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Adsorption Calcium Brackish Water A Source of Clean Water for Coastal Communities with Modified Silica from Rice Husk Ash

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Abstract: *Many Indonesian residents have difficulty obtaining clean water, especially people who live in coastal areas. Those who live there can only consume brackish water, which is considered to have side effects on health due to the excessive calcium content. This research offers a simple method: adsorption using abundant raw materials, namely rice husk ash from agricultural waste from burning rice husks. Using husks directly has less effectiveness, so it needs to go through additional processes such as removing impurities and modifying the structure. This research aims to reduce calcium levels in brackish water using silica modified from rice husk ash. Making adsorbents is done by sieving, heating, filtering, washing to obtain silica, then modifying and optimizing important parameters until its application to brackish water. The difference between pre-modified and modified silica can be seen in the characterization with FTIR at wave numbers from 470 cm*$^{-1}$ *to 430 cm*$^{-1}$*, namely deeper bending vibrations, which are also clarified by the surface condition with more structured pores seen in the SEM results. The modified silica works optimally at a mass of 0.4 grams, pH 7.5, contact time of 40 minutes, and concentration of 50 ppm. It has an adsorption capacity of 3.2 mg/g and follows the Langmuir isotherm pattern.*

Keywords*: Adsorption, Brackish Water, Calcium, Rice Husk Ash, Silica.*

INTRODUCTION

Most of Indonesia's population has difficulty getting clean water, especially people who live in coastal areas. The water resources in this area are generally of poor quality, due to the groundwater being brackish and even salty. Brackish water is water whose salinity is lower than the average salinity of normal sea water (<35 ppm) and higher than 0.5 ppm, which occurs due to mixing of sea water with fresh water either naturally or artificially (Du et al., 2020). Brackish water that has not undergone processing cannot be used as clean water because it can cause various diseases, including tooth decay, diarrhea, liver damage, and others. This is caused by very high levels of salt, hardness, and metal solids such as calcium and magnesium (Chakraborty et al., 2019).

Hard water is not dangerous water, but high levels of calcium can cause the water to become cloudy, making it unfit for consumption. According to the Regulation of the Minister of Health of the Republic of Indonesia Number 492/MENKES/PER/IV/2010 concerning drinking water quality requirements, which includes several water quality parameters, the maximum hardness limit for water is 500 mg/L (Ca^{2+} and Mg^{2+}) and the minimum allowable level is 75 mg /L. (Said & Biyanti, 2023). Hard water that has exceeded the maximum limit can cause several health problems such as blockage of heart blood vessels (cardiovascular disease) and kidney stones (urolithiasis) (Rahaman et al., 2020). Hard water is also unsuitable for washing because ions such as Ca^{2+} and Mg^{2+} will bind to the remaining carboxylic acid in the soap and form a precipitate so the soap does not foam (Emami et al., 2022). Therefore, to overcome this problem, a method that functions as a medium to reduce hardness levels is needed.

Methods that can be applied to reduce hardness are the ion exchange method (Hermawan et al., 2023), adsorption (Yang et al., 2024), filtration (Srivastava et al., 2022), membrane separation (Dudziak & Kudlek, 2019) and precipitation (coagulation) (Wang et al., 2022). One adsorbent that has good prospects is rice husks because they are resistant to weathering and have pretty high ash and carbon content (Singh et al., 2017). It can also reduce agricultural waste, impacting environmental efficiency (Kaharuddin, et al., 2024).

