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SUSTAINABLE REGENERATION OF MORDENITE MINERAL AS ION EXCHANGER FOR REMOVAL IRON AND MANGANESE IN GROUNDWATER

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Abstract

Access to clean water is a basic need for humans. At present, groundwater quality is a fundamental consideration factor in its utilization. Metal removal in ground water with filtering techniques has been done quite a lot with various media, one of which is zeolite. However, the pore blockage often becomes a problem, so it inhibits the process and shortens the life of the stone in adsorbing iron and manganese, to overcome this problem regeneration is needed so that Mordenite can be reused. This research aims to study the method of regeneration chemically and biologically so as to improve the ability of Mordenite in removing iron and manganese by using

*a continuous bed reactor with up-flow system for 60 minutes. Activation and regeneration of Mordenite from natural zeolite is carried out chemically using NH_4Cl by immersion method, while biological regeneration is carried out by utilizes *Thiobacillus ferrooxidans* bacteria. The removal efficiency obtained will decrease as the adsorbent is used. Efforts to increase the adsorption capacity will continue to be carried out by giving chemical and biological regeneration. Furthermore, adsorption capacity and removal efficiency in each variation of Mordenite mineral adsorbents are included in the scope of the research.*

Keywords

Groundwater, Adsorption, Iron, Manganese, Mineral Mordenite, Regeneration, *Thiobacillus Ferrooxidans*

1. Introduction

Groundwater is a natural resource that is very important for life and a source of water in various human activities. Everyone knows that without water, there will be no life. However, excessive use of natural resources to support human civilization at the present level exerts excessive pressure on the environment. Excessive exploitation of ground water results in various problems, especially in terms of quantity. However, often the problem of decreasing the quantity of ground water which is closely related to land subsidence and ground water does not get serious attention and treatment (Sun, et al., 2009; Lijzen, et al., 2014). In addition, seeing the many and varied needs for groundwater, the quantity of groundwater is no longer the only thing that is of significant concern. At present, groundwater quality is a fundamental consideration factor in its utilization (Heath, 2004). Problems that are always present regarding the quality of groundwater are rooted in various human activities. The waste produced due to human activities, both in liquid, solid and gas forms, is a threat which if not anticipated early and precisely will be a factor causing a decrease in groundwater quality. There are still many people in various regions who use ground water to source their daily lives, agriculture, industry and other activities. Therefore, the quality and quantity of groundwater is important to maintain.

Groundwater pollution generally results from the discharge of human activities such as the remaining disposal of factories containing hazardous chemicals, pesticides, or because of the presence of metals in the groundwater, both toxic and essential. Iron and manganese are metal elements which are not uncommon in ground water. The existence of these two metals if the

concentration exceeds the quality standard of the Minister of Health Regulation Minister Decree No: 492/Menkes/Per/IV/2010 concerning Clean Water Quality Standards, it will be dangerous for humans. Iron is one of the chemical elements that can be found in almost every place on earth, in all geological layers, and all bodies of water. In general, iron in water can be dissolved as Fe^{2+} (ferrous) or Fe^{3+} (ferries) suspended as colloidal grains and combined with organic substances or inorganic solids such as clay. In surface water, iron content is rarely found greater than 1 mg/l, but in groundwater iron content is much higher due to the presence of sediments (Febrina and Ayuna, 2014). Iron is needed by the body in the formation of hemoglobin. Even though iron is needed by the body, but in large doses it can damage the intestinal wall because the body cannot excrete iron. Iron levels greater than 1 mg/l will cause irritation to the eyes and skin. If iron solubility in water exceeds 10 mg/l will cause water to smell like rotten eggs. Another metal element is manganese. According to Fauziah (2014), excess water containing manganese (Mn) causes taste, color (brown/purple/black), and turbidity. In small amounts (< 0.5 mg/l), manganese in water does not cause health problems, but is beneficial in maintaining healthy brain and bones, plays a role in hair and nail growth, and produces enzymes for metabolism that function to convert carbohydrates and proteins into energy. But in large quantities (> 0.5 mg/l), manganese in water is neurotoxic. Symptoms that arise in the form of symptoms of the nervous system, insomnia, then weak in the legs and facial muscles so that facial expressions become frozen and the face looks like a mask.

