

Zevi & Septiyani, 2020

Volume 6 Issue 2, pp. 26-40

Date of Publication: 20th July 2020

DOI- <https://dx.doi.org/10.20319/mijst.2020.62.2640>

This paper can be cited as: Zevi, Y. & Septiyani E., (2020). Technology for Iron and Manganese Ion Removal from Groundwater: A Review. MATTER: International Journal of Science and Technology, 6(2), 26-40.

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TECHNOLOGY FOR IRON AND MANGANESE ION REMOVAL FROM GROUNDWATER: A REVIEW

Yuniati Zevi

*Department of Environmental Engineering, Faculty of Civil and Environmental Engineering, Bandung
Institute of Technology, Bandung, Indonesia*
yz59@cornell.edu

Elda Septiyani

*Department of Environmental Engineering, Faculty of Civil and Environmental Engineering, Bandung
Institute of Technology, Bandung, Indonesia*
eldaseptiyani23@gmail.com

Abstract

High concentrations of iron and manganese often cause issues. Based on grab sampling results in West Java, the average value of Fe concentration in groundwater is 0.97 mg/l and 0.64 mg/l for Mn. The results exceeded quality standard limit of 0.3 mg/l for iron and 0.1 mg/l for manganese. From these conditions, technology to remove iron and manganese is needed. One of the process to remove iron and manganese is to adsorb the two compounds by filtration method. The filtration method utilized mordenite minerals contained in Sukabumi Green Natural Stone. This study had two types of adsorbents which were activated and natural. Besides, the batch process in this experiment offers a result that activated and natural mordenite were able in diminishing the concentration of Fe and Mn from groundwater. Generally, batch experiment processes rely on the initial concentration and detention time during the adsorption, the process then carried out using continuous experiment. The continuous experiment process indicates clogging, so that the efficiency of removal obtained decrease

with the usage period of the adsorbent. Furthermore, regeneration is needed to make the lifetime of mineral be longer and can be reused. The regeneration method utilized chemical and biological regeneration.

Keywords

Groundwater, Iron, Manganese, Mineral Mordenite, Regeneration

1. Introduction

Many water resources are polluted regarding environmental health, especially groundwater. Indonesia's urban areas also have trouble having adequate access to clean water. The contamination of surface and groundwater occurs naturally as a result of rapid industrialization in the last few decades (Farrag, et al., 2016). According to Daniel (2011), the presence of iron and manganese in groundwater results directly from penetration by underground rock formation leading to natural substances. Most of the iron and manganese dissolve in aquifers and collect as the water flows through the rocks. In fact, the aquifers must act as a water supply.

In groundwater, the existence of iron is usually present in a soluble form as ferrous iron (Fe^{2+} or $\text{Fe}(\text{OH})^+$) or complexed forms as ferric iron (Fe^{3+}) and/or associated with organic matter (Ghosh, et al., 2008). Generally, iron is present together with manganese. Both ions may have adverse environmental and human impacts at some different levels. In addition, it may find in water at fluctuating concentration levels (Gage, et al., 2001). Iron is an important mineral for humans and has beneficial effects on health. Besides, high concentrations of iron in water may cause contaminations (Sarin, et al., 2004; Van Halem, et al., 2012; Bordoloi, et al., 2013). Usually, iron has various concentrations up to 3-4 mg/L, but in some cases, it may reach 15 mg/L (Ellis, et al., 2000). It can influence the quality of water even at low concentrations. Indeed, oxygen induces its rapid oxidation to produce ferric hydroxide or oxyhydroxide precipitation for $\text{pH} > 6$, which can produce toxic derivatives and develop infections such as neoplasia, cardiomyopathy, and arthropathy (Weinberg, et al., 1998).