The chemical composition of rice husks, which will be used as an adsorbent, is fiber (31.37-49.92%), cellulose (34.34-43.80%), and lignin (21.40-46.97%). If the rice husk is ash, the main content that acts as an adsorbent is silica in the form of SiO_2 with a silica content of 86.90-97.30% (Daffalla et al., 2020). Silica from rice husk ash can be an adsorbent because it contains silanol groups. (-Si-OH) so it is more hydrous, has a pore structure and characteristics that do not change (remains amorphous) until combustion at a temperature of 600°C-750°C, and has a relatively high specific surface area so that it can be used as an adsorbent (Santana Costa & Paranhos, 2018). The adsorption ability of silica from rice husk ash as an adsorbent in adsorbing Ca^{2+} ions is influenced by several conditions, including the pH of the solution, the weight of the adsorbent, and the contact time (Erdoo Kukwa et al., 2020). The Ca^{2+} ion adsorption process will be maximal under optimum conditions. Adsorption of Ca^{2+} metal ions in hard water using tea dregs as an adsorbent. It was found that optimum adsorption occurred at pH 8 with the highest adsorption efficiency of 31.56% (Rezgui et al., 2022).

The use of rice husk ash silica as an adsorbent without modification has a weakness, namely that the active site is only a silanol group (Si-OH), which has low acidity and has oxygen as a donor atom, which is slightly weak, so it is less capable of being used as an adsorbent directly for adsorption. Specific metal ions (Yefremova et al., 2023). Therefore, to increase the silica adsorption capacity of rice husk ash, it can be done through modification, for example by carrying out activation, which aims to enlarge the silica pores by breaking hydrocarbon bonds or oxidizing surface molecules (Yefremova et al., 2023).

Modifications carried out by activation can be done in two ways: physical and chemical. Chemicals, especially alkaline compounds such as NaOH, are preferred because they are hygroscopic and alkaline when reacting with water (Kiani & Baltrusaitis, 2021). This fundamental nature indicates that the NaOH solution is rich in OH ions, which can add silanol (-Si-OH) active groups to the adsorbent to increase its adsorption capacity (Kiani & Baltrusaitis, 2021). Apart from that, NaOH will also react with silica (SiO_2) to produce Na_2SiO_3 and H_2O, where the Na^+ ions on the surface of the adsorbent can be used to exchange with Ca^{2+} ions in brackish water, so that the silica from husk ash is modified (Kiani & Baltrusaitis, 2021). Based on this description, there is great potential to reduce calcium levels in brackish water as a source of clean water for coastal communities with modified silica adsorbents from rice husk ash.

RESEARCH METHODS

This research method starts from the adsorbent preparation stage by sieving the rice husk ash. The fine ash is added with NaOH by heating and filtering, then neutralized with H_2SO_4 and filtered again as a washing process to produce purer silica (Harimu et al., 2019).

The resulting silica was characterized to see the morphology before application, and the other part was modified (activated) with NaOH and characterized to see the comparison before and after modification. Once the adsorbent is ready, it continues with the parameter optimization stage (mass, pH, contact time, and concentration). This is done to obtain optimum data for each parameter before testing it on brackish water samples. Followed by the Application stage to determine the ability of the modified material to reduce calcium levels in brackish water under optimum conditions, as well as the data processing stage and drawing conclusions.