The presence of iron and manganese in high concentrations will endanger human health when utilizing ground water, therefore further processing is needed to reduce the concentration of iron and manganese before groundwater is utilized. There are several technologies that have been developed in reducing levels of iron and manganese in water, including ozone technology (El-Araby, et al., 2009), adsorption (Dewita, 2017), coagulation and flocculation (Fu-wang, et al., 2009), sand filter (Astari and Iqbal, 2009), filtration (Febrina and Ayuna, 2014), ultrafiltration (Choo, 2005), biofiltration (Thompson, et al., 2016). Dewita (2017) has conducted a study to reduce the levels of iron and manganese in water by the adsorption method that uses an adsorbent in the form of Mordenite minerals obtained from activated zeolites. The research was conducted with a batch reactor system by testing the ability of mordenite minerals to remove iron and manganese, determine the reaction isotherm and kinetics, and determine the most appropriate activation method in activating the Mordenite minerals contained in zeolite. Mordenite minerals that can be found in Sukabumi Green Natural Stone have high potential in

the removal of iron and manganese in water. Mordenite is a type of zeolite which has the most silica content with the formula compound $\text{Na}_8\text{Al}_3\text{Si}_{40}\text{O}_{96}\cdot 24\text{H}_2\text{O}$ (Opera, 2006). However, in its application for household scale, there are still problems, namely a short service life because adsorbents experience clogging so that a method is needed to carry out the regeneration process. This study aims to examine the ability of Mordenite mineral adsorption in removing iron and manganese in water and determine the most appropriate regeneration method so that it can extend the lifetime of minerals.

2. Experimental

2.1 Artificial Sample Preparation

The groundwater solution used in this study was an artificial sample made by mixing aquadest with $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$ to add the concentration of Iron and $\text{MnSO}_4\cdot \text{H}_2\text{O}$ to add Manganese concentration. By looking at the Larasati (2013) study, shallow groundwater that has an iron concentration greater than the Manganese concentration is groundwater in the Kopo area, Bandung. Therefore, this research was conducted by making samples with characteristics of Iron and Manganese which similar to shallow groundwater in the Kopo area, Bandung in 2013, which contained 0.535 ppm Iron and 1,519 ppm Manganese. Then the sample was varied to 0.5;1;1.5 ppm for Iron and 1.5;2;2.5 ppm for Manganese. Furthermore, the optimum concentration will be used in the adsorption process using an adsorbent that has been chemically and biologically regenerated by immersion method for 24;48;72 hours. The sample solution used is a single solution of Fe, Mn, and mixed solution.

2.2 Adsorbent Preparation

The Mordenite adsorbent used in this study was obtained from the destruction of Sukabumi Green Natural Stone containing Mordenite type Zeolite. The destruction of Sukabumi Green Natural Stone was carried out using a roll crusher and jaw crusher at the Metallurgy Laboratory ITB. To obtain the desired particle size, namely Zeolite particles measuring 40-70 mesh, sieving analysis was carried out. The particle size is based on the results of a study by Novandy (2014) in determining the optimum particle size for removal of Iron and Manganese in water.

In this study, Mordenite activation was carried out using a solution of 1M NH_4Cl . The use of NH_4Cl was based on the results of Novandy (2014) study, where the use of NH_4Cl would activate Mordenite better and be able to set aside more Iron and Manganese compared to

activation using NaOH, NaCl, and Na₂CO₃. Activation is done by soaking Mordenite in 1M NH₄Cl solution with a volume of 1.5 L for 24;48;72 hours. After soaking, Mordenite is filtered with Whatman 93 filter paper to separate the adsorbent and the solution. Next, the Mordenite adsorbent is washed with distilled water 3 (three) times and dried in an oven 100°C for 24 hours before it can be used as an adsorbent. This activation process is also used to regenerate chemically adsorbents. While biological regeneration will use the bacteria *Thiobacillus ferrooxidan*. The adsorbent will be soaked for 24;48;72 hours in a liquid that has been dissolved with bacterial isolates, then the process is the same as chemical regeneration.

2.3 Thiobacillus ferrooxidan Preparation

This study uses liquid media from Leathen et al. (1956) with a pH of 3.5. The media composition is presented in Table 1.