Through minerals, rocks, soil, atmosphere, and various hydrosphere constituents, manganese (Mn) is found. The World Health Organization (WHO) estimated that Mn serves as an important micronutrient for the health maintenance of microorganisms, plants, and animals at a certain level to encourage growth and development. However, manganese may lead to toxicity in aquatic and terrestrial ecosystems above a certain deficiency level (WHO, 2004). Gonzales-Reyez, et al (2007) added that at small amounts of human blood, manganese may be convulsive or poisonous if found at extremely large rates of soil, water, and food. Considering the negative impact of iron and manganese

contaminant to human activity, an appropriate removal for iron and manganese from groundwater needs to be taken.

Water management is primarily concerned with rising the contaminant content to acceptable levels. This involves making sure that other things such as taste, scent, clearness, and color do not affect people. This also ensures that there are no operational issues in circulatory systems for the organic elements of water (Payment, 2009). There are several technologies that have been developed in reducing levels of iron and manganese, including ozone technology, adsorption, coagulation and flocculation, sand filter, biofiltration, ultrafiltration, and filtration (Choo et al, 2005; Astari and Iqbal, 2009; Fu-wang et al, 2009; Febrina and Ayuna, 2014; Demir, 2002; Thompson et al, 2016). According to Dewita (2017) which 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, still have problems. The mineral often clogging, causing short life span. To overcome this problem, a regeneration process is needed to extend the lifetime of minerals. The primary objective of this analysis would be the adsorption of iron and manganese ions with mordenite mineral concurrently extracted. The analysis discusses reactor configuration, flow setup, media forms used, the role of effective microbes involved and benefits and challenges of iron and manganese removal using mordenite minerals.

2. Iron and Manganese

2.1 Properties of Iron and Manganese

According to Marsidi et al (2018) study, the ferrous iron is soluble in two types which ferrous soluble iron and insoluble ferric particulate iron. This typically occurs therefore in the ferrous state. This is a smooth, ductile, blended, silver-gray (Periodic table Group VIII). Boiling and melting points is 1553 and 2750°C and a special gravity of 7,87 with a 55.845 g/mol molecular weight, respectively, are known (Lenntech Iron, 2016). Moreover, the breakdown of rocks and minerals, mine runoff, leachate wastewater or industrial wastewater may cause iron (Marsidi, et al., 2018).

Boiling and melting points of manganese are 2095 and 1244°C, respectively, and a certain weight of 7.44 for a molecular weight of 54.94 g/mol (Science Lab, 2016). Manganese reacts with water and diminishes in acid dilution. It is easy to oxidize but difficult to melt (Lenntech Manganese, 2016). Patil, et al (2015) added that manganese, aside from mechanical, electrochemical nutritional, and medicinal uses, is used mainly in ferrous metallurgy. This further increases the power and

longevity of the metal and its corrosion resistance. Manganese is also used as a catalyst. In plants and animals, it is important, but it may also trigger an animal body to be deformed (Patnaik, 2002).

2.2 Effects of High Iron and Manganese Concentration

An overdose of chemical elements can offer several problems. Sharma, et al (2005) explained that Fe and Mn cause esthetic and operational issues such as odor and brown, stain and deposition, resulting in high turbidity in water supply. The water treatment plant still has a concern owing to heavy iron and manganese content. It blocks the filter portion, shortens the backwashing intensity as hydrolysis and trivalent iron are used. When the raw water arrives from a well that usually produces more iron and manganese, this is more complicated (Qin, et al., 2008).

High levels of iron can contribute to serious health issues, including anorexia, diarrhea, malnutrition, gastrointestinal vein obstruction, and death (Yavuz, et al., 2005; Namdeo, et al., 2008). According to Patil, et al (2015), while essential to human health, manganese produces an unpleasant taste if it reaches a limit of 0.1 mg/L and induces environmental contamination. Manganese also forms a crust on tubes at a concentration of 0.2 mg/L, which can be lost as a black precipitation and leads to bio accumulation (Patnaik, 2002; WHO, 2011). Neculita and Rosa (2018) added that Mn is paramagnetic and can be found by magnetic resonance imaging (MRI) imaging. Weiss (2011) said that inhalation of airborne Mn may contribute to lifelong complications, such as neurological cardiovascular, and reproductive disorders (US DHHS, 2012; Rose et al., 2017). Guilarte (2010) and O'Neal and Zheng (2015) explained that an accumulation of manganese in the brain often results in specific type of Parkinsonism and neurological syndromes such as cognitive, psychiatric, and behavior disorders.

