Simultaneous Recovery of Ammonium and Phosphate from Leachate by Using Activated Zeolite

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Abstract. Ammonium and phosphate in leachate are potentially to contaminate both surface and groundwater. Zeolite has a high affinity so it can be used as an adsorbent for ammonium (NH₄⁺), phosphate (PO₄³⁻) and other organic compounds. This research work aims to eliminate these pollutants stimulatingly by using activated zeolite. Leachate used for this experiment has initial ammonium concentration of 508.2 mg/L, and phosphate concentration of 7.77 mg/L. The zeolite has particle size of 100 mesh. Adsorption experiments were carried out with physically activated zeolite and physically-chemically activated zeolite with a dose variation of 15 - 120 g/L with a contact time of 12 hours. The results showed that a physical activated zeolite dosage of 120 g/L results in the smallest concentration of ammonium residue of 72.6 mg/L and a phosphate residue of 0.37 mg/L. The 45 g/L chemically-physically activated zeolite dose of 45 g/L produces an ammonium residue of 198 mg/L and phosphate residue of 0.74 mg/L. The ammonium adsorption with the both activated zeolites can be described very well using the 1st order kinetics model.

Keywords: activated zeolite, ammonium, leachate, phosphate

1. Introduction

Leachate generation is a major problem for municipal solid waste (MSW) landfills and causes significant threat to surface water and groundwater. Leachate can be defined as a liquid that passes through a landfill and has extracted dissolved and suspended matter from it. Leachate results from precipitation entering the landfill from moisture that exists in the waste when it is composed [1]. Leachate also comes from rainwater that hits the disposed wastes or ground water from around the landfills.

Leachate contains various pollutants in high concentrations, such as organic matters, nutrients (nitrogen, phosphorus), and various metals including heavy metals [2]. Some pollutants in leachate are classified as difficult or cannot be degraded naturally, for example heavy metals, polymers and synthetic organic materials [3]. Many landfills in Indonesia are not equipped with adequate leachate management facilities. In many cases leachate is only collected in ponds and then immediately disposed of into the environment. This practice has the potential to pollute surface water and ground water.

In general, leachate treatment can be realized physically, chemically and biologically or in its combination. However, the physical and biological treatment of leachate is generally not able to produce the quality of leachate that meets the quality standards, especially seen from the content of ammonium, phosphate and heavy metals. Therefore, further treatment is needed to produce effluents which are safely disposed of into the environment [4]. One of the potential leachate treatment methods for this purpose is the adsorption with zeolite.
The removal of the ammonium from wastewater by adsorption with zeolites is found to promising. The advantages of the zeolite include high selectivity towards the ammonium ions in present of the cations in the wastewater, wide spreading in nature, and low cost [5]. Zeolites are natural, structured crystalline rocks, mainly composed of SiO$_4$ and AlO$_4$ bonds that are connected by oxygen atoms so that they have interconnected cavities in all directions [6, 7]. Elimination of ammonium from leachate by zeolite through a mechanism of ion exchange, which is the side of zeolite cations containing calcium ions exchanging with ammonium ions. Furthermore, these calcium ions react with phosphate and form deposits [7] [8]. On the other hand, geologically Indonesia has the potential to produce zeolites such as those found in Lampung, West Java, Central Java, East Java, East Nusa Tenggara, and Sulawesi with 447,490,160 tons of resources [9]. Similar values were also reported by the Ministry of Energy and Mineral Resources (2015) [10], where in Indonesia there were 432 million tons of natural zeolite spread in various regions. Moreover, used zeolite to adsorb ammonium can also be reactivated to be reused or used as slow release fertilizer [2].

The removal of the ammonium ions with zeolites is a result of ion exchange and/or adsorption. Both processes take place parallel, usually one of them is prevailing depending on the solid to liquid ratio [5]. The adsorptive of ammonium ions using zeolite has been studied by many researchers [5, 11, 12], however the interest towards experimental studies of this process for specific wastewater still exists. The reason exists in that fact that the effective of the process is strongly depend on the properties of the applied zeolite and the wastewater being treated.