Table 1: Composition of Liquid Medium

Material	Composition
K ₂ HPO ₄	0,05 gram
(NH ₄) ₂ SO ₄	0,15 gram
Ca(NO ₂) ₂	0,01 gram
MgSO ₄ .7H ₂ O	0,50 gram
KCl	0,05 gram
FeSO ₄ .7H ₂ O	1,00 gram

(Source: Nurseha, 2000)

The chemical is mixed into 800 ml of distilled water except FeSO₄.7H₂O, stirred and sterilized at 121°C and cooled. For FeSO₄.7H₂O, the distilled water was prepared and the pH was set, which was pH 3.5, as much as 200 ml, after which FeSO₄.7H₂O was added and heated to 50°C, then cooled. The two solutions are then mixed. This media is then divided into isolation tubes that have been sterile. After that, bacteria are grown in the media and will be used as a regeneration material from the mordenite mineral.

2.4 Characterization of Mordenite Composite

Mordenite composite were characterized by Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) in Puslitbang Teknologi Mineral dan Batu Bara Bandung.

2.5 Continuous Experiment

Variations of conducted experiments were shown in Table 2.

Table 2: Conducted Experiments

Experiment	Adsorbent	Sample
I	Chemical Activation	Single Solution
II	Chemical Regeneration	Single Solution
III	Chemical Regeneration	Mixed Solution
IV	Biological Regeneration	Single Solution
V	Biological Regeneration	Mixed Solution

2.6 Parameter Measurement

2.6.1 pH and Temperature Measurement

pH and temperature are measured at the beginning and end of the experiment. This pH and temperature were measured by a pH meter and a digital thermometer by dipping the measuring probe into the sample to be measured. Before each set of measurements is carried out, calibration of the pH-meter and digital thermometer must be done by comparing the results of the reading with standard solutions with known characteristics

2.6.2 Measuring Efficiency of Iron and Manganese Removal

The effectiveness of iron and manganese allowance is calculated by measuring the difference in concentration of iron and manganese after and before being given treatment. The measurement of the concentration of Iron and Manganese was carried out by using the Flame AAS type Atomic Absorption Spectroscopy (AAS), which was conducted at the ITB Environmental Engineering Water Quality Laboratory. Measurements were made by dipping a small hose on AAS to enter and analyze samples at AAS. The results of reading the concentration of each water sample can be immediately known on the computer used for rendering AAS data. Furthermore, the removal efficiency was calculated using standard removal efficiency equation as shown below:

$$\text{Removal efficiency} = \frac{\text{Initial Concentration (ppm)} - \text{Final Concentration (ppm)}}{\text{Initial Concentration (ppm)}} \times 100\% \quad (1)$$

3. Result and Discussion

3.1 Characterization of Mineral Mordenite

The initial characterization for adsorbent was XRD (*X-Ray Diffraction*) methods to see the mineral/crystal type of Green Sukabumi Stone. Peaks 2θ intensities indicates that Green Sukabumi Stone consist of mainly pure zeolite mineral which is mordenite (Novandy, 2014). It

was confirmed by the result of XRD, which showed characteristic quartz, mordenite, albite, and illite before activation in the zeolite. For activated zeolite material, the characteristic was reported contain quartz, mordenite, albite, illite, and clinoptilolite which indicates the presence of NH_4^+ as the activated solution.

Utilization of natural zeolite minerals for heavy metal adsorption can be done by using or without using treatment, such as chemical activation which could enhance the adsorption capacity by producing a wider surface area through the formation of the porous structure and also eliminates polluting compounds. Chemical activation was conducted by using NH_4Cl solution. According to (Novandy, 2014), There is an exchange of NH_4^+ with alkaline ion on mordenite. The exchange will reduce the mass percentage of some elements so that the selectivity of mordenite to iron and manganese were increases. Figure 1 shows the result of SEM analysis on mordenite.

