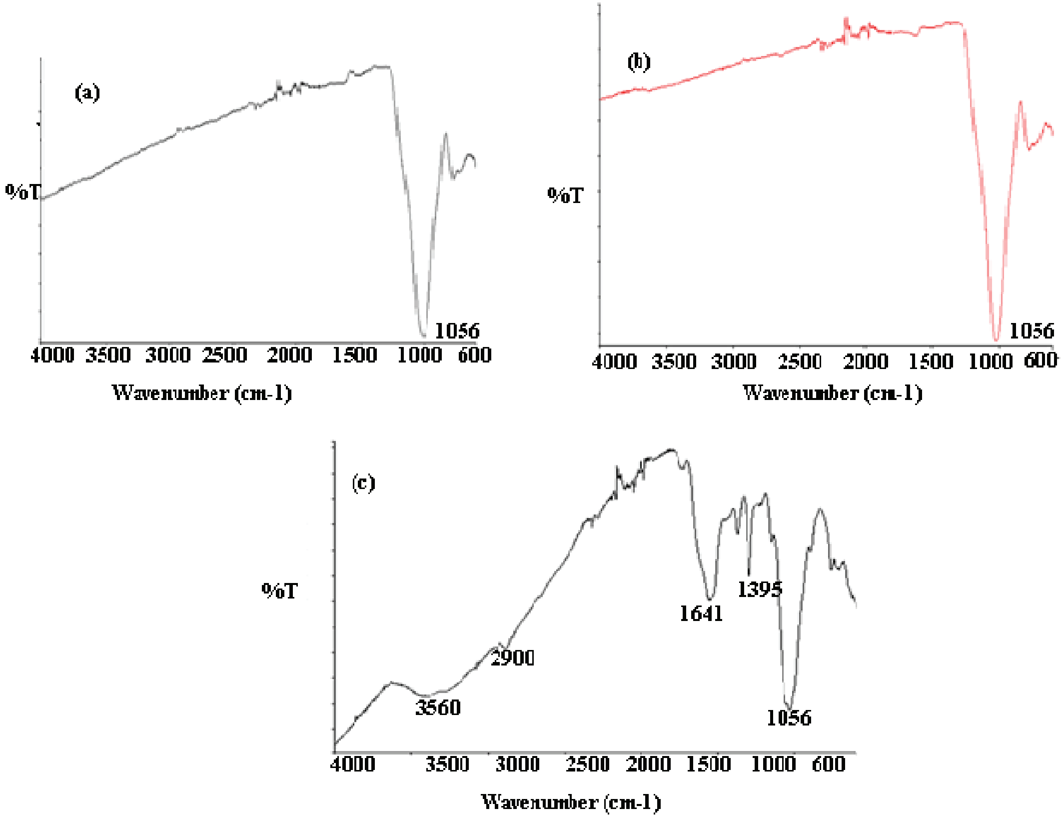

Fig. 1. a) FTIR Spectra of perlite, b) perlite with oxalic acid and c) chitosan coated perlite.

chitosan, the average surface area was calculated as 1.6002 m² g^{-1} as p/p_0 1.0741 m² g^{-1}.

3.3. SEM results

SEM pictures of pure chitosan, pure perlite, perlite modified by oxalic acid and chitosan coated perlite are shown in Fig. 2a–d.

The SEM images of a) pure chitosan and pure perlite shows b) particle sizes between 100 and 150 µm. Oxalic acid modified perlite can be seen with c shape. Composite particles in the chitosan coated perlite sample were distributed spherically in Fig. d when it is coated with oxalic acid, the spaces are smooth and small spaces are formed in the surface.

3.4. Elemental and thermogravimetric analysis results

Elemental composition of oxalic acid perlite and chitosan coated perlite samples are given in Table 3. When elemental analysis results of chitosan coated perlite are compared to elemental analysis results of oxalic acid perlite, it is seen that the amount of carbon of which chitosan coated perlite consists is higher than the amount of carbon that oxalic acid perlite has in its structure. As can be seen from Table 3.