The aim of this research works was to evaluate the zeolite ability to eliminate ammonium and phosphate from leachate water, with the main attentions given to the study of the effect of the activation method, zeolite dose and contact time on elimination of ammonium, phosphate and organic matter. The ammonium adsorption process by zeolite is described by the adsorption kinetics model.

2. Material and Method
2.1. Material
The materials used in the study consisted of leachate taken from the Galura landfill and natural zeolite taken from Lampung. The zeolite used for the experiment has a size of 100 mesh. The chemicals used for laboratory analysis included 3N NaOH, mengsel indicator, 2% boric acid, 6N NaOH, 0.02N H$_2$SO$_4$, K$_2$Cr$_2$O$_4$, COD (chemical oxygen demand) acid, and Fe indicator.

2.2. Equipment
The equipment used in this experiment included shakers, distillation devices, HACH spectrophotometers, pH meters, and turbid-meters. The supporting equipment consisted of filter paper, cup glass, Erlenmeyer, Mohr pipette, spatula, biuret, mass balance, sample bottle, 20 L jerry can, funnel, measuring cup, and bulb.

2.3. Research Procedure
Characteristics of leachate. Leachate is taken from the landfill of Galura in Bogor Regency. Leachate is put into jerry cans and stored in the cooling chamber. Leachate samples that have been taken then analysed the initial characteristics including measurements of pH, temperature, colour, turbidity, TSS (total suspended solids), ammonium, phosphate and COD concentrations using the method according to the APHA standard (2005) [13]. Leachate used for this study is blackish brown and has TSS, ammonium and phosphate content that are above the standard (table 1). This shows that the leachate requires treatment before being discharged into the environment.

Activation of Natural Zeolites. Activation of natural zeolite is intended to remove organic and inorganic impurities present in zeolite to improve its adsorption ability. Zeolite activation is carried out by physical treatment and chemical-physical treatment. A total of 100 grams of zeolite is first washed with 500 ml of distilled water. The washing process is carried out using a stirrer at a speed of 500 rpm at 100°C for three hours. After the washing process, zeolite is dried into an oven and crushed back to 100 mesh.
Furthermore, zeolites are activated physically and chemically-physically. Physical activation is carried out by heating zeolite into the furnace at 400°C for three hours. Chemical-physical activation is done by soaking zeolite into 3N NaOH solution using a ratio of 1:3 and stirring at a speed of 500 rpm at 80°C. Figure 1 shows natural zeolite and zeolite which have been physically activated and zeolite which is chemically-activated. Physical activated zeolite is pale white and cleaner than zeolite that has not been activated. This is because the calcination process of water vapour which is firmly bound to the bond structure of the zeolite evaporates and the impurities that are bound weakly to the main zeolite bond melt. Zeolites activated with bases have a yellow colour. This is due to the formation of an oxide compound as a result of the reaction between zeolite and NaOH [16]. The difference in this activation method affects the ability of zeolites to absorb ammonium and other pollutants.

| Parameter   | Unit | Quality standard | Value          |
|-------------|------|------------------|----------------|
| pH          | -    | 6 – 9            | 7.7 ± 0.0      |
| Temperature | °C   | -                | 24.3 ± 0.0     |
| Color       | PtCo | -                | 3190 ± 240     |
| Turbidity   | NTU  | -                | 128.5± 3.5     |
| TSS         | mg/L | 100a             | 459.5 ± 4.95   |
| Ammonium    | mg/L | 60a              | 508.2 ± 0.0    |
| Phosphate   | mg/L | 5b               | 7.77 ± 0.25    |
| COD         | mg/L | 300a             | 593.2 ± 0.0    |

*aRegulation of Ministry for Environment and Forestry No. 59/menlhk/Setjen/kum.1/7/2016 Regarding Wastewater Quality Standard [14]