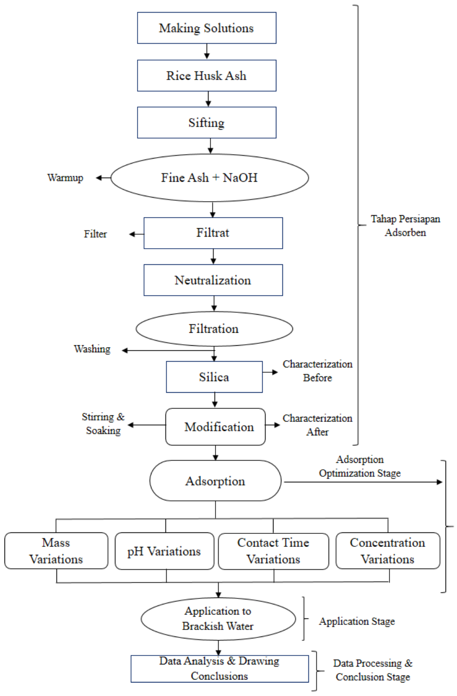

Figure 1. Research Flow Diagram

Materials and Tools

The materials used in this study were rice husk ash, distilled water, brackish water, 0.2 M Natrium hydroxide (NaOH) solution, 12% sodium hydroxide (NaOH) solution, 1 N sodium hydroxide (NaOH) solution, 1 M sulfuric acid (H_2SO_4) solution, 0.01 M Na2-EDTA solution, Eriochrome Black T (EBT) indicator, murexide indicator, pH 10 buffer solution and filter paper. The equipment used in this study were analytical scales, spatulas, stirring rods, 500 mL beakers, magnetic stirrers, 50 mL burettes, 250 mL Erlenmeyer flasks, 100 mL, 250 mL and 500 mL measuring flasks, fillers, 10 mL volumetric pipettes, 10 mL and 25 mL measuring pipettes, glass funnels, hotplates, spray bottles, brown bottles, ovens, pH meters, porcelain cups, and stopwatches/timers.

RESULTS AND DISCUSSION

Silica extraction from rice husk ash

The extraction of silica from rice husk ash using sodium hydroxide (NaOH) solution efficiently obtains high-quality amorphous silica from abundant agricultural waste. Rice husks, once burned, produce ash that is rich in silica, about 85-95%, along with small amounts of other compounds such as carbon and minerals (Nzereogu et al., 2023). The process begins by burning the rice husks at high temperatures, usually between 600 and 800°C, to remove organic material and leave behind an ash that consists mostly of silica. It is important to note that the temperature and duration of firing affect the properties of the resulting silica, including the degree of crystallinity and particle size. The use of a NaOH solution with a concentration of 12% (Harimu et al., 2019), who extracted silica from rice husk ash through precipitation and obtained the highest silica content at a NaOH concentration of 12%, namely 36.7%. The reaction of binding NaOH with SiO_2, neutralization with acid and washing with distilled water.

$$SiO_{2(s)} + 2\ NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \qquad \text{(Eq. 1)}$$

$$Na_2SiO_{3(aq)} + H_2SO_{4(aq)} \rightarrow H_2SiO_{3(aq)} + Na_2SO_{4(aq)} \qquad \text{(Eq. 2)}$$

$$H_2SiO_{3(aq)} \rightarrow SiO_{2(s)} + H_2O_{(l)} \qquad \text{(Eq. 3)}$$

Characterization

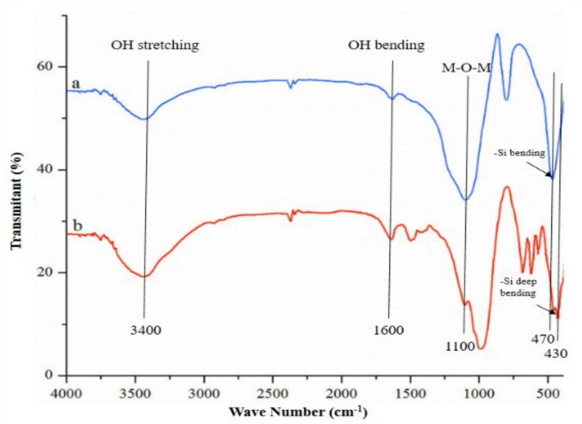

Figure 2. FTIR Spectrum (a) Extracted silica (b) Modified Silica

Silica in rice husk ash either before or after modification, namely with the appearance of absorption bands in the area of 3430 cm^{-1} (–OH group stretching), 1600 cm^{-1} (-OH group bending vibration). This shows that there are hydrogen bonds in the H_2O molecules in rice husk ash. 1100 cm^{-1} M-O-M vibration (M=Si or Al), as well as bending vibration at 470 cm^{-1} absorption (Figure 2a). Modified silica shows pore opening, namely in the 430 cm^{-1} (Figure 2b) absorption band, namely deeper bending vibrations (deep bending) caused by weakening of the Si-O- bonds, causing impurities in the pores to be released and the pores to be cleaner (Qomariyah & Hidayah, 2021). The opening of these pores allows for more effective adsorption capabilities in reducing brackish water metal ions (Ca^{2+}).