3. Properties of Mineral Mordenite

Mordenite is one of zeolite's type that can be used as a filtration media. This emerges from volcanic sediments with $(Ca,Na_2,K_2)Al_2Si_{10}O_{24}\cdot 7(H_2O)$ as a chemical composition. Natural stone green Sukabumi, West Java is one of the mordenite mineral resources. The large amount of waste products in natural stone cutting was expected to be a useful material in the water treatment process. With unique shape, green color, and mordenite compound content, green Sukabumi stone expected can be used as a filter (Novandy, 2014). Mordenite has an idealized chemical composition cell of $Na_8Al_8Si_{40}O_{96}\cdot 24H_2O$ which contain a ton of silica, the topological symmetry group of spatial units (Opera, 2006).

4. Technology for Removal of Iron and Manganese

4.1 Initial Characterization of Mineral Mordenite

According to Novandy (2014), 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. The crystal structure of Mordenite is a container that includes the five-membered ring chains of fibrous silicates and aluminum tetrahedra. SEM (Scanning Electron Microscope) methods was also performed to see the chemical composition and also the physical shape of the pore natural and activated mordenite of green Sukabumi stone (**Figure 1**).

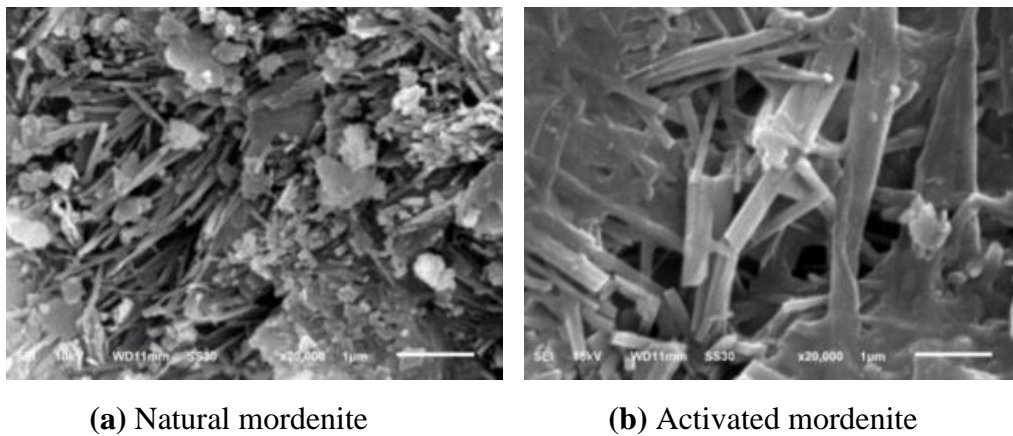


Figure 1: SEM magnification 20000x

Figure 1(a) reveals some features of natural mordenite's fibers surface morphology. The dark spots are those pores and cavities that can allow the solution flow into the pores and increase the adsorption kinetics. In **Figure 1(b)** activation using NH_4Cl shows mordenite pore closure that indicates NH_4^+ ions already attach on the surface of mordenite. This activation process can increase the specific surface area from $14.087 \text{ m}^2/\text{g}$ to $16,015 \text{ m}^2/\text{g}$ and reduce average pore diameter from $10,4455 \text{ nm}$ to $7,91808 \text{ nm}$ (Result of BET Surface Test). The increasing of adsorption surface area is one of the factor to increase adsorption capacity. NH_4^+ ions easily exchange to alkali and alkaline earth ions in zeolite. According to Panayatova (2001), activation by treatment with NH_4Cl causes the decrease in Ca^{2+} , Mg^{2+} , and K^+ . The decrease of some amount of alkali and alkaline earth cations in mordenite can be seen according to **Table 1**.