*bIndonesian Government Regulation No. 82/2001 [15]

![Figure 1. Natural zeolite (a), physical activate zeolite (b), and chemical-physically activated zeolite (c)](image)

**Zeolite Adsorption Process.** The study consisted of two stages, namely the first stage was to determine the adsorption rate during the adsorption process, and the second stage was determining the dose of zeolite. In the first stage, adsorption was carried out by inserting 250 ml of leachate into a 250 ml Erlenmeyer. Leachate water is then added to activated zeolite. Leachate water is stirred with shakers at 200 rpm for 1, 3, 6, 12 and 24 hours. At this stage, the study was carried out with a zeolite dose of 60 g/L leachate.

In the second stage, the adsorption research was carried out by varying the dosage of zeolite, with ranges of 15, 30, 45, 60, 75, 90, 105, and 120 g/L. In this research, 12 hours of adsorption time was
applied, because it was considered sufficient to achieve the steady state according to the results of the preliminary experiments. After the adsorption process, leachate is deposited for 8 hours then filtered with filter paper and stored in sample bottles for analysis of its characteristics. All experiments were carried out with two replications for each treatment.

3. Results and Discussion

3.1. Changes in pH

The longer the contact time with zeolite, the pH of leachate increases. An increase in pH occurs because of the presence of alkali cations released into leachate during the adsorption process [17]. Chemical-physical activated zeolite increased the pH of leachate water to 9.98 during a contact time of 24 hours. Physically activated zeolite increases the pH of leachate water to 7.97 at the same contact time. Chemical-physical activated zeolite increase pH higher than physical-activated zeolite. Activation of zeolite with NaOH causes zeolite to absorb Na$^+$ and release OH$^-$ when in leachate so that the pH of leachate becomes more alkaline [18]. Leachate also tends to experience an increase in pH as more zeolites are added. The highest pH change in chemically-activated zeolite was obtained from a zeolite dose of 120 g/L, namely up to pH 9.3. Physically activated zeolite gives the highest pH change at a zeolite dose of 15 g/L which is to pH 7.97. The effect of adding zeolite to changes in pH can be seen in figure 2.

Ammonium in the bulk solution exists in both ionized and molecular forms, so that pH and temperature of the solution affect the forms of ammonium in solution. When pH<7, more than 95% of the ammonium existed in ionized form (NH$_4^+$); when pH approached 11, only about 1% of ammonium was left in ionized form [19].

3.2. Ammonium Removal

Figure 3 shows a decrease in ammonium concentration in leachate during the adsorption time (a), and a decrease in concentration at various doses of activated zeolite (b). Chemical-physical activated zeolite can reduce ammonium concentration greater than physically activated zeolite. This shows that physically-chemically activated zeolite has a higher ammonium adsorption capacity compared to physically activated zeolite. The results of this study are in line, and thus confirm the results of the Ngapa (2017) [20]. The highest decrease was 24 hours with residual concentration of 151.8 mg/L for zeolite chemically-activated and 257.4 mg/L for zeolite physically activated. The time of 1 h was chosen as the best contact time based on the effectiveness factor. Further increase in contact time did not show only a slight decrease in ammonium concentration (figure 3).