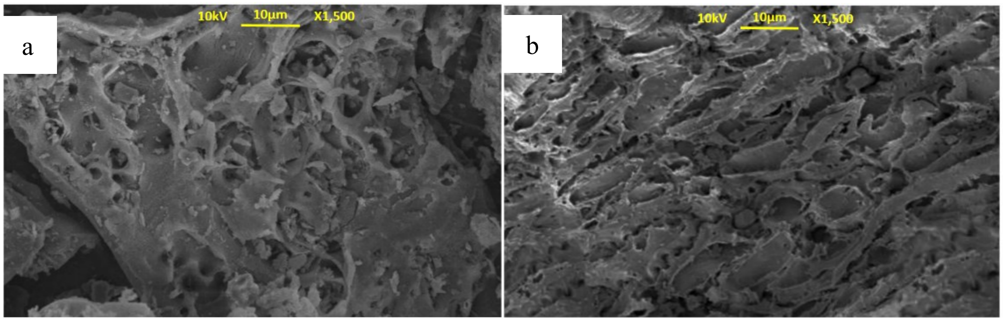

Figure 3. SEM (a) Extracted silica (b) Modified Silica

Silica from rice husk ash that has not been activated shows a less regular surface with an uneven pore size distribution, tending to have less regular pores (micropores to mesopores). Due to the natural formation process (burning rice husks), its porosity is limited and is not fully optimized for applications that require a large surface area or uniform pores. The surface is rough with various small gaps formed from ash residue. The existing pores are not very structured, and there are visible amorphous particles of silica (Figure 3a). The modified silica showed significant changes in its pore structure and surface morphology. This chemical activation process causes changes in pore size and distribution. Silica exhibits more open and well-defined pores. The pores will be larger and more uniform, increasing the pore size compared to unactivated silica (Figure 3b). This shows that the modification was successful and opened up more space in the silica structure, increasing its porosity (Halim et al., 2018).

Silica modification from rice husk ash

The modification is carried out by activating silica from rice husk ash. Activation of silica with natrium hydroxide (NaOH) is a chemical process used to improve silica's surface properties and reactivity. This activation aims to modify the structure or chemical composition of the silica surface, thereby making it more effective in applying calcium ion adsorption in brackish water. NaOH can decompose into Natrium (Na^+) and hydrogen chloride (OH^-). The OH^- formed can react with the silica surface, causing the formation of new active sites, such as silanol groups (-Si-OH) or modifications to the silica structure. At the same time, Na^+ ions can be used to compare with Ca^{2+} ions in brackish water so that silica can be used as an adsorbent as well as an ion exchange medium, thereby allowing the surface properties of the silica to change, including increasing the specific surface area or forming new pores (Figure 4).

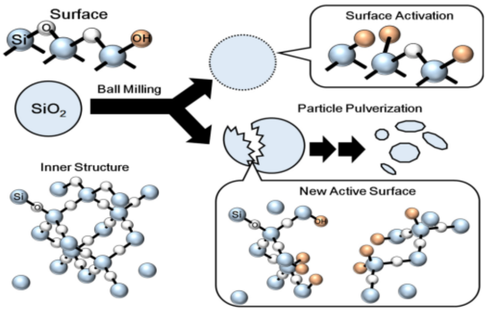

Figure 4. schematic illustration of surface activation (Nakashima et al., 2019)

The texture of the rice husk ash silica obtained after the activation process changes from the initial form of silica obtained from the extraction process, namely that the silica grains become finer and have a clean white color. This is thought to be caused by removing impurity compounds found on the silica surface, causing the silica to become cleaner and more porous. This is because activation affects the crystallinity and pore size of the amorphous material due to the release of metal oxides and other compounds on the surface of the rice husk ash silica. After activation, metal oxides previously attached to the amorphous solid of rice husk ash will be released from the amorphous solid and leave a cavity where they were previously bound (Figure 5). The presence of several cavities causes amorphous solids to have a larger surface area and become more unstable, thus becoming more reactive (Islam et al., 2024).

