MN^{2+} AND CU^{2+} ADSORPTION WITH A NATURAL ADSORBENT: EXPANDED PERLITE

TURP, S. M.

Department of Environmental Engineering, Bitlis Eren University, Bitlis, Turkey
(e-mail: smturp@gmail.com)

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Abstract. Water containing heavy metals causes many diseases including, in particular, cancer. For this reason, it must be completely purified before being discharged. In this study, the surface area of expanded perlite (EP) was determined to be 406.259 m^2/g by the method proposed by Brunauer, Emmet and Teller (BET). The removal efficiencies in terms of the heavy metals Mn^{2+} and Cu^{2+} were investigated according to this value. The optimum point of adsorption of manganese and copper with expanded perlite is 30 min and 5 min, respectively. The optimum absorbent dosage is 0.5 g for manganese and 0.4 g for copper. In the case of Mn^{2+} and Cu^{2+} adsorption, Log_q/Log_C values were plotted to determine the Freundlich isotherm on the expanded perlite. In addition, Langmuir isotherm constants were determined by plotting 1/q and 1/C values. It has been found that with 5 mg/L Mn^{2+} and Cu^{2+}, the Mn^{2+} heavy metals are compatible with the Langmuir isotherm model with $R^2 = 0.93$, and the Cu^{2+} heavy metals are compatible with the Langmuir isotherm model with $R^2 = 0.99$.
Keywords: *adsorption isotherms, heavy metal treatment, expanded perlite, Mn and Cu treatment*

Introduction

Industrial wastewaters contain various heavy metals, pesticides, salts and detergents, which cause chemical and physical changes in the water. Industrial waste pollutes not only the receiving environment, but also agricultural land and forests, and the surface and groundwater resources around it. The heavy metals in the wastewater resulting from industrial activities are often found in waste water from minefields, leakage waters of solid waste sources. These wastewaters accumulate in sediments mixed with rivers, lakes and underground waters. Even in a region remote from the discharge point, the level of heavy metal pollution is preserved. At the same time, metal compounds can be transformed into other compounds during transport. Many economical and effective methods for heavy metal removal have been used and new treatment methods have been developed. From these methods; ion exchange, chemical precipitation, reverse osmosis, evaporation, membrane filtration and biosorption are widely used, but the most effective method is seen as adsorption. The advantages and disadvantages of these methods used in heavy metal removal in *Table 1* are explained in detail (Hamutoglu et al., 2012).

It can cause pollution in agricultural land within the area affected by industrial establishments, the accumulation of heavy metal in soil, a decrease in productivity with regard to growing crops, and accordingly, the devaluation of agricultural land (Gonullu, 2004).

Copper and manganese, which are found in trace amounts in nature and in living things, are stored as heavy metals, especially in human and animal livers. Exposure to high doses of copper and manganese for long periods are harmful to health (Mudhoo et al., 2012). Regulations in Turkey with regard to water intended for human consumption in order to ensure the quality of the water, is regulated by technical and hygiene conditions (TS-266). The maximum values for copper Cu (II) and manganese Mn (II) were determined to be 2 mg/L and 0.05 mg/L, respectively (Published Official Gazette,