The thermogravimetric analysis method (TGA), which is very common in terms of simple handling and precise results, was used in examining the thermal behaviors of composite materials. As shown in Fig. 3, thermal stability was low due to the organic structure of chitosan, and thermal stability of modified perlite was increased. For oxalic acid perlite the initial degradation temperature was 280°C and the weight loss was 3.5%. In the case of chitosan coated perlite, initial degradation temperature was 200°C and weight loss was 65%.

3.5. The effect of the adsorbent dose

One of the most important parameters in terms of examination of heavy metal removal with adsorption method is the amount of adsorbent used. Being an economic and usable amount is very important for the applicability of the discontinuous study to the real systems.

The result of the effect of adsorption yield of adsorbent doses for all fractions used is given in Fig. 4. Accord-

Table 3
Elemental composition of oxalic acid perlite and chitosan coated perlite

	Carbon (C) %	Hydrogen (H) %	Nitrogen (N) %	Sulphur (S) %
Oxalic acid perlite	3.147	0.800	0.489	0.262
Chitosan coated perlite	13.970	2.670	2.255	0.211

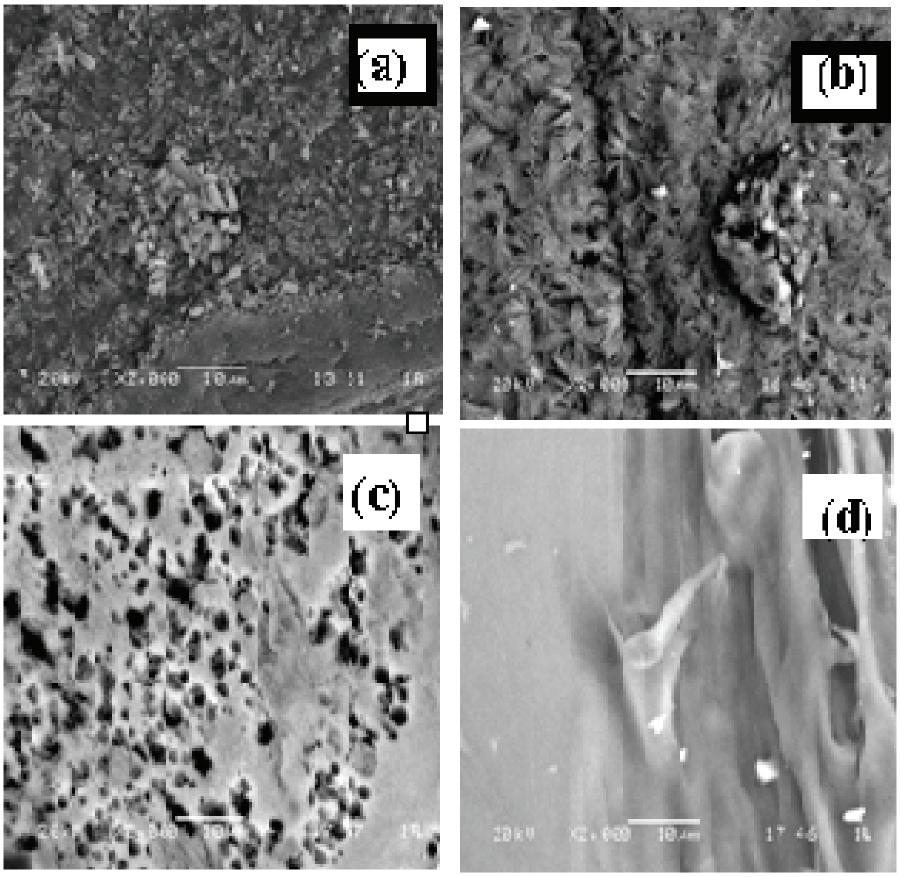

Fig. 2. Scanning electron micrographs a) pure chitosan, b) pure perlite, c) oxalic acid perlite and d) chitosan coated perlite.