Table 1. EDS/EDX Test Results of Mordenite

Elements	%mass	
	Natural Mordenite	Activated Mordenite
C	5.57	3.81

O	46.86	49.66
Ca	1.95	1.1
Na	1.58	0.13
K	1.7	0.24
Al	7.28	7.63
Si	33.54	37.07
Mg	0.14	0
Fe	1.37	0.36
Mn	0	0
Total	100	100
Si/Al	4.607	4.858

Zeolite selectivity in the handling of gas, which is a major property of cations and anion. Selectivity is an exchangers' feature known as field strength in zeolite pore as the Silica and Aluminium ratio of the components. The increasing field strength value also increase the selectivity for cations like iron and manganese. For mordenite zeolite, the value of the field strength is usually around 4.17 to 5.0 (Margeta et al., 2013). This corresponds to the value of element ratio Si and Al natural mordenite around 4.607 and increase to 4.858 for activated mordenite. The increasing field strength value of activated mordenite shows that selectivity for iron and manganese ion also increase. Activation process using NH_4^+ ions can change the surface structure of mordenite thus influence adsorption capacity for iron and manganese ions.

4.2 Reactor Dimension

The 0.2 meter elevated, 0.045 meter in diameter fixed bed continuous reactor has an up-flow mechanism (Satria, 2015). The continuous reactors used during this research are shown in **Figure 2**. Configuration **(a)** is used without aeration. Configuration **(b)** is used with aeration as its pre-treatment. Activated Mordenite placed in the reactor gradually under saturated condition to make the media bed fixed for the entire running period. Water will gradually fill the reactor up on a certain velocity that has set based on each optimum hydraulic detention time. To make the flow superficial, porous media is set at the inlet and outlet of the reactor. Generally, effective use of a continuous reactor requires homogenous flow. A porous media is one example of a hold up to attain a more homogeneous flow with higher velocity (Fadavi, 2007).

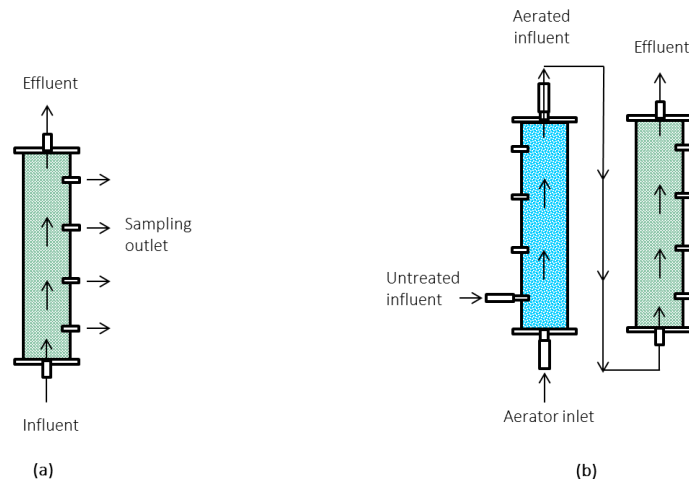


Figure 2: Fixed Bed Continuous Reactor for Experiment (a) Without Aeration and (b) With Aeration

4.3 Mordenite Activation

Activation is obtained through 2 methods. The first method is obtained by immersing zeolite in 1 M NH_4Cl for 24 hours. This method is used for regenerating the used zeolite up to twice regeneration. The second method is obtained by placing the zeolite and NH_4Cl in 250 mL Erlenmeyer flask and shook it by using 150 rpm shaker for 5 hours. After contacting the media, the Zeolite then filtered by using Whatman 93 filter paper to separate it from the NH_4Cl solution. The filtered media then rinsed 3 times by distilled water to clean up the excess salt, then dried in 100°C oven for 24 hours. Once it dried in 24 hours, it is ready to be stored and used for the experiment (Novandy, 2014).

