

The Analysis of the Ability NaOH Activated Rice Husk Ash Silica to Reduce Cl Ion Levels

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Abstract

Research has been carried out to analyze the ability of NaOH-activated rice husk Ash Silica to reduce Cl Ion Levels. This study aimed to determine the optimum mass, optimum pH, optimum contact time and apply NaOH-activated rice husk ash silica to reduce Cl ion levels in seawater. The method used in this study was an experimental method with the parameters studied, namely optimum mass, optimum pH, optimum contact time, and the ability of NaOH-activated rice husk ash silica to reduce Cl ion levels in seawater. The results showed that the optimum adsorbed percentage of Cl ions occurred at an optimum mass of 0.025 gram, optimum pH of 7, and optimum contact time of 30 minutes with an adsorbed percentage of 45.7% and an adsorption capacity of 9.14 mg/g. The Cl ion content of seawater samples that can be reduced using NaOH-activated rice husk ash silica is 16.925 ppm with an adsorbed percentage of 33.85%, so it can be concluded that NaOH-activated rice husk ash silica can be used to reduce Cl ion levels.

Keywords: Rice Husk Ash, Silica, NaOH Activation, Cl Ion, Seawater

INTRODUCTION

Water is the most essential need in human life, especially clean and healthy fresh water, but not all areas have good water resources, one of which is in coastal areas. The largest water source, seawater, surrounds people who live in coastal areas, but they do not know how to convert this seawater into fresh water to meet their needs.

Seawater contains various dissolved salt elements. These elements consist of Chloride 55%, Sodium 30.6%, Sulfate 7.7%, Magnesium 3.7%, Calcium 1.2%, Potassium 1.1%, Bicarbonate 0.4%, and Bromide 0.2% (Paweka, 2017). Chloride content is one of the differences between freshwater and seawater (Muaya et al., 2015). This excess chloride can decrease water quality caused by high salinity (Rabbani et al., 2015). Therefore, it is necessary to apply a treatment process by reducing the chloride level in seawater so that it can be fresh water.

The desalination method is one of the alternatives to the freshwater supply in coastal areas. Various forms of desalination methods include adsorption processes and ion exchange processes. One of the adsorbents that has good prospects is rice husks. If the rice husk is in the form of rice husk ash, the main content that can act as an adsorbent is silica in the form of SiO₂, with a content of 86.90-97.30% (Harimu et al., 2019). Silica

is derived from rice husk ash and can be used as an adsorbent because it contains a silanol group (-SiOH), the structure and pore characteristics of silica in rice husk do not change (remain amorphous) until combustion to a temperature of 600-750 °C, and have a relatively high specific surface area, so it can be used as an adsorbent (Masrofah, 2017).

The problem with using rice husk ash as an adsorbent is generally because it has low adsorption power, so activation is needed to increase its adsorption power. Rice husk ash can be activated by soaking it using chemicals such as NaOH. Chemical activation with NaOH can enlarge the surface area due to the loss of silica that covers the adsorbent pores. Na₂SiO₃ formed from the reaction between NaOH and SiO₂ in adsorbents tends to be easily soluble in water, so when the adsorbent is washed with aquadest, Na₂SiO₃ will be dissolved (Van & Thu, 2019). This reduction in Na₂SiO₃ will open the adsorbent pores, increasing the surface area. In addition, in this activation process, the OH⁻ ion from the NaOH solution can add an active OH⁻ group on the surface of silica so that silica can be used as an adsorbent and an ion exchange medium.

Cl ion adsorption will be maximum if carried out under optimal conditions. These optimum conditions include optimum mass, optimum pH, and optimum contact time of the adsorbent. The optimum pH occurs at pH 7 with an adsorbed percentage of 89.384. The

optimum contact time for the adsorption of Cl ions is 4 hours, with an adsorbed percentage of 90.138%. Sahra et al., (2021) has also conducted a study on the adsorption of Pb²⁺ and Cd²⁺ using NaOH-activated rice husk ash silica, obtaining an adsorption percentage of Pb²⁺ of 88.12% and an adsorption percentage of Cd²⁺ of 90.44% in the pH range of 6.