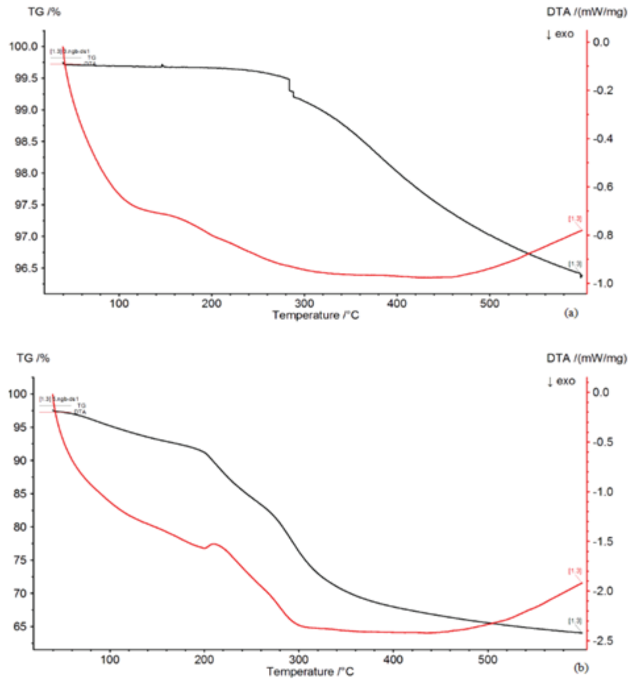

Fig. 3. Thermogravimetric and differential thermogravimetric analysis a) oxalic acid perlite b) chitosan coated perlite.

ing to this result, while the adsorbent amount is effective at small doses, it produced maximum with each heavy metal at 2 g L^{-1}, increasing the dose amount was observed and it remained stable and decreased after this dose. More increase in adsorbent dosage did not have significant effect in adsorption percentage. Therefore, these values have been selected for further adsorption experiments.

At the end of the study it was decided to use 2 g L^{-1} adsorbent dose at adsorption parameters, calculation of isotherms and the other steps of study. The highest yields that were seen chitosan coated perlite were reached at 98% for Cu, 95% for Pb and 84% for Zn.

3.6. The effect of pH

Another important parameter in discontinuous adsorption experiments is pH. pH is one of the important parameters to consider in adsorption processes since H^+ and OH^- ions present in aqueous solutions affect the ability to adhere to solid particles [21]. When the pH value of the aqueous phase is reduced, the solid surface will generally become more positive or less negative [22]. Similar multi experimental studies were applied to different perlite and chitosan activation ranging from 2 g L^{-1} to 100 mg L^{-1}, Cu(II), Zn(II) and Pb(II) concentration at different pH ranges (3–9). As seen in Fig. 5 the effect of the pH is important on all adsorbent variations and the adsorption yield is increased when pH is increased from 3 to