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2012). In studies carried out for the removal of heavy metals which cause water pollution as a result of industrial activities, Xie et al. (2017) used sulfur micro-particles to remove copper from the aqueous solution. The adsorption process reached an equilibrium in 30 min and the optimum removal occurred at pH 4.5, ensuring compliance with the Freundlich isotherm model (Xie et al., 2017). Dai et al. compared two different carbon materials in their work. In addition, they investigated adsorption isotherms, contact time, pH and ionic strength. The Langmuir isotherm model is suitable for copper and zinc (Yingjie, et al., 2017). Mishra et al. (2017) in experiments involving Cu (II) and Ni (II) in red and black soil, kinetic and isothermal studies of metal ions were carried out to determine contact time, adsorbent dosage and metal concentration. Cu (II) and Ni (II) reached equilibrium after 480 min and 300 min of shaking, respectively. The maximum removal efficiency for Cu (II) by red soil was 97.3%, for Ni (II) by black soil was 99.9%. An examination of kinetic and adsorption isotherms has shown that adsorption experiments involving the pseudo-second-order equation and the Freundlich isotherm model, respectively (Mishra et al., 2017). Zahar et al. (2015) studied various variables in experiments to evaluate the ability of steel slag to remove manganese from an aqueous solution. The variables included contact time, adsorbent dosage, pH and initial manganese concentration. The equilibrium contact time was 10 h. An absorbent dosage of 1 g was found to be sufficient for removing heavy metals from the aqueous solution. A pH of 6 for manganese adsorption on steel slag was found to be the optimum. The Langmuir isotherm model adapted to Mn (II) adsorption by steel slag. In the Mn (II) adsorption of steel slag, the Langmuir isotherm was conformed (Zahar et al., 2015). Ateş and Akgül (2016) used natural zeolite obtained from the Manisa-Demirci region, modified with NaOH aqueous solution (0.5-2.0 mol/dm^3), and worked on manganese adsorption. The maximum manganese adsorption was obtained using NaOH-modified zeolite. Manganese adsorption with natural zeolite was consistent with the Langmuir isotherm model, whereas the use of manganese adsorption on modified zeolite was consistent with the Freundlich isotherm model (Ateş and Akgül, 2016). In 2010, Mesci and Turan investigated the removal of Cu (II) and Zn (II) by adsorption in the leachate of industrial using illite. They investigated the effect of illite, a natural clay species, as an adsorbent on the adsorption process in terms of pH, dosage and contact time. Experimental studies have shown that illite is effective in adsorbing Cu (II) and removing Zn (II), and can be used as an alternative to other adsorbents in the treatment of leachate water due to its low cost. Although active carbon is widely applied in adsorption processes, it is very costly. They found that illite, an inexpensive material that could be an alternative to activated carbon, had sufficient binding capacity for the removal of Cu (II) and Zn (II) from the leachate (Mesci and Turan, 2010). Xu et al. investigated the effect of operating parameters and electrolytes on the removal of toxic metals (cadmium, zinc and manganese) from synthetic mine molten effluent by bulk electrocoagulation. The results have shown that the removal of heavy metals increases the efficiency of the solution by increasing the pH and the current density of the solution. Fe-Fe electrode combination is more effective than other combinations (Al-AI, Al-Fe and Fe-Al). The interaction of heavy metal ions negatively affected the removal of Zn^{2+} by increasing the concentration of Zn^{2+} at the beginning. The single chlorine system confirms the optimized removal over Mn^{2+}. Xu et al. (2017) suggested that adding a small amount of sodium chloride to a high sulphate and hardness solution could accelerate the removal of heavy metals. In 2014, Ardali et al. used expanded perlite as an adsorbent in the removal of Cu (II) in

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industrial waste leachate. In the study, the Cu (II) removal from industrial waste leachate was investigated by the discontinuous adsorption technique. The expanded perlite industrial leachate can be successfully used as an adsorbent for Cu (II) removal. Particle-to-particle diffusion and Elovich kinetic models are used. The removal rate of Cu (II) increases with an increase in the concentration of expanded perlite. The results have shown that expanded perlite leachate is a potential adsorbent for Cu (II) removal (Ardalı et al., 2014).

Table 1. *Advantages and disadvantages of methods used in heavy metal removal (Hamutoglu et al., 2012)*

Methods	Advantages	Disadvantages
Chemical precipitation and filtration	Simple and cheap	Difficult separation at high concentrations, ineffective, waste sludge formation
Electrochemical methods	Recovery of metal	Expensive, only active at high concentrations
Chemical oxidation and reduction	Inactivation	Ambient sensitivity
Ion exchange	Effective treatment and recovery of pure waste metal	Sensitive to particles and expensive resins
Evaporation	Pure waste disposal	Additional energy requirement, expensive, waste sludge formation
Reverse osmosis	Pure waste for recycling	High pressure, membrane size, expensive
Adsorption	Activated carbon use of sorbents	Application for all metals

In this study, the expanded perlite adsorption potential of manganese and copper heavy metals were evaluated. The adsorption experiments were carried out according to various parameters such as contact time, initial heavy metal concentration and adsorbent dosage. Isotherm models were applied to the data obtained from the experiments to compare the adsorption capacity of expanded heavy metal with expanded perlite.