The absence of research on the adsorption of Cl ions using NaOH-activated rice husk ash silica makes researchers increasingly interested in knowing more about the ability of NaOH-activated rice husk ash silica to reduce Cl ion levels in seawater. The analysis of the ability of NaOH-activated rice husk ash silica to reduce Cl ion levels in seawater is essential. This reason is intended to be used as a data source for rice husk ash, which can be used to reduce Cl ion levels in seawater.

METHODOLOGY

Materials and Instrumentals

The equipment used in this study is analytical scales, magnetic stirrers, burets, Erlenmeyer flasks, measuring flasks, volume pipettes, measuring pipettes, goblet glasses, droppers, filters, statics, clamps, hotplates, spatula, glass funnels, ovens, spray bottles, universal pH, desiccants, porcelain cups, stirring rods, pH meters, and stopwatches. The instrumental equipment used is a Fourier Transform Infrared (FTIR) spectrometer (PerkinElmer Spectrum) to identify functional groups. The materials used in this study are aquadest, seawater, NaCl, sodium hydroxide (NaOH) solids, H₂SO₄ 98%, HCl, AgNO₃, K₂CrO₄, filter paper, and rice husk ash from rice milling in Lapulu Village, Tinanggea District, South Konawe Regency, Southeast Sulawesi.

Procedure

Separation of Silica from Rice Husk Ash Using 12% NaOH

40 grams of rice husk ash was put into a 500 mL beaker. The beaker was added with 240 mL of NaOH with a concentration of 12%, then heated at 85°C for 90 minutes while stirring using a magnetic stirrer. After that, it was filtered using filter paper, and then the filtrate was neutralized with 0.5 M sulfuric acid until there was a gel precipitate and let stand for 18 hours. The gel precipitate formed was then filtered, washed using hot distilled water, as much as 1000 mL, dried in an oven at 110 °C for 5 hours, and weighed, and the percentage of silica yield was obtained (Harimu et al., 2019).

$$\text{Silica Yield} = \frac{\text{Weight of silica}}{\text{Sample weight}} \times 100\% \quad (1)$$

Activation of Rice Husk Ash Silica with NaOH Solvent 0.2 M

Chemical activation was carried out by adding a base NaOH solution of 0.2 M. Silica of rice husk ash was put into the NaOH solution with a ratio of 1 gram of silica of rice husk ash in 20 ml of solution. The beaker was covered with aluminium foil and stirred using a magnetic stirrer for 2 hours. After 2 hours, they were allowed to stand for 24 hours. Then, it was filtered using filter paper, rinsed with aquadest, and dried in the oven at 105 °C for 3 hours until it was constantly weighed. The drying result was cooled in a desiccator (Rahimawati et al., 2019).

Determination of Optimum Mass for Adsorbing Cl Ions

4 pieces of 100 mL beaker were prepared containing 50 mL of 50 ppm Cl ion solution each and the pH was adjusted to pH 9 with NaOH. Then, rice husk ash silica was put into four pieces of the beaker with a sequentially varied mass of 0.01, 0.025, 0.05, and 0.1 grams. The beakers were covered with aluminium foil and stirred using a magnetic stirrer for 30 minutes. After that, it was filtered, and the filtrate was stored in a beaker. Then, the concentration of Cl ions was determined using the argentometry method.

Determination of Optimum pH for Adsorbing Cl Ions

5 pieces of 100 mL beaker were prepared containing 50 mL of 50 ppm Cl ion solution each, and the pH was adjusted with pH variations of 6; 7; 7, 5, 8, and 9 with the addition of NaOH or HCl. Then, each optimum mass of silica was inserted into the beaker. The beakers were covered with aluminum foil and stirred using a magnetic stirrer for 30 minutes. After that, it was filtered, and the filtrate was stored in a chemical glass. Then, the concentration of Cl ions was determined using the argentometry method.