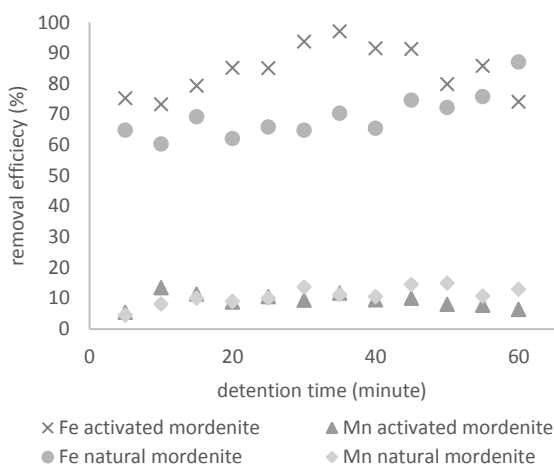
4.4 Batch and Continuous Experiment

Dewita (2017) reported that a batch analysis performed to examine the capacity of mordenite to adsorb iron and manganese from groundwater as it was the first attempt to determine mordenite's ability to use natural groundwater. Batch experiment was conducted with certain time detentions to determine the optimum value of detention time. After a lot trial that showed mordenite could absorb iron and manganese from groundwater, a continuous experiment was conducted. Continuous experiments aimed as an approach to know the ability of mordenite to be implemented in larger scale such as household water treatment. Continuous experiment was conducted with down-flow system. According to Ali (2013), adsorption process was optimized using batch system the followed by continuous bed system in order to applied continuously.

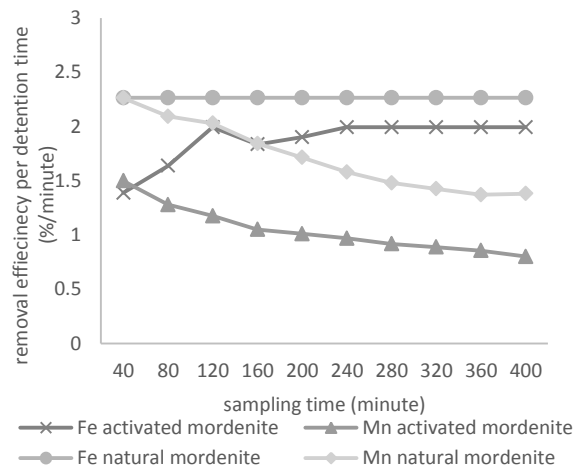
Adsorbents used both at batch and continuous experiment were in natural and activation state. First activation was conducted physically with $350\text{-}520^\circ$ of heating temperature. Physical activation was firstly selected in the research with practical consideration since it was easy and cheaper compared with chemical activation to be applied in larger scale. Then, chemical activation then was

finally conducted since it was the selected of optimum chemical activation from previous research by Novandy (2014).

In the previous experiment, there are four steps of the process. The four stages are carried out sequentially using the same adsorbent. According to Zevi, et al (2018) study, batch system in experiment I showed graph which tend to increase during the process. The reduction of iron was higher than manganese same as in the previous analysis of Novandy (2014) and Satria (2015). Based on Sharma (2001), the existence of ions and other compounds in the solution can impact the adsorption of adsorbate while according to (Lo, et al., 2012), from the earliest point until 60 minutes of detention time, reduction of heavy metal in batch adsorption was increased. Activated mordenite give more optimal results compared to natural mordenite. This happens due to the activation treatment which makes some pores open and increases the specific surface area. An increase in the surface area generate the adsorption capacity to increase as well. This situation is proven by the SEM test result as previously described. Result of experiment are shown in **Figure 3**.