Material and method

Adsorbent

The perlite used as an adsorbent material is an amorphous glassy volcanic rock with a pearl luster. When the perlite is heated to a softening temperature range of 760-1090 °C, it expands to about 20 times its constant volume. This expansion is due to the presence of 2-5% trapped water in the structure of the raw pearlite rock. As a result of this expansion, small amounts of granules form as hot and soft glassy particles. Turkey contains approximately 40% of the world reserves of perlite (Şahinoğlu, 2013). In Turkey, expanded perlite has a great variety of applications, while crude perlite is more limited in terms of fields of application. This property is mainly related to the chemical and physical properties of perlite (*Table 2*).

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***Table 2.** Chemical and physical properties of expanded perlite*

Substance	Rate (%)
SiO_2	71-75
Al_2O_3	12.5-18
Na_2O	2.9-4
K_2O	4-5
CaO	0.5-0.2
Fe_2O_3	0.1-1.5
MgO	0.03-0.5
Humidity	Max 0.5%
pH	6
Softening point	890-1100 °C
Melting point	1280-1380 °C
Color	White, gray

Adsorption study

The adsorption isotherms of the expanded perlite manganese and copper were made according to the Batch method (Atasoy and Yesılnacar, 2017). 1000 mg/L Mn^{2+} and Cu^{2+} stock solutions were prepared from $MnSO_4.7H_2O$ (Merck) and $CuSO_4.5H_2O$ (Merck) heavy metal salts, and aqueous solutions were prepared at 8 different concentrations between 1 and 250 mg/L. Removal of heavy metals was carried out in a laboratory environment. All aqueous solutions were prepared using deionized water (ELGA Purelab Option-Q). The prepared samples were supplemented with 0.1, 0.2, 0.3, 0.4, 0.5 g of expanded perlite in 500 mL beakers, respectively. The samples were mixed at 500 rpm using a magnetic stirrer (2 mag Mix 15 Eco) for different amounts of time (5, 10, 20, 30 min). Examples were measured on the Perkin Elmer Optima 5300 DV Optical Emission brand ICP-OES instrument. The recovery efficiencies were also calculated using the following formula (*Eq. 1*).

$$\text{Efficiency (\%)} = \frac{E_i - E_e}{E_i} \times 10 \qquad \text{(Eq.1)}$$

where:
E_i = Initial concentration (mg/L)
E_e = Exit concentration (mg/L)

Data analysis

Adsorption data were calculated using the Langmuir and Freundlich isotherm models. The analysis of the adsorption data is made according to the Langmuir equation (*Eq. 2*) and the Freundlich isotherm equation (*Eq. 3*).

$$\frac{1}{q_e} = \frac{1}{q_m \times K} + \frac{1}{C_e} \qquad \text{(Eq.2)}$$

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$$\log C_s = \log K_f + \frac{1}{nf} \times \log C_e \qquad \text{(Eq.3)}$$

The equation indicates a line between the values of C_e/C_S, and the angle of the slope gives $1/q_e$, whereas the angle of the slope gives 1/Ce in the slope on the *y* axis. K_f and $1/n_f$, Freundlich constants, C_s (mg/g) give the amount of expanded perlite adsorber, while C_e (mg/mL) gives the amount of Mn^{2+} and Cu^{2+} in solution. Since the equation specifies a straight line between logC_e and logC_s, the slope of the rectangle will give the value $1/n_f$, and the point on the y-axis the slope point, logK_f (Cooney, 1998).

Results and discussion

Effect of pH

To investigate the effect of pH on the adsorption of Mn (II) and Cu (II) with expanded perlite, the initial heavy metal concentration was 5 mg/L and the temperature was 25 ° C. The changes in pH value are shown in *Figure 1*. In *Figure 1*, the adsorption efficiency is increased at pH = 5, where adsorption is less at pH = 2. When the values in the graph are taken into account, the best pH-dependent removal efficiencies for Mn (II) and Cu (II) ions are; For Mn (II), pH = 5 – 81%, pH = 7 – 86%, pH = 9 – 87%, pH = 11 – 85%, and also for Cu (II), pH = 5 – 85%, pH = 7 – 84%, pH = 9 – 80% and 81% at pH = 11. Since the removal efficiency is lower than the higher pH values at low pH values, the excess H^+ ion positively expands the expanded perlite surface of the released H^+ ion, which affects the pushing force between the molecules, thereby causing a decrease in the amount of heavy metal adsorbed. Xie et al. Sulfur microparticles were used as adsorbents in copper removal experiments from aqueous solutions. In the adsorption experiments, the pH effect was investigated, and the optimum removal was carried out at pH 4.5 (Xie et al., 2017).