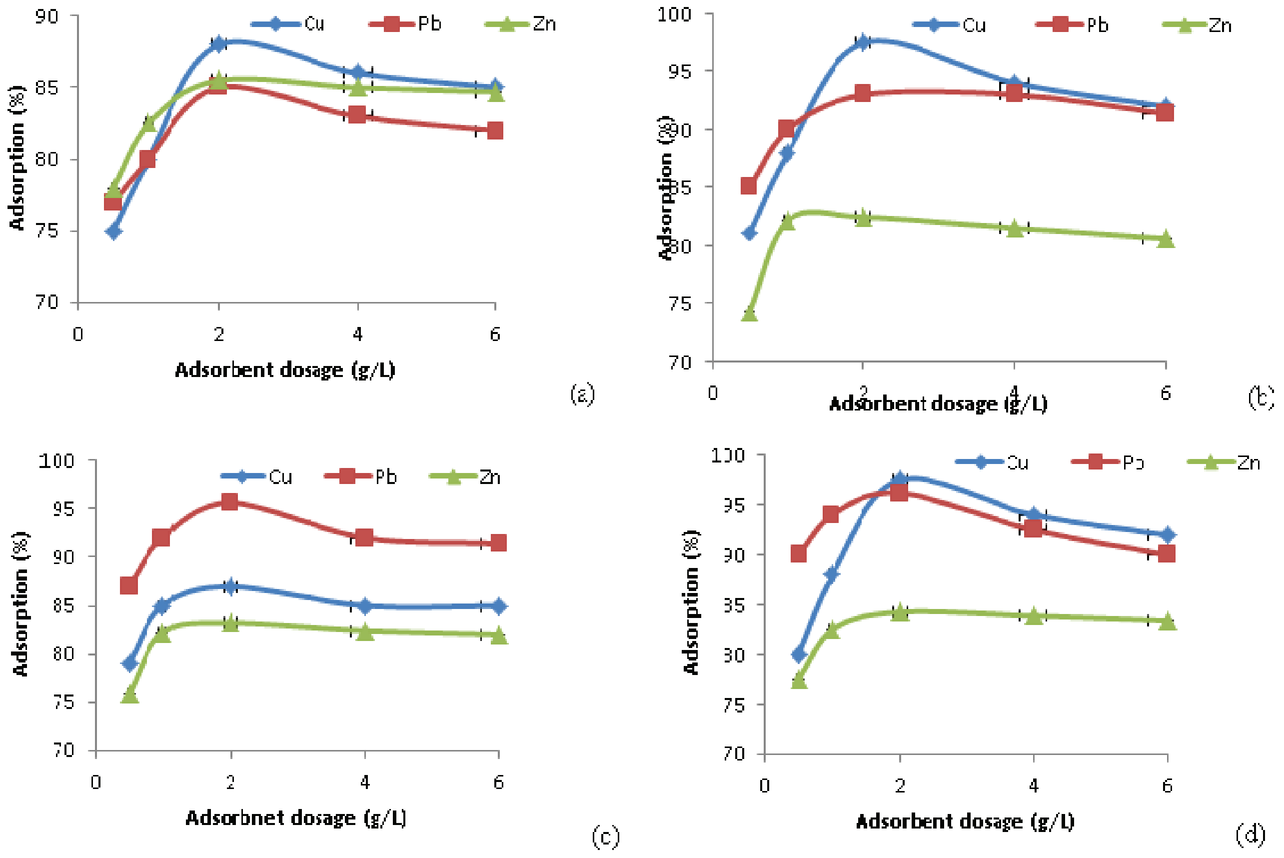

Fig. 4. The effect of adsorbent dosage on the adsorption of Cu (II), Pb(II) and Zn (II) onto a) pure chitosan b) pure perlite c) oxalic acid perlite d) chitosan coated perlite (metal concentration 100 mg/L, pH 7, temperature, 25°C.