(a) Experiment I: Batch System



(b) Experiment II: Continuous System

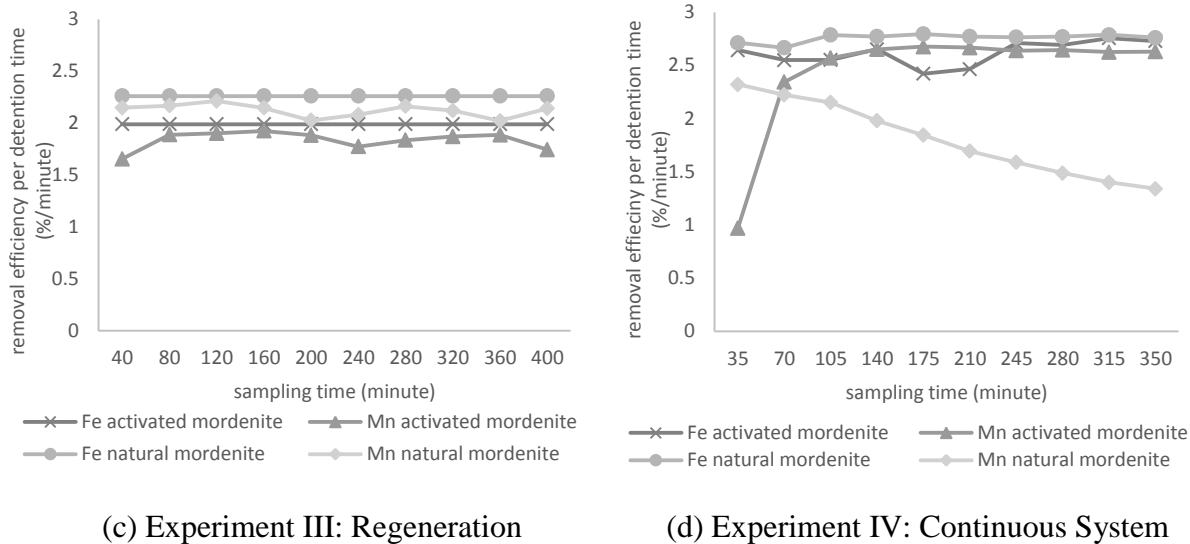
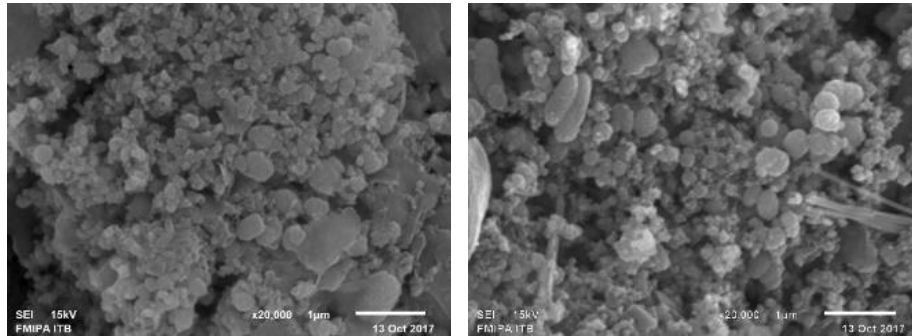


Figure 3: Iron and Manganese Removal Efficiency

The experiments were successful to reach the quality standard and reduce iron and manganese concentration. Satria (2015) reported that in experiment II, there was no major difference between physically activated mordenite and natural mordenite. Activating the adsorbent characteristics may influence the initial properties of used mordenite. In addition, Activation process using NH_4^+ ions can change the surface structure of mordenite thus influence adsorption capacity for iron and manganese ions. The method of chemical adsorption was reported by Novandy (2014) to be slower than the physique. Moreover, the processing of iron and manganese for the continuous process often depends on the adsorption properties that could be modified during the activity of the reactor. Throughout the continuous reactor using down-flow system in removing iron and manganese is not only part of the adsorption and ion exchange process but were also part of the filtration process. On the other hand, after the adsorbent used for several days in the process, the efficiency of chemical regeneration in experiment III was slightly increase. This happens due to chemical regeneration treatment which makes the adsorbate detached from the adsorbent surface. Regeneration treatment causes the adsorbent pores to open and able to re-adsorb contaminants in the solution.