As shown in *Figure 1,* the pH adsorption study of Mn^{2+} and Cu^{2+} heavy metals was carried out at pH 2-11. The best adsorption for Mn^{2+} heavy metal was achieved at pH 5.5-6. As well as the best adsorption at pH 5 in Cu^{2+} heavy metal.

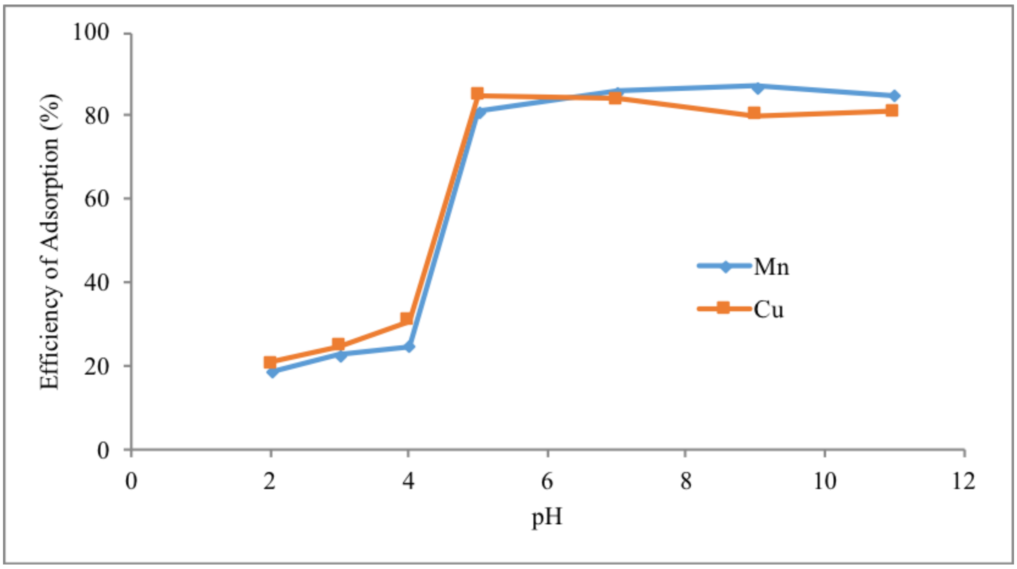

Figure 1. *Adsorption ratio depending on pH of Mn^{2+} and Cu^{2+} heavy metals*

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Effect of contact time

According to the data obtained during the contact period, Mn^{2+} and Cu^{2+} ions showed a tendency to adsorb rapidly in the first 5 min on the expanded perlite. The initial concentration was chosen to be 5 mg/L, which is the best yield in terms of the two heavy metals. The optimum time point in terms of the best adsorption values for Mn^{2+} and Cu^{2+} was assumed to be 30 min and 5 min respectively. The contact times needed to achieve the optimum point were also identified in the isotherm study. As the adsorbent and adsorbed molecules increase the collision time, the efficiency of adsorption increases with the increase of contact time, and after a while, it reaches equilibrium. Compared to the amount of ions adsorbed at the time of initial adsorption by rapid adsorption, it is easy to see that the adsorption level drops. The contact time at which the turning point was reached (30 min for Mn (II), 5 min for Cu(II)) was then used to determine the adsorbent dosing and isotherm studies. Mishra et al. (2017) reported that copper reached equilibrium after 300 min of contact with copper. *Figure 2* shows the percentages of Mn^{2+} and Cu^{2+} adsorbed by the expanded perlite.