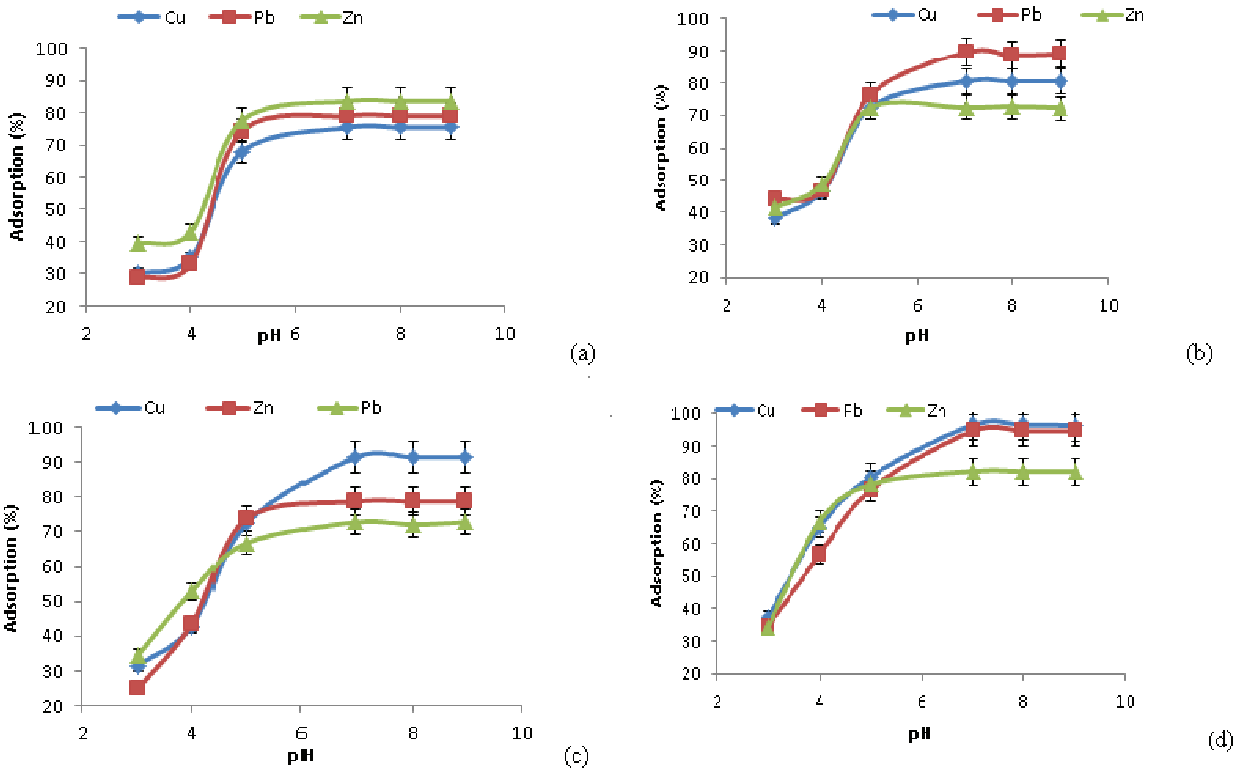

Fig. 5. The yield graphic in connection with the pH according to three different activated adsorbent for copper, zinc, lead synthetic samples a) pure perlite b) oxalic acid perlite and c) chitosan coated perlite (metal concentration 100 mg/L, time 60 min; temperature 25°C).

9. pH 7 was accepted as the most suitable value and continued to other studies. Similar results can be seen in literature [4].

3.7. Effect of the mixing time

The mixing time is one of the important parameters for the metal ions to be able to hold onto the solid particles and to be able to carry out this process with full capacity. The experiment results were obtained after the mixture was run at 200 rpm for 15–120 min and 293 K temperature for three different activation types for the most positive dose of 2 g L^{-1}. This was determined according to the perlite and chitosan concentration of 100 mg L^{-1} at pH: 7 in order to examine the effect of the mixing time on adsorption of Copper (II) ion. For all adsorbents, the metal removal in the solvents was removed within the first 30 min and the system reached a certain degree of saturation after 60 min to reach the equilibrium. When the contact time is increased to 120 min, there is no appreciable increase in the metals retained in the adsorbent. As seen in this Fig. 6, adsorption of these metals increased by contact time and reached to a maximum value. More increase in contact time had no more sensible changes in adsorption percentage.

3.8. Adsorption isotherms

3.8.1. Langmuir isotherm

The Langmuir isotherm assumes that adsorption occurs by single layer in a plate which contains limited number of adsorption regions that has identical adsorption energies and adsorbate which does not migrate on surface plate. To be clearer, it says by accepting the fact that every absorbent point will absorb only one molecule, the forming layer will be one molecule in thickness. In Langmuir isotherm, adsorption increases linearly with absorbent concentration increase. It has reached the maximum adsorption capacity in equilibrium and the surface is coated with a single layer [23]. All the results of the studies in connection with the Langmuir equation according to the experiment results performed with copper, zinc, lead solution and the graphic of the Langmuir equation is given in Fig. 7.

$$(C_e/Q_e = 1/K_L \cdot X_m + C_e/X_m) \tag{2}$$

Here, q_e (mg g^{-1}) the amount of adsorbate adsorbed per unit mass of active carbon, C_e (mg L^{-1}) equilibrium concentration of adsorbate in solution, q_{max} (mg g^{-1}) and K_L (mg L^{-1}) are Langmuir constants depend on Maximum mono layer adsorption capacity and equilibrium constant or bond energy, respectively.

3.8.2. Freundlich isotherm

All the results of the studies in connection with the Freundlich equation according to the experiment results performed with copper solution and the graphic depicting about Freundlich equation is given in Fig. 8.