Determination of Optimum Contact Time for Adsorbing Cl Ions

4 pieces of 100 mL beakers were prepared containing 50 mL of 50 ppm Cl ion solution each and the pH was adjusted to the optimum pH with the addition of NaOH or HCl. Then, each optimum mass of silica was inserted into the beaker. The beaker were covered with aluminum foil, then stirred using a magnetic stirrer for 15, 30, 45 and 60 minutes. After that, it was filtered and the filtrate was stored in a chemical glass and then the concentration of Cl ions was determined using the argentometry method.

Application Stages of NaOH Activated Silica in Seawater

Prepare 1 piece of 100 mL beaker containing 50 mL of seawater which has been set with the optimum mass, optimum pH, and optimum contact time. Then, the concentration of Cl ions from seawater was determined using the argentometry method.

Data Analysis

Determination of chloride (Cl) levels (Badan Standardisasi Nasional, 2009):

$$\text{Cl levels mg/L} = \frac{(A-B) \times N \times 35450}{V} \times f \quad (2)$$

Meanwhile, to calculate the percentage of Cl ions adsorbed and adsorption capacity, calculations are carried out with the following equation:

$$\% \text{ Adsorbed} = \frac{C_0 - C_e}{C_0} \times 100 \% \quad (3)$$

$$\text{Adsorption capacity (Q)} = \frac{C_0 - C_e}{m} \times V \quad (4)$$

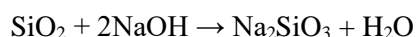
RESULTS AND DISCUSSION

Separation of Silica from Rice Husk Ash Using 12% NaOH

The separation of silica from rice husk ash using 12% NaOH solvent was carried out with samples from the preparation of rice husk ash that had passed the sieve of 140 mesh. This condition is based on the research from Arsa & Achmad (2020), which states that the extracted material should be uniformly sized to facilitate contact between the material and the solvent so that the extraction can occur properly.

NaOH solvent is used to obtain silica from rice husk ash, which is based on the high solubility of silica, which is relatively high in alkaline solvents, especially strong bases such as alkaline hydroxy. This is supported by Kalapathy et al. (2000), who stated that the solubility of silica from rice husk ash is very low at a pH less than 10 and will increase at a pH more than 10. Therefore, the separation of silica from rice husk ash is carried out using an alkaline solvent, namely NaOH 12%.

The use of a 12% NaOH concentration is based on the research of Harimu et al., (2019) which stated that after a variation of NaOH concentration to precipitate silica from rice husk ash, the highest silica content was obtained at a 12% NaOH concentration, which was 36.7%. The reaction that occurs between silica from rice husk ash and NaOH to form sodium silica is as follows:



The results of silica separation from rice husk ash using NaOH are presented in Table 1. Silica in the form of white coarse powder was obtained as much as

8.5514 grams from 40 grams of rice husk ash, so a silica yield of 21.3785% was obtained. According to (Lede et al., 2021), the difference in the percentage of silica produced can be caused by the influence of the type of soil where rice plants grow.

Table 1. Silica yield

No.	Stages	Initial mass (grams)	Final mass (grams)	Silica Yield (%)
1	Separation of Silica from Rice Husk Ash Using 12% NaOH	40	8.5514	21.38
2	Activation of Rice Husk Ash Silica with NaOH Solvent 0.2 M	6	3.1853	53.09

Activation of Rice Husk Ash Silica with NaOH Solvent 0.2 M

Chemical activation is carried out by soaking silica of rice husk ash in a 0.2 M NaOH solution for 24 hours. This chemical activation aims to clean the surface of the pores, rearrange the position of interchangeable atoms, and be able to dissolve the impurities that cover the pores of the silica surface so that the surface area of silica increases (Atikah, 2017). The choice of NaOH as an activator is because NaOH can reduce other materials and has other advantages such as being stable at room temperature, not too dangerous, and low in price (Mastiani et al., 2018). In addition, in this activation process, OH ions from the NaOH solution can add an active OH⁻ group to the silica surface so that silica can be used as an adsorbent and an ion exchange medium.