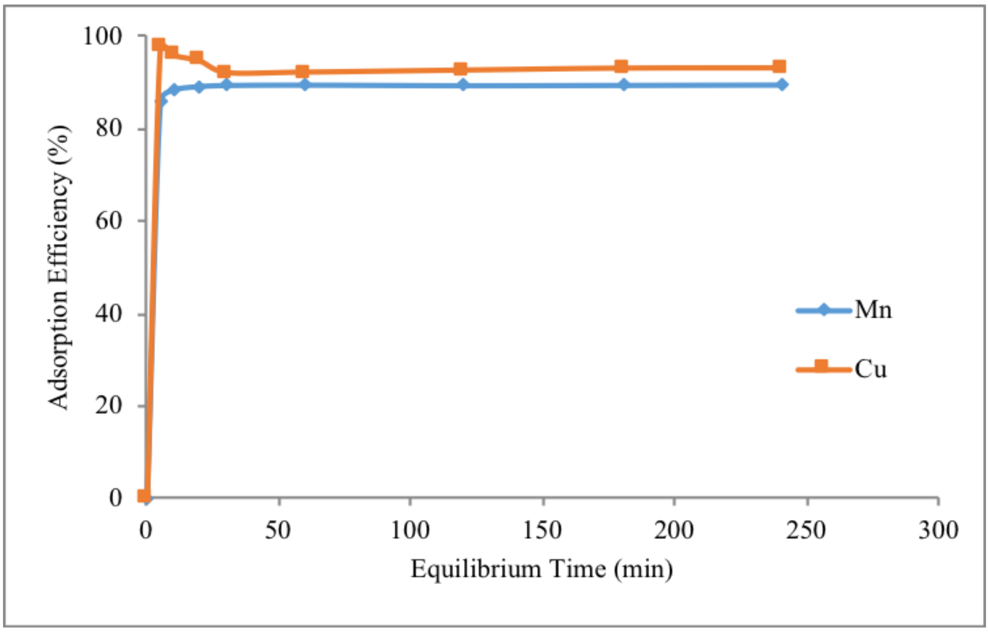

Figure 2. *Adsorption ratio depending on contact time of Mn^{2+} and Cu^{2+} heavy metals: adsorbent dosage - 0.5 g for manganese, 0.4 g for copper, 5 mg/L for initial concentration, pH - 5, T - 25 °C and shaking speed - 500 rpm*

Optimum adsorbent dosage

The adsorbate dose was studied by taking 0.1, 0.2, 0.3, 0.4, 0.5 g respectively. The adsorbent dosage experiment was carried out depending on the results obtained as a result of the experiments performed to determine the contact period. The amount of heavy metals adsorbed per gram of adsorbent was higher in the lower adsorption dose, higher in the higher adsorption dose. Although the removal efficiencies for Cu (II) were nearly similar, the best efficiency was 0.4 g of expanded perlite and the best efficiency of Mn was 0.5 g of expanded perlite. Zahar et al. found that 1 g of adsorbent dosing was sufficient for heavy metal removal from aqueous solutions (Zahar et al., 2015). The

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effect of the adsorbent dose in terms of heavy metal removal at an initial concentration of 5 mg/L manganese and copper is shown in *Figure 3*.

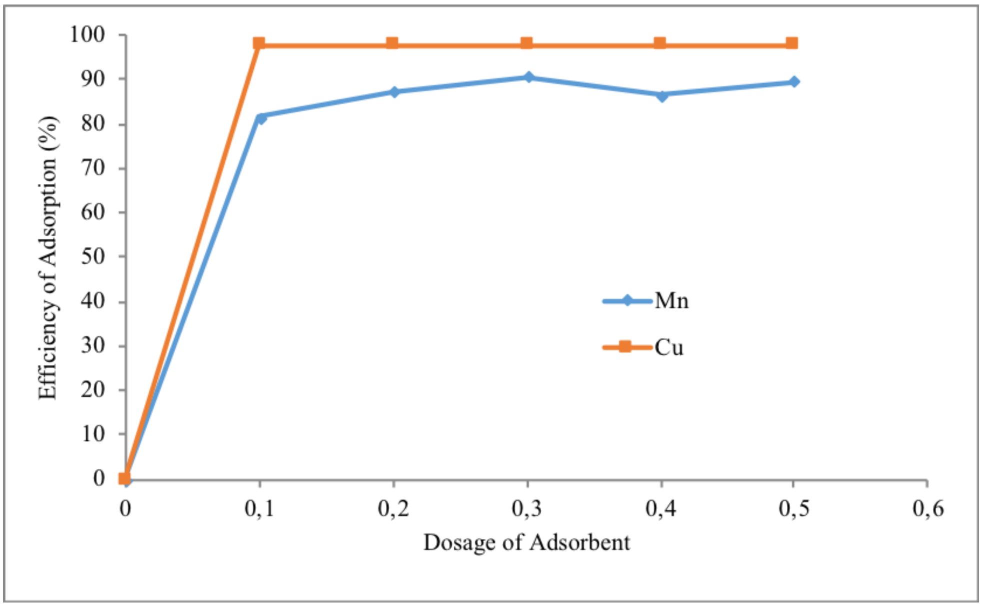

Figure 3. *The effect of the adsorbent dosage on the adsorption of Mn^{2+} and Cu^{2+} heavy metals: initial concentration - 5 mg/L, pH - 5, T – 25 °C and agitation speed - 500 rpm*

Effect of initial concentration

At different initial concentrations for each metal, 0.5 g adsorbent for manganese and 0.4 g adsorbent for copper, 25 °C of temperature, pH 5 for each heavy metal, shaking speed of 500 rpm, contact time of 30 min for manganese, 5 min for copper. The effect of the initial concentration on the adsorption yield according to the determined properties is shown in *Figure 4*.