$$(\log Q_e = \log K_F + 1/n \log C_e) \tag{3}$$

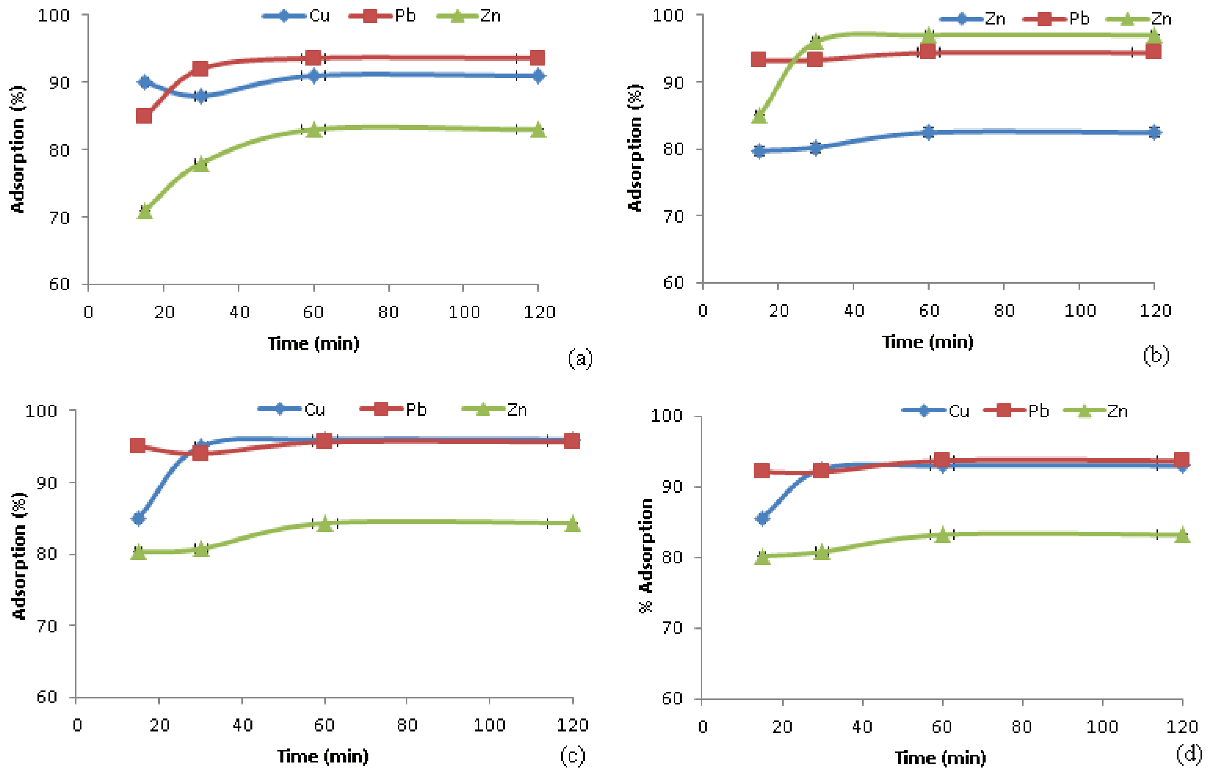

Fig. 6. The yield graphic in connection with the mixing time according to three different activated adsorbent for copper, zinc, lead synthetic samples a) pure perlite b) oxalic acid perlite and c) chitosan coated perlite (metal concentration 100 mg/L, pH 7, temperature 25°C).