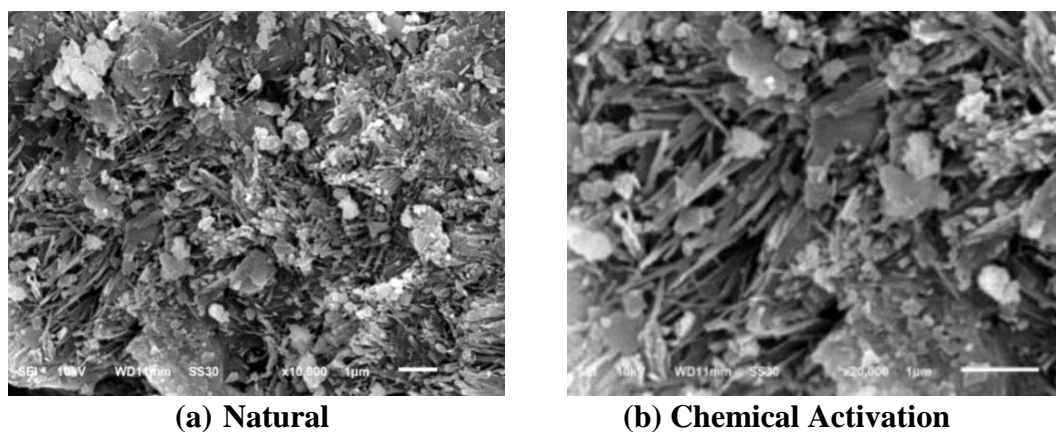
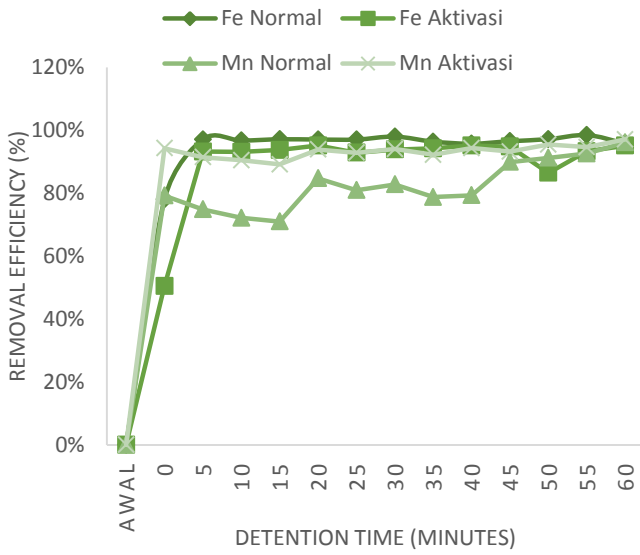