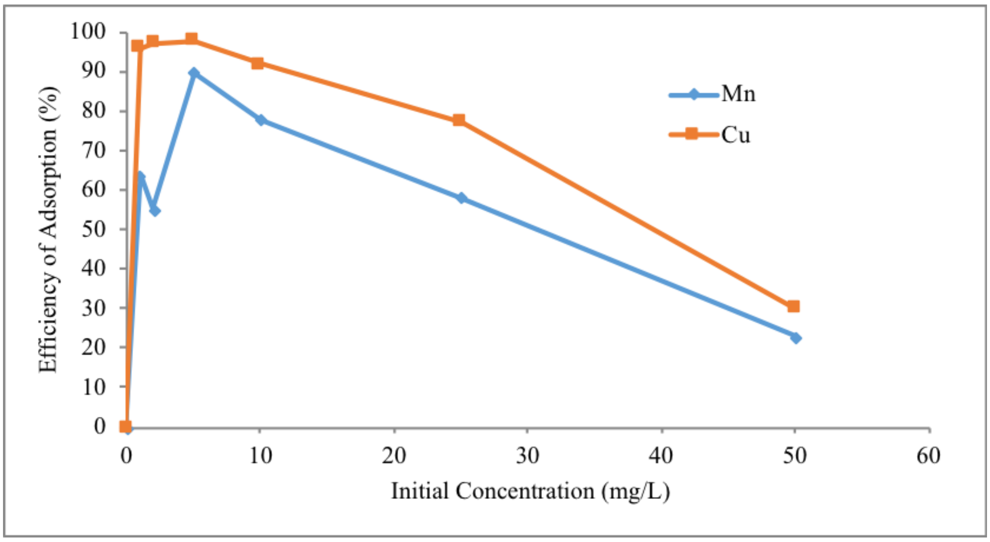

Figure 4. *Effect of initial concentration on the adsorption of Mn^{2+} and Cu^{2+} heavy metals: Adsorbent dosage - 0.5 g for Mn^{2+}, 0.4 g for Cu^{2+}, shaking time - 30 min for Mn^{2+}, 5 min for Cu^{2+}, pH-5, T- 25 °C and shaking speed - 500 rpm*

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Adsorption isotherms

Manganese adsorption isotherm

Freundlich and Langmuir isotherm curves and equations for manganese adsorption applied with regard to the expanded perlite are shown in *Figure 5a* and *b*. In *Table 3*, isotherm constants and correlation coefficients are given. At the initial concentration of 5 mg/L of Mn^{2+} heavy metal, the turning point was assumed to be 30 min since 0.5 g of expanded perlite achieved the highest adsorption value. In the study, Mn^{2+} heavy metals were found to conform to the Langmuir isotherm model with $R^2 = 0.93$.