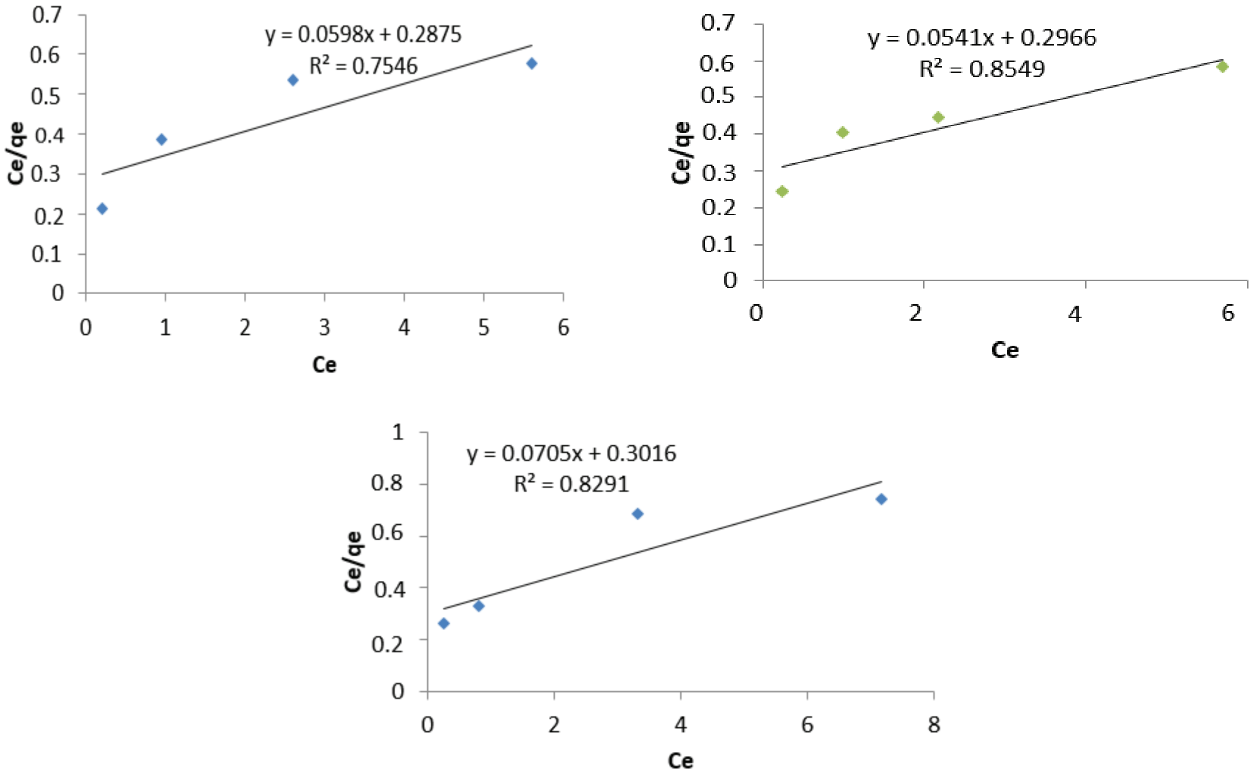

Fig. 7. Langmuir isotherm graphic for a) copper, b) lead, and c) zinc chitosan perlite experiments.