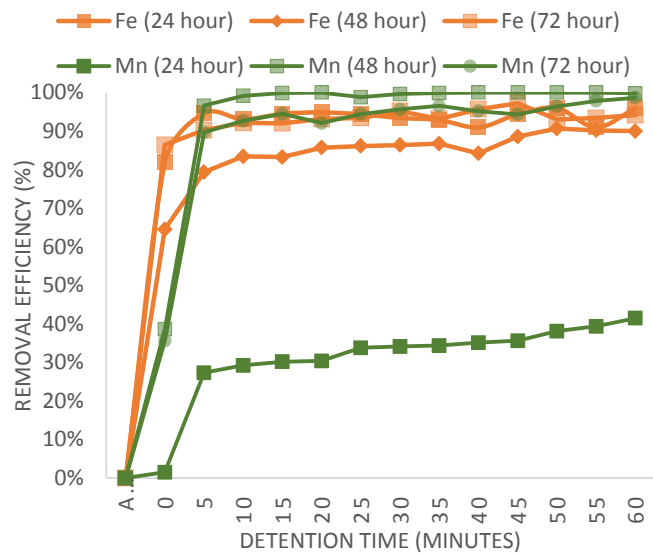
Figure 1: Mineral Mordenite

3.2 Iron and Manganese Removal Efficiency

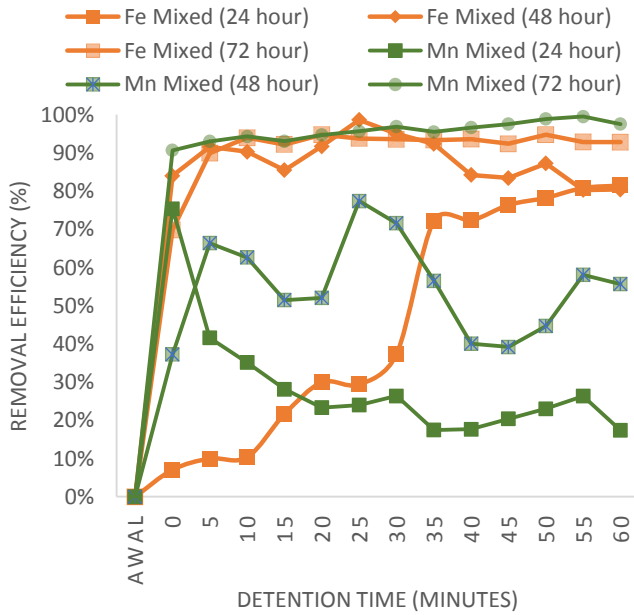
Results of each experiment are shown in Figure 2. Iron and manganese removal efficiency from batch system in experiment I was fluctuated but tend to increase with the longer detention time. According to (Sharma, 2001), The presence of ions and other compounds in the solution may affect the adsorption of adsorbate while according to (Lo, et al., 2012), heavy metal removal in batch adsorption process increases from the early stage until 60 minutes of detention time. Iron removal was also higher than manganese same as in the previous study (Novandy, 2014; Satria, 2015).



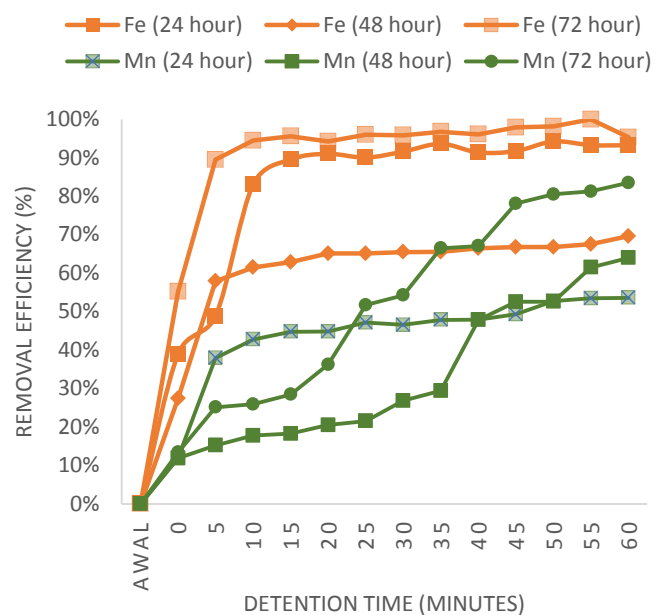
(a) Experiment I: Chemical Activation



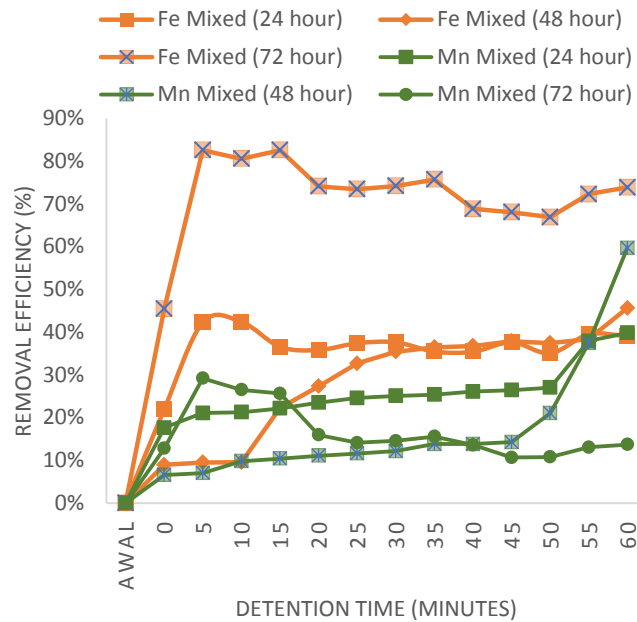
(b) Experiment II: Chemical Regeneration (Single)



(c) Experiment III: Chemical Regeneration (Mixed)



(d) Experiment IV: Biological Regeneration (Single)



(e) Experiment V: Biological Regeneration (Mixed)

Figure 2: Iron and Manganese Removal Efficiency

The result shows that the removal efficiency obtained will decrease as the adsorbent is used. Continuous experiments were able to remove iron and manganese removal and were successful to meet the standard. Result of experiment I shows that there are changes that occur between activated and normal minerals. The results show that the mineral activation process gives better results compared to non-activated minerals. Demir (2002) explained that there are two major factors which have positive influence over the NH_4^+ retention capacity of clinoptilolite, *i.e.* either by increasing relative content of alkaline metal cations (exchangeable cations) or by decreasing zeolite particle size (increasing surface area). The statement supports the phenomenon that occurs because NH_4^+ which is used as an ingredient in the activation process has an influence on clinoptilolite found in mordenite minerals. The effect given is to allow for an increasing surface area so that the absorption of iron and manganese becomes more effective.