The silica results obtained after activation with NaOH 0.2 M are presented in Table 1. Based on the research results, the activated silica is obtained of 3.1853 grams from 6 grams of silica, so the silica yield is 53.088%. The reduction in silica obtained is suspected to be due to impurities that cover the pores of the surface of soluble silica in NaOH.

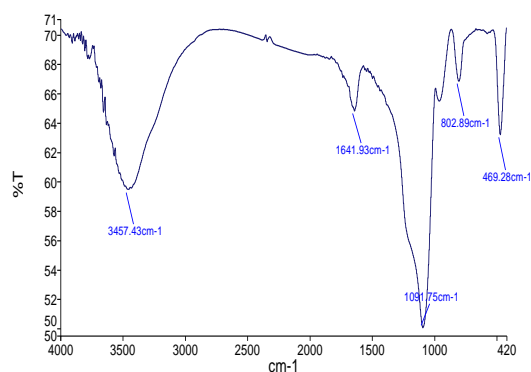
Characterization of Silica Using FTIR

FTIR spectroscopic analysis was conducted to detect, identify, and analyze the presence of silanol and siloxane functional groups from SiO₂ in rice husk ash. The silanol and siloxane groups are the active sides on the silica gel surface that can be used for adsorption. The analysis results of the silica function group of rice

husk ash using FTIR are shown through the spectrum in Figure 1.

The silica spectrum in Figure 1 shows the presence of an absorption peak with a wave number of 3457.43 cm^{-1} , which shows the -OH tensile vibration of the Si-OH functional group. Absorption at wave number 1641.93 cm^{-1} indicates bending vibrations from the Si-OH functional group. The strong absorption band at wave number 1091.75 cm^{-1} is the symmetrical linear vibration of Si-O from the siloxane (Si-O-Si). Wave numbers of 802.89 cm^{-1} and 469.28 cm^{-1} show the presence of tensile-bound vibrations and Si-O bending vibrations of the Si-O-Si functional groups. The absorption bands on the silica spectrum show that the functional groups made from rice husk ash are silanol (Si-OH) and siloxane (Si-O-Si) groups.

Figure 1. FTIR spectrum of silica



Determination of Optimum Mass for Adsorbing Cl Ions

The optimum mass determines the best adsorbent mass for adsorbing Cl ions. The effect of NaOH-activated silica mass on the percentage of Cl ions adsorbed and adsorption capacity can be seen in Table 2.

Table 2. Effect of mass on % adsorbed and adsorption capacity

Silica mass (g)	C_0 (ppm)	C_e (ppm)	% Adsorbed	Adsorption capacity (mg/g)
0.01	50	40.16	19.68	9.84
0.025	50	35.45	29.08	5.82
0.05	50	36.62	26.76	2.66
0.1	50	38.99	22.01	1.1

Based on Table 2, it can be seen that the adsorbent mass can influence the adsorption process. Table 2 shows an increase in the adsorbed percentage along with the rise to a mass of 0.025 grams and then a

decrease in the adsorbed percentage to a mass of 0.1 grams. Based on these data, it can be seen that the optimal adsorbent mass for adsorbing Cl ions is 0.025 grams. This is based on the fact that the most significant percentage of adsorbed matter occurred at a mass of 0.025 grams, and it can be seen that the largest concentration of Cl ions absorbed by silica happened at a mass of 0.025 grams. Therefore, the best adsorbent mass for adsorbing Cl ions is 0.025 grams.