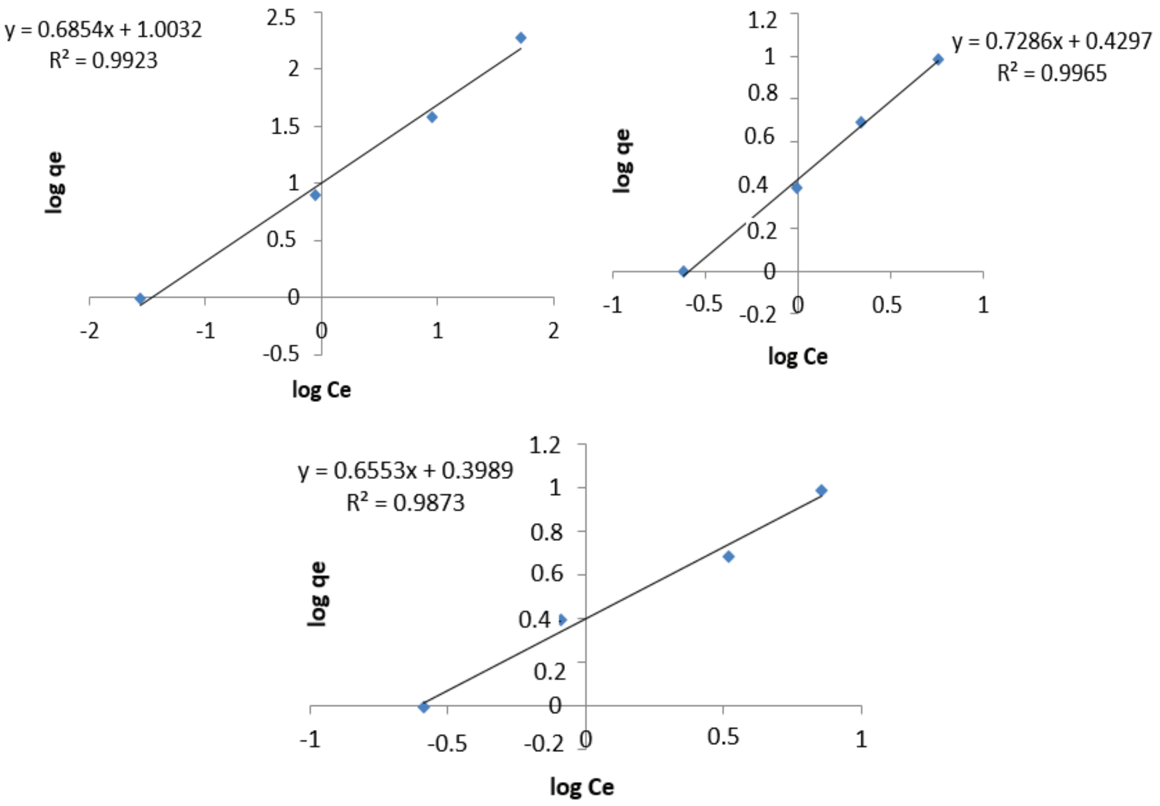

Fig. 8. Freundlich isotherm graphic for a) copper, b) lead, and c) zinc chitosan perlite experiments.

Table 4
Isotherm models for the adsorption of Cu, Pb and Zn onto EP

Heavy metals	Langmuir isotherm model			Freundlich isotherm model		
	Q_m (mg g^{-1})	K_L (mg L^{-1})	R^2	n^{-1}	K_F (mg g^{-1})	R^2
Cu	16.72	0.21	0.75	0.99	2.726	0.99
Pb	18.48	0.18	0.85	1.37	1.536	0.99
Zn	14.184	0.23	0.82	2.5	1.490	0.99

K_F (mg g^{-1}) and n are Freundlich constants depend on adsorption capacity and density (or surface heterogeneity) [24]. Langmuir and Freundlich adsorption isotherms were used while forming the adsorption isotherms. It was determined that Cu^{2+}, Pb^{2+}, Zn^{2+} removal with perlite demonstrated suitability to the Freundlich isotherm. The calculated constants for Freundlich isotherm and Langmuir isotherm models are summarized in Table 3. It was determined that none of the heavy metal was suitable to the Langmuir isotherm with 0.75, 0.85 and 0.82 determination coefficient. The Cu^{2+}, Pb^{2+}, Zn^{2+} metals have 0.99 determination coefficient to the Freundlich isotherm.

The value of 0.99, which is the obtained r coefficient found for Freundlich, was the expected result. Because the adsorption mechanism in liquid solutions are the most suitable to the Freundlich isotherm. The literature data also certifies these findings.

When the constants related to Freundlich are examined, it was decided that there is an affinity relation between k and heavy metals perlite. Because it's similar to the order of selectivity of k values and the order of mostly adsorbed metals. Accordingly, interest is Pb > Cu > Zn.

4. Conclusion

This study examined the heavy metal removal yields obtained between the 15 and 120 min time range, with various pH values and stable temperature for perlite, perlite with oxalic acid and chitosan coated perlitedose amounts for the three different activation forms between 0.4–6 g L^{-1} ranges. As a result, the optimum adsorbent dose is determined to be 2 g L^{-1}. Optimum pH value was determined between 7 and 9, similar to the examined literature studies. As the most suitable contact time of 60 min for the mixing time, the best yield is determined to be the oxalic perlite type activation.

Langmuir and Freundlich adsorption isotherms were used while forming the adsorption isotherms. It was determined that Cu^{2+}, Pb^{2+}, Zn^{2+} removal with perlite demonstrated suitability to the Freundlich isotherm. It was determined that none of the heavy metal was suitable to the Langmuir isotherm with 0.75, 0.85 and 0.82 determination coefficient. The Cu^{2+}, Pb^{2+}, Zn^{2+} metals have 0.99 determination coefficient to the Freundlich isotherm. The value of 0.99, which is the obtained r coefficient found for Freundlich, was the expected result. Because the adsorption mechanism in liquid solutions are the most suitable to the Freundlich isotherm. The literature data also certifies these findings. As the result of all the studies, Gördes perlite which is economic in terms of cost and availability in this region was determined as a good adsorbent for Cu, Pb and Zn heavy metals.

Acknowledgement

This work was supported by the Sakarya University Scientific Research Foundation (Project Number: 2010-01-12-010). We here by thank to Fatih Mehmet Kosar for language support and www.academicproofreading.com for proof reading.

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