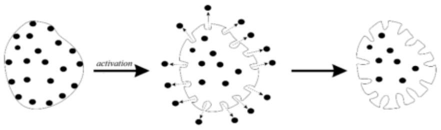

Figure 5. assumed release metal oxides from amorphous solids of rice husk ash

Optimation Stage

Carried out to determine the optimum conditions for each test parameter before conducting it on brackish water samples. The percentage of adsorbed Ca^{2+} ions continues to increase as the mass of the adsorbent increases, where the lowest percent of Ca adsorbed occurs at a mass of 0.05 grams at 35.2% and reaches optimum conditions at a mass of 0.4 grams with a percent of Ca adsorbed at 56.8%. Again, there was a decrease in mass of 0.5 grams (Figure 3a). This is because all the active groups and adsorbent pores have attracted and bound Ca^{2+} ions optimally at a mass of 0.4 grams. Apart from that, at a mass of 0.1 gram of ion, the adsorption capacity has been reached, namely 3.2 mg/g, so that increasing the mass of the adsorbent does not affect the number of metal ions adsorbed; in fact, it decreases as the mass of the adsorbent increases. This is supported by research (Harimu et al., 2019) which used rice husk ash to adsorb Cu^{2+} and Pb^{2+} ions.

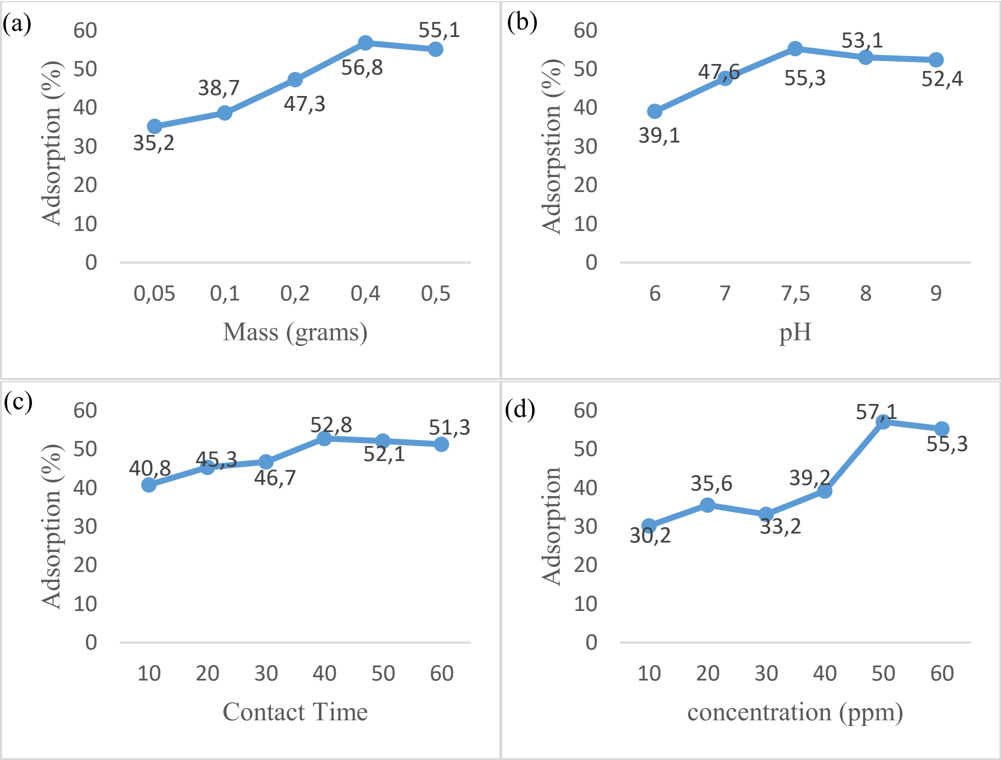

Figure 3. optimum adsorption for each parameter (a) mass, (b) pH (c) contact Time (d) concetration