The decrease in the adsorbed percentage after a mass of 0.025 grams is thought to be because the Cl ions have been completely adsorbed at a certain adsorbent mass. So that, the increase in the adsorbent mass does not affect the number of adsorbed ions. Even the adsorption ability of Cl ions tends to decrease with increasing adsorbent mass. In addition, it can also be caused by adding adsorbents in the solution, which can increase the active group of silanol in the adsorbent so that the Cl ion enlarges the formation of hydrogen bonds in the solution. This decreases the number of active silanol groups bound to Cl ions, which reduces the number of bound ions. In addition, according to (Harimu et al., 2019) it is suspected that with the increase in the number of hydrogen bonds formed, the pore structure and active groups in the adsorbent will change, as a result of which the number of pores that trap Cl ions will decrease.

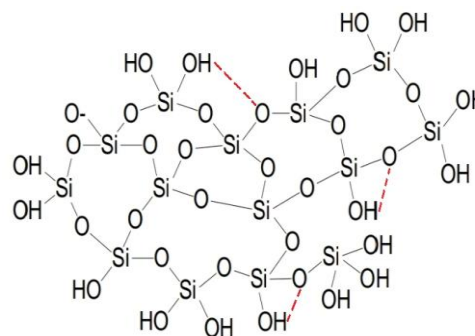


Figure 2. Formation of hydrogen bonds

The largest Cl ion adsorption capacity occurred at a mass of 0.01 g of silica, 9.83 mg/g. Meanwhile, the smallest Cl ion adsorption capacity occurs at a mass of 0.1 g of silica, which is 1.1 mg/g. This data shows that the more silica mass is used, the lower the adsorption capacity. According to (Harimu et al., 2019), this is because the increase in the mass of silica is proportional to the number of particles and the surface area of silica, causing an increase in the active side of adsorption, in this case, SiOH, which allows the

formation of hydrogen bonds that affect the number of Cl ions that are bound.

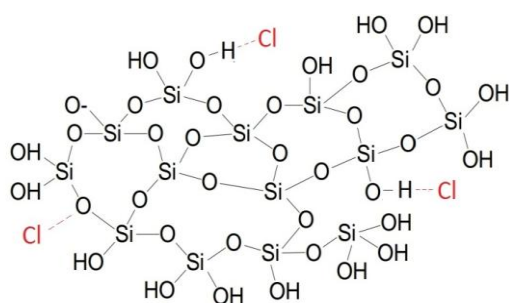


Figure 3. Reaction mechanism of adsorbates and adsorbents

Determination of Optimum pH for Adsorbing of Cl Ions

The optimal pH of Cl ion adsorption was determined to determine the best pH for adsorbing Cl ions using several pH variations, namely 6, 7, 7.5, 8, and 9. In determining optimal pH, the optimum mass of NaOH-activated silica obtained is 0.025 grams. The effect of pH on the percentage of Cl ions adsorbed and adsorption capacity can be seen in Table 3.

Table 3. Effect of pH on % adsorbed and adsorption capacity

pH	C ₀ (ppm)	C _e (ppm)	% Adsorbed	Adsorption capacity (mg/g)
6	50	29.54	40.92	8.18
7	50	27.15	45.7	9.14
7.5	50	28.36	43.28	8.66
8	50	31.9	36.2	7.24
9	50	35.45	29.08	5.82

Table 3 shows an increase in the adsorption percentage along with the increase to pH 7 and then a decrease in the adsorbed percentage to pH 9. Based on this data, it can be seen that the optimum pH to reduce the level of Cl ions is pH 7. The high adsorption ability at pH 7 is due to the properties of the active group on the silica surface namely, the active group of silanol (SiOH) tends to be in its ionic state so that the process of forming bonds between Cl ions and silanol groups is easier to occur. At pH 6, the silanol group undergoes protonation to form SiOH₂⁺ so that the formation of bonds on the surface between the silanol group and the Cl ion is reduced. On the other hand, at pH 8 and pH 9, the absorption of Cl ions decreases due to the abundance of OH⁻ ions in the solution. There is competition between OH⁻ ions and Cl ions to bind to

the active silica group, so the absorption is not optimal. This statement is supported by (Adawiah et al., 2020), which states that the amount of OH⁻ increases along with the increase in pH so that there is competition between the adsorbed substance and OH in the adsorption process on the silica surface, which causes the adsorption capacity to decrease.