Figure 2. Effect of contact time (a) and activated zeolite dose (b) on pH of leachate
Increasing the dose of zeolite is expected to provide a greater decrease in ammonium concentration [21]. The highest decrease for chemically-physical activated zeolite occurred at a dose of 45 g/L with residual ammonium concentration of 198 mg/L. While for physically activated zeolites, the highest ammonium reduction was obtained from a dose of 120 g/L with a residual ammonium concentration of 72.60 mg/L. The adsorption with chemical-physical activated zeolite increased again after the zeolite dose of 60 g/L. This is due to a change in the pH of the leachate which is getting bigger (pH> 9) with increasing doses of zeolite. Ammonia at pH 5 to 7 takes the form of ammonium ion (NH\textsubscript{4}\textsuperscript{+}) which is the main compound that can make ion exchange so that in this condition the efficiency of decreasing ammonium is high. Above pH 8 the equilibrium shifts rapidly toward ammonium instead of its ion (NH\textsubscript{3}) which makes ammonia the dominant compound in leachate. Ammonia has a low ion exchange ability so that at pH> 8 ammonium reduction efficiency is lower [22]. In addition calcium ions in physically activated zeolites have a higher exchange rate than sodium ions bound to chemical-physical activated zeolites, so that at the same dose and contact time the physically activated zeolites can reduce ammonium more than chemical-physically activated zeolites. Therefore, the dose of 120 g/L for physically activated zeolite and the dose of 45 g/L for physically activated zeolite were chosen as the optimum dose in reducing of leachate ammonium.

### 3.3. Phosphate Removal

The addition of zeolite at each contact time produced residual concentrations ranging from 0.61 - 0.84 mg/L for physically activated zeolites and amounted to 3.18 - 4.40 mg/L for chemically-physically activated zeolites. The lowest residue was obtained at contact time of 6 hours with the physically activated zeolite and a contact time of 24 hours with chemically-physically activated zeolite. The decrease in phosphate based on the contact time between zeolites and leachate can be seen in figure 4.

The capacity of zeolite to reduce phosphate is achieved optimally under conditions of neutral to slightly alkaline solutions [23]. The use of chemically-physically activated zeolite results in the pH of leachate very alkaline (pH> 9) so that the decrease in phosphate becomes lower. The contact time of 12 h was chosen as the best contact time as the contact time for decreasing ammonium concentration. The chemically-physically activated zeolite dose of 15 g/L produced the smallest phosphate residue, which was 0.53 mg / L. The smallest phosphate residue of 0.37 mg / L was obtained at a dose of 120 g/L zeolite physically activated. The efficiency of phosphate reduction increases with increasing doses of zeolite because of the increase in the number of adsorbents and calcium ions which can precipitate phosphate [24] (figure 4). The mechanism of elimination of dissolved phosphate due to the reaction of ions released from zeolite with phosphate dissolved and forms a solid phase (and settles).
Figure 4. Effect of contact time (a) and activated zeolite dose (b) on phosphate concentration

3.4. COD Removal
COD shows the total amount of oxygen needed to oxidize organic matter which is easily and decomposed and also difficult to decompose in waters [25]. Thus, COD can be used as an indicator of the amount of organic matter in water. The decrease in COD concentration based at various contact times is shown in figure 5.

Figure 5. Effect of contact time (a) and effect of zeolite dose (b) on COD leachate

Chemically-physically activated zeolite and physically activated zeolite produced the lowest residue at a contact time of 24 h, which was equal to 304.88 mg/L and 269.17 mg/L, respectively. The COD concentration of leachate tends to decrease with increasing the contact time. According to research by Amosa et al. (2014) [26], zeolite can reduce the COD concentration maximum at a contact time of 80 minutes and become saturated after that. The saturated zeolite adsorption ability decreases so that the longer the contact time, the less COD decrease occurs. The chemical-physically activated zeolite dose of 15 g/L produced the smallest COD residue of 329.60 mg/L while the smallest COD residue of 315.87 mg/L was obtained at a dose of 120 g/L for physically activated zeolite. The more doses of zeolite are added, the greater the COD concentration due to increased adsorption capacity [27]. The concentration
of COD of the leachate after adsorption is still above the quality standard. The results of the study by Malekmohammadi et al. (2016) [28] also showed that zeolite had a low ability to adsorb COD and was only able to reduce 10% of COD concentration. Therefore, zeolite is suggested to only be applied as an advanced leachate treatment, after leachate is treated biologically to eliminate as much as organic matter.