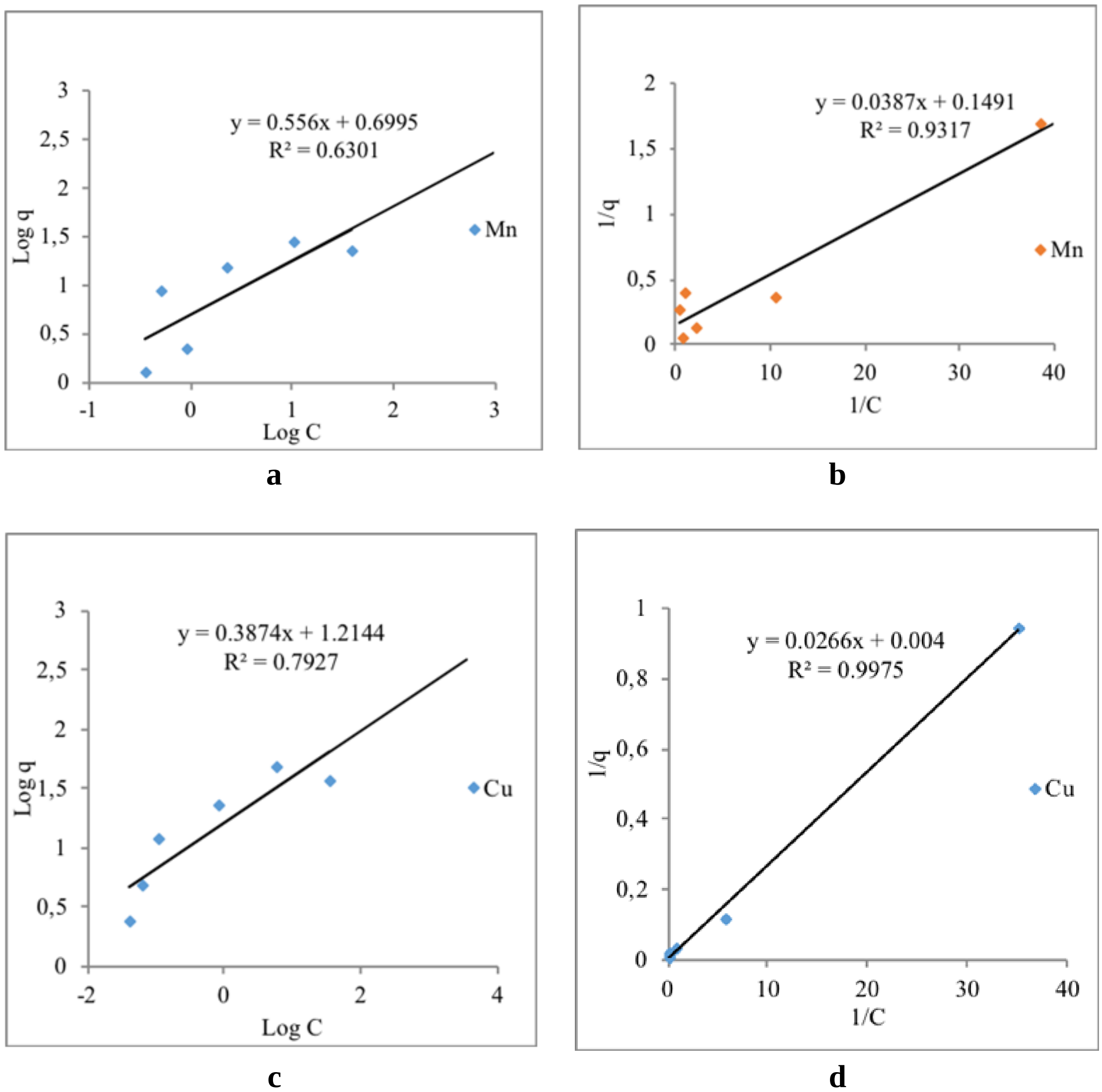

Figure 5. ***a*** *Mn^{2+} Freundlich isotherm.* ***b*** *Mn^{2+} Langmuir isotherm.* ***c*** *Cu^{2+} Freundlich isotherm.* ***d*** *Cu^{2+} Langmuir isotherm*

A correlation value of 0.93 for Langmuir isotherm was obtained. It can be said that Langmuir isotherm is compatible with this high correlation value. Langmuir isotherms indicate that the adsorbent surface is homogeneous, and the surface is covered with a single layer.

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Table 3. *Freundlich and Langmuir isotherm constants for manganese*

Freundlich isotherm	**K_f**	**$\log_{Kf}$**	**n_f**	**$1/n_f$**	**R^2**
	5.006	0.6995	1.7985	0.556	0.63
Langmuir isotherm	**$1/b.Q_0$**	**$1/Q_0$**	**b**	**Q_0**	**R^2**
	0.1491	0.0387	0.259	25.83	0.93

Copper adsorption isotherm

The Freundlich and Langmuir isotherm curves and equations for copper adsorption on the expanded perlite are shown in *Figure 5c* and *d,* respectively. In *Table 4,* the isotherm constants and correlation coefficients are given. At an initial concentration of 5 mg/L Cu^{2+}, the turning point was assumed to be 5 min, since 0.4 g of expanded perlite achieved the highest adsorption value at this time. In the study, Cu^{2+} heavy metals were found to conform to the Langmuir isotherm model with $R^2 = 0.99$.