Determination of Optimum Contact Time for Adsorbing Cl Ions

The optimal contact time of Cl ion adsorption is determined to determine the best contact time for adsorbing Cl ions. The effect of contact time on the adsorbed percentage of Cl ions and adsorption capacity can be seen in Table 4.

Table 4. Effect of contact time on % adsorbed and adsorption capacity

Time (minutes)	C ₀ (ppm)	C _e (ppm)	% Adsorbed	Adsorption capacity (mg/g)
15	50	38.99	22.02	4.4
30	50	27.15	45.7	9.14
45	50	35.45	29.08	5.82
60	50	37.79	24.42	4.88

The variation contact time of adsorption is presented in Table 4. Contact time is decisive in the adsorption process, where contact time is the length of time needed to stir between NaOH-activated silica as an adsorbent and Cl solution as an adsorbate. In addition, contact time is required to determine the time it takes to achieve adsorption equilibrium. Table 4 shows an increase in the percentage of adsorption along with the rise up to the 30-minute contact time and then a decrease in the percentage of adsorption up to the 60-minute contact time. The contact time that produced the most adsorbed occurred at 30 minutes, which was 45.7%, with an adsorption capacity of 9.14 mg/g. Based on this data, it can be seen that the optimum contact time for adsorbing Cl ions is 30 minutes.

The increase in the percentage of Cl ion adsorption at a contact time of 15 minutes to 30 minutes is due to the existence of empty spaces in the silica that have not been filled because the active group in the adsorbent has not interacted optimally or there has not been an equilibrium. After a contact time of 30 minutes, there was a significant decrease in adsorption percentage at 45 and 60 minutes. Based on this, it can be concluded that adsorption has reached equilibrium at a contact time of 30 minutes. According to (Harimu et al., 2020), this is suspected to be due to a longer contact time after an equilibrium occurs, making the adsorbate that has been adsorbed before, in this case,

the Cl ions are rereleased because the mechanical effect of stirring with a relatively longer time makes the bond that has occurred between the adsorbate and the active group break again. This is also supported by the research of (Yustinah et al., 2020), the longer the adsorption process, the greater the adsorbate that can be adsorbed by the adsorbent, and at some point, it will reach equilibrium. After equilibrium occurs, the increase in adsorption process time will not increase adsorption.

Application Stages of NaOH Activated Silica in Seawater

The application of using NaOH activated silica to reduce the level of Cl ions in seawater was carried out under optimal conditions, including the optimum mass, optimum pH, and the optimum contact time that had been obtained previously, namely a mass of 0.025 grams, pH 7 and a contact time of 30 minutes. This is because the adsorption of Cl ions in seawater will be maximum if carried out under optimal conditions.

Based on the research results, the reduction of Cl ion levels in seawater using NaOH activated silica was obtained by 16.925 ppm with an adsorption percentage of 33.85% and an adsorption capacity of 6.77 mg/g. From these data, it can be seen that the percentage of adsorption using seawater is lower than using pure Cl solution. This is suspected of competition with cations and anions in seawater other than Cl ions, including Na, Mg, Ca, SO₄, K, and Br. These cations and anions may affect the adsorption results of Cl ions by NaOH-activated rice husk ash silica.

CONCLUSION

NaOH-activated rice husk ash silica can adsorb Cl ions. The optimum mass of NaOH-activated rice husk ash silica to reduce the Cl ion level was 0.025 grams, with an adsorption percentage of 29.08%. The optimal pH of Cl solution for the adsorption process using NaOH-activated rice husk ash silica was pH 7, with an adsorption percentage of 45.7%. The optimum contact time of NaOH-activated rice husk ash silica to reduce Cl ion levels was 30 minutes with an adsorption percentage of 45.7%. The Cl ion content from seawater samples that can be lowered using NaOH-activated rice husk ash silica was 16.925 ppm with an adsorbed 33.85%.

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