3.5. Kinetics of Ammonium Adsorption

The rate of elimination of ammonium from leachate can be described using the help of the kinetic model. Adsorption kinetics describes the rate of adsorption of a substance by an adsorbent in a certain period of time [29]. There are generally four kinetic models that can be used, namely order 0, 1, 2, and 3. The mechanism involved during the present sorption process and the potential rate controlling such as chemical reaction processes was also studied using kinetic models by Kučić et al. [29]. The kinetic parameters are helpful for the prediction of adsorption rate, which gives important information for designing and modelling the processes.

Based on the results of this study, both physical activated and chemical-physically activated zeolites follow the order 1 kinetic model. First order reaction is a reaction whose reaction rate depends on one substance that reacts or is adsorbed. The equation of the first order kinetics model can be expressed by the equation: \( \ln C_e = -k \cdot t + \ln C_0 \). From the equation, it can be seen that the relationship between \( \ln C_e \) vs \( t \) is linear, with the slope \( k \) and intercept \( \ln C_0 \). The \( \ln C_e \) vs \( t \) plot results in a slope \( k \) for physically activated zeolite and chemical-physically activated zeolite as presented in table 2. The results of zeolite doses between 15 - 120 g/L showed adsorption by physically activated zeolite following the 1st order kinetics model: \( \ln C_e = -0.011 \cdot t + 5.818 \) with \( R^2 = 0.989 \), while adsorption with chemical-physically activated zeolite follows the 1st order kinetics model: \( \ln C_e = -0.032 \cdot t + 5.815 \) with \( R^2 = 0.980 \) (figure 6). \( C_e \) is the ammonium concentration in effluent, \( C_0 = \) initial ammonium concentration (mg/L), \( k = \) the reaction rate coefficient (h\(^{-1}\)), \( t = \) contact time (h). The results of the distribution of adsorption kinetics can be seen in figure 6.

| Model kinetika | Persamaan | Physical activated zeolite | Chemical-physical activated zeolite |
|---------------|-----------|---------------------------|------------------------------------|
| Fist order    | \( \ln C_e = -k \cdot t + \ln C_0 \) | 0.989 0.0111 | 0.980 0.032 |

Remarks: \( C_e = \) influent ammonium concentration effluent (mg/L), \( C_0 = \) initial ammonium concentration (mg/L), \( k = \) the reaction rate coefficient (h\(^{-1}\)), and \( t = \) contact time (h).

Based on the picture, the ammonium concentration obtained from the study corresponds to the ammonium concentration obtained from theoretical calculations following the kinetics model. This means that the physically activated zeolite kinetics and physically-chemically activated zeolite models are in accordance with the 1st order kinetic model. The first order kinetic model equation can then be used to design the adsorption system for leachate treatment. By determining the concentration of ammonium in effluent (\( C_e \)), and knowing the concentration at influent (\( C_0 \)), the time of adsorption can be determined.
4. Conclusion and Recommendation

4.1. Conclusion
Most leachate pollutant parameter values are above the specified quality standard. The optimum contact time of zeolite is physically activated and chemically-physically activated which is 12 hours. The optimum dose of physically activated zeolite was 120 g/L and produced the smallest residues for ammonium and phosphate, each at 72.6 mg/L and 0.37 mg/L. Changes in pH during the non-significant adsorption process occurred with the addition of a dose of physically activated zeolite, i.e., pH ranged from 6-8. The optimum dose for physically-chemically activated zeolite was identified at 45 g/L. Ammonium residue obtained was 198 mg/L and phosphate residue was 0.74 mg/L. The zeolite adsorption rate is physically activated and chemically-physically activated following the first order kinetics model, with the equation $\ln C_e = -0.0111t + 5.8182$ for zeolite physically activated and $\ln C_e = -0.032t + 5.8152$ for chemically-physically activated zeolites. With the help of the model, the adsorption time ($t$) needed can be determined if the ammonium concentration in effluent ($C_o$) is determined and ammonium influent ($C_o$) is known.

4.2. Recommendation
Further studies on the effect of pH on the ammonium adsorption process are needed for better improvement of leachate treatment designs.

5. References

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