pH is an important parameter in the adsorption process of metal ions in solution. Determining the optimum pH aims to determine the pH of the solution required by NaOH-modified rice husk ash silica to adsorb Ca2+ ions optimally. Determination of the optimum pH was carried out with variations of 6, 7, 7.5, 8, and 9 using the optimum mass that had been obtained previously. The optimal pH of the Ca2+ ion solution that the rice husk ash silica adsorbent can adsorb occurs at pH 7.5 and decreases at pH 8 and 9 (Figure 3b). The highest adsorption percentage occurred at pH 7.5 at 55.3% with an adsorption capacity of 4.2 mg/g. This is because the active group on the silica surface, namely the silanol group (SiOH), undergoes ionization to become SiO^-, so the bond formation process between the Ca^{2+} metal ion and the silanol group occurs more easily. As a result, more and more Ca^{2+} ions are bound to the surface of the adsorbent.

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As a result, the process of absorbing Ca^{2+} ions by the adsorbent tends to decrease as the pH of the solution increases. At pH 6 and 7, the percentage of silica adsorption on Ca^{2+}

ions shows that the absorption efficiency at this pH is still low. This is because at low pH the surface of the adsorbent is surrounded by large amounts of H^+ ions, so it undergoes protonation to form $SiOH_2^+$ which causes electrostatic rejection of Ca^{2+}. Additionally, abundant H^+ ions also cause competition between Ca^{2+} and H^+ ions to form bonds with silanol groups. The formation of bonds on the surface between silanol groups and metal ions is reduced because there is competition between Ca^{2+} and H^+ ions to form bonds with silanol groups (Jabraoui et al., 2022).

Determination of the optimum contact time is carried out using the conditions of optimum mass and optimum pH that have been obtained previously. The increase in the adsorption percentage of modified rice husk ash silica towards Ca2+ ions from 10 to 30 minutes has an adsorption percentage of still less. It is suspected that this is because there are still empty spaces on the silica adsorbent that are not yet filled. After all, the active groups on the adsorbent are not all bound to Ca^{2+} ions (Hastuti et al., 2019) At 40 minutes, all active groups and silica pores were filled with Ca^{2+} ions, so optimal absorption of Ca^{2+} ions occurred at that time (Figure 3c). Also, long contact time with regular stirring will cause the adsorbent pores to work perfectly so that more Ca^{2+} ions are adsorbed because more collisions occur between Ca^{2+} ions and the adsorbent with a capacity reaching 3.1 mg/g. At 50 and 60 minutes, there was a decrease in adsorption, but it was not significant. This is because the interaction between Ca^{2+} ions and silica has passed saturation, resulting in rapid and continuous collisions between adsorbent particles and Ca^{2+} ions, so there is a possibility that the adsorbent will rerelease the adsorbate (Nizam et al., 2021). At long contact times, the adsorption capacity and adsorption percentage decrease due to the equilibrium conditions in the adsorption-desorption process, so that the amount of weakly adsorbed Ca^{2+} ions can be re-desorbed due to the stirring/shaking effect (Harimu et al., 2022).

Concentration variations were carried out using the conditions of optimum mass, pH, and contact time obtained previously. The concentration of the solution greatly influences the adsorption of Ca^{2+} ions by the silica adsorbent of rice husk ash. The optimum concentration of Ca^{2+} ion solution that can be optimally adsorbed by rice husk ash silica occurs at 50 ppm. Increased adsorption from a concentration of 10 ppm to a concentration of 50 ppm (Figure 3d). This is because there has not been an equilibrium between the surface of the active site of the adsorbent and the number of Ca^{2+} ions in the solution (Astari & Utami, 2018). At a concentration of 60 ppm, there was a significant decrease in adsorption. It is thought that this was caused by the surface of the active site of the adsorbent being saturated with adsorbate, so that increasing the concentration would not increase the amount of adsorbed adsorbate. It is also suspected that increasing the concentration of the Ca^{2+} solution also increases the Ca^{2+} ions in the solution, thereby causing higher movement between the particles. The movement between particles obstructs the adsorption process of Ca^{2+} ions on the silica surface, thereby reducing the adsorption capacity (Sharma & Ahammed, 2023).