On the other hand, chemical regeneration in experiment II-III was able to change the efficiency after the adsorbent used for several days, and the result is fluctuating for mixed solution. Different mechanism happened to affect the removal efficiency since activation could influence the initial characteristic of the used mordenite. Novandy (2014) said that chemical adsorption mechanism was slower than physical mechanism. Besides that, for the continuous process, the removal of iron and manganese were also influenced by the adsorbent characteristics

which could be changed during the reactor operation. The iron and manganese removal in continuous reactor using up-flow system was not only involved in the mechanism of adsorption and ion exchange, but also involved in the filtration mechanism. Goel (2005) added that removal for heavy metals, such as Fe^{3+} , Mn^{2+} , Zn^{2+} , and Cu^{2+} using zeolite adsorbents was not only involved in ion exchange mechanism, but also involved in the precipitation result of metal hydroxides from the solution.

Then it was continued to experiment IV and V that show significant difference of iron and manganese removal between single and mixed solution after biological regeneration. Bioregeneration is defined as the process by which the adsorbent surface is being renewed through microbial action. The important role of bioregeneration process in extending the adsorption capacity of adsorbents has been reported by many researchers (de Jonge et al., 1996a; Orshansky and Narkis, 1997; Silva et al., 2004; Syamsiah and Hadi, 2004; Lee and Lim, 2005; Aktas and Çeçen, 2007). Gamal (2018) added bioregeneration helps to increase the service period of the adsorbents which involves the use of microbial colonies to regenerate the capacities and surfaces of the carbon. According to Klimenko (2003), effective bioregeneration processes depend on numerous factors including reversibility of adsorption, the presence of microbial organisms capable of metabolizing the adsorbate, the settings of optimal microbial growth conditions including nutrients (nitrogen, phosphorus, sulfur, etc.) temperature, dissolved oxygen, etc. and optimization of microbial and adsorbate concentration ratios. Sirotkin et al. (2002) added the adsorption–desorption balance, the residence time and the spatial distribution of molecules in pores among other factors determining the efficiency of bioregeneration. *Thiobacillus ferrooxidans* provide important role in this process, because Thiobacilli constitute a group of Gram negative chemoautotrophic bacteria, which can obtain energy for growth from the oxidation of a variety of inorganic sulphur compounds. *Thiobacillus ferrooxidans*, an obligatory autotrophic species, can also oxidize iron (II) ions. This organism is very important in many mineral leaching operations, especially in the bacterial leaching of sulphide ores for metals recovery (Rossi, 1990; Ehrlich and Brierley, 1990; Barret, et al., 1993; Rawlings and Silver, 1995; Agate, 1996; Rawlings, 1998). Regeneration experiments were able to increase the efficiency removal. For iron removal, the efficiency was successful to reach more than 80%. When the mordenite take more time to contact with bacteria, the efficiency removal will increase as well. As can be seen, iron removal efficiency is higher than manganese removal efficiency. This shows that iron precipitation is more effective. But we cannot say that manganese is not

effective because minerals are still able to absorb manganese after going through the regeneration process. From the result of this experiment, we can conclude that biological regeneration has a significant effect on both parameters.

4. Conclusions

Adsorption experiments were performed to see the ability of natural mordenite green Sukabumi stone to adsorb iron and manganese ions. But the removal efficiency obtained will decrease as the adsorbent is used. To overcome this problem, a regeneration process is needed. The activated mineral gives better results compared to non-activated minerals. This phenomenon occurs because of the positive influence over the NH_4^+ retention capacity of clinoptilolite. On the other hand, biological regeneration also has a significant effect in extending the lifetime of mordenite mineral. Even though the removal efficiency of iron is more effective than manganese, but this process can still improve the ability of minerals to remove manganese. It happened because *Thiobacillus ferrooxidans* has a capability to oxidize iron (II) ions and use it as an energy source of life.

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