Furthermore, at this stage, the initial adsorbent used is the adsorbent that has been given chemical regeneration treatment. The result in experiment IV showed significant difference between chemically activated mordenite and natural mordenite. Removal efficiency slightly increase in the beginning and continuous to decrease with the longer detention time. These results indicate that the adsorbent has reached its saturation point or clogging has occurred in the pores so that the adsorbent capacity also decreases. Goel (2005) added removal by zeolite adsorbent of heavy metals, such as Fe^{3+} ,

Mn^{2+} , Zn^{2+} , and Cu^{2+} required not only ion exchange processes, but also metal hydroxides precipitation of the solution. **Figure 4** shows that the adsorbent surface accumulation provides evidence of adsorption during the experiments.



(a) Natural mordenite

(b) Activated mordenite

Figure 4: Mordenite Condition after Adsorption Process

4.5 Bioregeneration

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 (Gamal, et al., 2018). Effective bioregeneration processes rely upon various factors such as reversibility of adsorption, the availability of adsorbent metabolized microbial species, the optimum conditions of microbial growth, including temperature nutrients (nitrogen, phosphorus, sulfur) and dissolve oxygen (Klimenko et al., 2003). Sirotkin et al. (2002) reported that the combination of adsorption desorption, residence time and spatial distribution of carbon pores molecules among other variables evaluating bioregeneration effectiveness. Another study demonstrated that using *Thiobacillus ferrooxidan* bacteria in regeneration process. The effective results are shown in **Figure 5**.

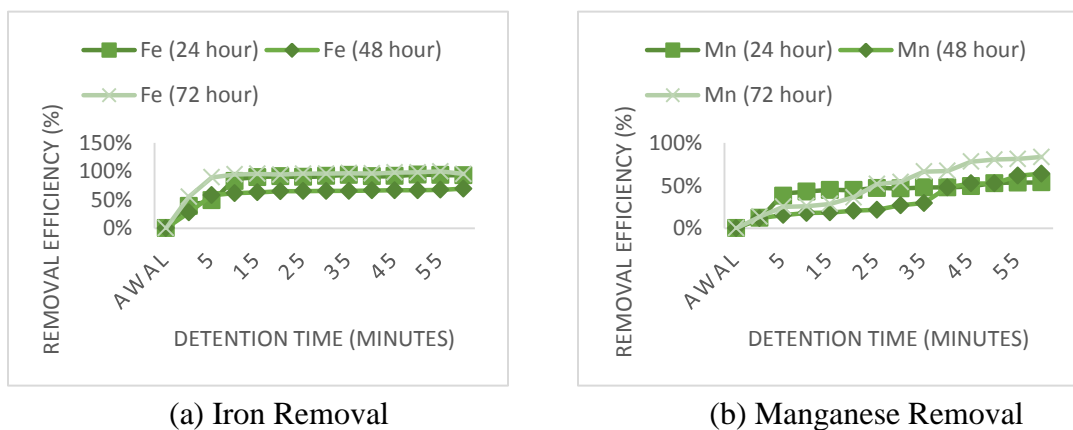


Figure 5: Iron and Manganese Removal Efficiency after Regeneration

Based on Septiyani (2018) study, 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. At the other side, the reduction of manganese capacity did not vary greatly. The result shows that the regeneration with the *Thiobacillus ferrooxidans* degrading bacterium extended the life of mordenite mineral significantly.

5. Conclusions

Adsorption for iron and manganese removal from natural groundwater utilized fixed bed continuous reactor which filled with mordenite offer a result that the mineral has a significant role to adsorb both iron and manganese. Chemical activation showed better result compared to natural mordenite. Based on EDS/EDX test result, the value of element ratio Si and Al of mordenite was increased from around 4.607 for natural mordenite to 4.858 for activated mordenite. The increasing field strength value of activated mordenite shows that selectivity for iron and manganese ion also increase. It can be concluded that activation process using NH_4^+ ions can change the surface structure of mordenite thus influence adsorption capacity for iron and manganese ions. However, the adsorbent often reached its saturation point, so the regeneration process is needed to overcome the clogging problem. Based on previous study, bioregeneration experiment utilized a *Thiobacillus ferrooxidans* which has a capability to oxidize iron ions, improve the ability of minerals to adsorb iron and manganese, and extending the lifetime of mordenite mineral.

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