Application to Brackish Water

The application stage of using NaOH activated rice husk ash silica to reduce calcium ions in brackish water is carried out under the conditions of mass, pH, contact time and optimum concentration of the calcium ion solution that has been obtained previously.

The decrease in the concentration of calcium ion (Ca^{2+}) at this application stage is relatively small compared to the decrease in the concentration of pure calcium ion (Ca^{2+}), this is thought to be influenced by several factors such as the presence of other metals in brackish water, both in the form of cations and anions. It is also adsorbed into the pores of the adsorbent, thereby blocking the adsorption process of Ca^{2+} ions in brackish water. (Islamiyati et al., 2022)which states that brackish water contains seven main ions which are also the main causes of salinity in brackish water, namely: sodium (Na^+), potassium (K^+), calcium (Ca^{2+}), magnesium (Mg^{2+}) , chloride (Cl^-), sulfate (SO_4^{2-}), and bicarbonate (HCO_3^-). The presence of these ions also allows them to be adsorbed on the silica surface, causing the adsorption of calcium ions to be reduced.

Table 1. Results of reducing Ca^{2+} ion levels with modified silica

Mass (grams)	pH	Contact time (minute)	C_0 (ppm)	C_t (ppm)	Adsorptoin (%)	q (mg/g)
0,4	7.5	40	50	38,4	29,41	3,2

Research on reducing levels of calcium ions (Ca^{2+}) using types of adsorbent other than silica adsorbent, rice husk ash, has been carried out by several researchers, including (Sobirin et al., 2015) who adsorbed calcium ions in water using magnetite from iron sand as an adsorbent and obtained adsorption capacity values. The largest calcium ion was 0.397 mg/g at an optimum contact time of 75 minutes. (Nurhidayanti et al., 2021) used acid and base activated water hyacinth to reduce the hardness of well water. The effectiveness value for reducing water hardness using hydrochloric acid (HCl) activated water hyacinth was 11.15%, while the effectiveness value for reducing water hardness using sodium activated water hyacinth was 11.15%. hydroxide (NaOH) of 6.16%. Apart from that, (Maulana et al., 2017) also carried out adsorption of Ca^{2+} metal ions in hard water using tea dregs as an adsorbent and obtained that optimum adsorption of Ca^{2+} metal ions occurred at optimum pH conditions, namely pH 8 with the highest adsorption efficiency of 31.56%. Based on this, it can be concluded that rice husk ash silica modified with NaOH can be used to reduce calcium ions in brackish water.

Isoterm Adsorption

In the process of adsorption of calcium ions from brackish water by modified silica, the mechanism involves interactions between positively charged calcium ions (Ca^{2+}) and negatively charged active sites on the silica surface. Silica modification allows its surface to have a higher affinity for Ca^{2+} ions through electrostatic interactions or coordination bonds. When calcium ions are bound to the surface of the adsorbent, there is a reduction in the concentration of calcium ions in the solution. Calcium ions in modified silica reach saturation and occur on a homogeneous surface. Therefore, it tends to follow the Langmuir isotherm.

CONCLUSIONS

Reducing calcium ion levels in brackish water as a source of clean water for coastal communities has been successfully carried out. Silica can be extracted easily from rice husk ash, modified, and works optimally at a mass of 0.4 grams, pH 7.5, contact time of 40 minutes, and concentration of 50 ppm in this study. It has an adsorption capacity of 3.2 mg/g and follows the Langmuir isotherm pattern.

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