Table 4. *Freundlich and Langmuir isotherm constants for copper*

Freundlich isotherm	**K_f**	**$\log K_f$**	**n_f**	**$1/n_f$**	**R^2**
	16.38	1.2144	2.58	0.38	0.79
Langmuir isotherm	**$1/b.Q_0$**	**$1/Q_0$**	**b**	**Q_0**	**R^2**
	0.004	0.0266	0.15	37.59	0.99

BET surface area of expanded perlite

BET surface area analysis was performed with the Quantachrome Nova Touch LX4 instrument. The BET device can detect surface area measurements, micro, meso and macro pore size and pore size distribution at low pressures and high resolution by physical adsorption method in solid or powder samples. Prior to the test, the samples are placed in the degas unit (up to 300 °C) which is vacuum-heated for purification and sludge treatment, after which the samples are analyzed with nitrogen gas, which is used as adsorbate in the liquid nitrogen temperature. As a result of these experiments, an “adsorption isotherm” is obtained which indicates how much nitrogen the substance holds at which pressure. Once the adsorption isotherm has appeared, parameters such as BET Surface Area (Single or Multipoint), Micropore Size Distribution (0.5 nm - 2 nm), Mesopore Size Distribution (2 nm - 50 nm), Total Pore Volume, Average Pore Size can be calculated. The surface area of the expanded perlite, adsorption, desorption and average pore width were measured. According to the results in *Table 5,* single point surface area of expanded perlite is 406.259 m^2/g, multipoint surface area of expanded perlite is 1171.73 m^2/g and the average pore size is 0.823 nm. The high surface area of the expanded perlite allows for the absorption of the absorbent feature. At the same time, the pore size is directly related to the diameter of the desired heavy metal molecule.

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Table 5. *Expanded of perlite values according to BET surface area analysis results*

BET analysis	Expanded perlite
Surface area	406.259 m^2/g
Pore volume	0.4605 cc/g
Total pore volume	0.4826 cc/g
Average pore size	0.823 nm
Molecular weight	28.0134 g
BJH* adsorption	143.344 m^2/g
BJH desorption	212.578 m^2/g
DH** adsorption	146.805 m^2/g
DH** desorption	221.815 m^2/g

*Barrett-Joyner-Halenda adsorption and desorption (BJH calculation is used for plotting a pore size distribution graph)
**Dollimore heal method (adsorption and desorption)

Conclusion

In this study, the expanded perlite was used as an adsorbent for the adsorption of heavy metals such as Mn (II) and Cu (II). The effects of contact time, pH, adsorbent dosage and inlet concentration parameters were investigated in adsorption experiments. According to the batch method, Mn^{2+} and Cu^{2+} ions showed fast adsorption tendency in 30 min for Mn^{2+} and 5 min for Cu^{2+} on the expanded perlite used as the adsorbent material as a result of the experiments to determine the turning point depending on the contact time. For both heavy metals these values were accepted as turning point. In the study where the amount of adsorbent is determined, the adsorbent amount in which a large part of the Mn^{2+} and Cu^{2+} concentrations are adsorbed is determined as 0.5 g/L for Mn and 0.4 g/L for Cu. It has been observed that the increase in the removal of the expanded perlite Mn^{2+} and Cu^{2+} is at pH 5. In the adsorption of Mn^{2+} and Cu^{2+}, $\log_q/\log_C$ values were plotted to determine the Freundlich isotherm, while Langmuir isotherm constants were determined by plotting $1/q$ and $1/C$ values. At an initial concentration of 0.5 mg/L Mn^{2+} heavy metal, the optimum point was assumed to be 30 min since 0.5 g of expanded perlite achieved the highest adsorption value at this time. In the study, Mn^{2+} heavy metals R^2 = 0.93 were obtained. It can be said that Langmuir isotherm is compatible with this high correlation value. Langmuir isotherm compliance indicates that the adsorbent surface is homogeneous and that the surface is covered by a single layer. At a concentration of 5 mg/L Cu^{2+}, the optimum point was assumed to be 5 min since 0.4 g of expanded perlite achieved the highest adsorption value at this time. In the study, Cu^{2+} heavy metals were found to conform to the Langmuir isotherm model with R^2 = 0.99. In this study, it has been found that economically expanded perlite can be used as an effective adsorbent material in the removal of heavy metals. Different processes can be developed by undertaking additional laboratory studies.

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