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Research Article

Effect of Adding Hydrochloric Acid to Alumina Oxide (LAO) Clay on Adsorption Capacity of Pb and Cd Metal Ions

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Abstract

Research has been conducted on the application of Ouw Natural Clay (LAO) as an adsorbent for Pb metal ions and Cd metal ions, following the addition of hydrochloric acid (HCl). The concentrations of HCl used were 2, 3, and 4 M. Characterization was performed to evaluate the differences in clay characteristics before and after the addition of HCl. The Surface Area Analyzer (SAA) was utilized for characterization, specifically to determine the specific surface area, pore volume, and pore size. The results of the characterization indicated an increase in specific surface area, from 31.37 m^2/g for pure LAO to 99.68 m^2/g for LAO-HCl 4 M. Furthermore, the addition of hydrochloric acid to the Ouw clay natural resulted in a reduction in the capacity to adsorb Pb metal ions and Cd metal ions.

Keywords: Cd, Pb, metalion, chloride acid, Ouw natural clay

INTRODUCTION

Clay is an abundant natural mineral in Indonesia, yet its utilization has not been optimized. Clay contains a significant amount of inorganic materials, which consist of a collection of minerals. The clay system is composed of two distinct parts: (1) relatively large and insoluble micelles (alumina silicate layers); and (2) a collection of weakly bound cations such as Na^+, K^+, and Ca^{2+} (Wijaya, Pratiwi, Sudiono, & Nurahmi, 2010). Clay is of great interest for research due to its layered structure and exchangeable cations (Wijaya et al., 2010; Xie et al., 2024). The key components in our natural clay (LAO) are montmorillonite, quartz, kaolinite, and illite, with montmorillonite being the dominant component. Montmorillonite, commonly known as bentonite, is a type of clay that exhibits easy expansion, a considerable surface area, and the ability to accommodate a large quantity of cations between its layers, making it widely used in various industries (Istinia, Wijaya, Tahir, & Mudasir, 2018; Sudhakaran, Mahadevan, Fathima S, & Krishnan, 2023).

Clay minerals, which are significant constituents in soil, serve as natural traps for pollutants carried by surface water or within the soil through adsorption or cation exchange capacity (CEC). In Indonesia, approximately 70% of air pollution is attributed to motor vehicle emissions, with motor vehicles contributing almost 100% of Pb and Cd metals, especially in major cities. The Pb and Cd metals released into the air can be absorbed and enter water bodies and the food chain through the climate cycle and diffusion process, thus posing a contamination risk to humans and the surrounding environment. Both metals are toxic and non-biodegradable (Kolo, Batu, Bani, Nana, & Kedang, 2024; Prasetiowati, 2014).

Various methods of waste treatment, including absorption, precipitation, electrodeposition, ion exchange, adsorption, and

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membrane separation, have been implemented. Adsorption has been proven to be a more effective technique for removing heavy metal pollutants compared to other processes (Eren & Afsin, 2008). Adsorption refers to the concentration of adsorbate molecules or ions on the surface layer of the adsorbent, either through physical or chemical bonding. Consequently, the adsorbent must possess unique surface properties specific to the type of adsorbate being targeted. The use of activated carbon or organic and inorganic resins for the adsorption process is a relatively effective technique, but it comes with high costs. Therefore, it is necessary to explore alternative cost-effective adsorbents (Eren & Afsin, 2008; Sagadevan et al., 2024; Singh & Baheti, 2024; Xie et al., 2024).

The capacity of clay as an adsorbent is supported by its physical and chemical characteristics. One method to enhance these characteristics is through acid activation, which results in clay with increased active sites and surface acidity. Bijang, Wahab, Maming, Ahmad, & Taba, (2015) has also investigated the effects of acid activation on clay, demonstrating that it can enhance surface area and the distance between layers. Activation is a treatment that aims to enlarge pores, either by breaking hydrocarbon bonds or oxidizing surface molecules, resulting in changes in physical and chemical properties, such as increased surface area, thereby affecting the adsorption capacity (Cao, Wang, Zhang, Ma, & Wang, 2021; Khalifa et al., 2020).

The natural clay investigated in this study is located in the Saparua District, Central Maluku Regency. This clay has been traditionally used for purposes such as roof tiles and pottery tools like sempe and belanga. Therefore, the utilization of LAO as an adsorbent for the highly dangerous heavy metals, Pb and Cd, provides an alternative solution to minimize environmental pollution, particularly in aquatic environments.

Numerous studies have been conducted on the adsorption capacity of clay for heavy metals. For example, HCl-activated bentonite has been explored as an adsorbent for Cd metal ions (Nugraha and Mahatmanti, 2017), the impact of hydrochloric acid on the structure of Na-Bentonite clay has been investigated (Bendou and Amrani, 2014), and sulfuric acid-activated clay has been utilized as a cyanide biosensor (Bijang, 2015). Based on the aforementioned information, this study specifically examines the effects of adding hydrochloric acid to Ouw natural clay (LAO) on the adsorption capacity of Pb and Cd metal ions.

MATERIALS AND METHODS

Material and instrumentations

Natural Clay Ouw (LAO), distilled water, hydrochloric acid (HCl) (p.a Merck), $AgNO_3$ (p.a Merck), $Pb(NO_3)_2$ (p.a Merck), $CdSO_4$ (p.a Merck), and filter paper. The tools employed comprise of: a set of glassware (Pyrex), an analytical balance (Ohaus), an electric heater (Cimarec2), a magnetic stirrer, a 100 mesh sieve, an Atomic Absorption Spectrophotometer (iCE 3000), a Surface Area Analyzer (SAA) (QuantachromeNova), an oven (Memmert), a vacuum pump (Vacuubrand GMBH+CO KG), a desiccator, and a shaker (GFL3005).

Sample preparation

Our natural clay was thoroughly soaked and washed with distilled water multiple times. It was then filtered using a vacuum pump to obtain a pure clay free from impurities such as sand, gravel, and plant roots. Additionally, the clay underwent a 4-hour drying process in an oven set at 80 °C. Once dried, the clay was ground and sieved using a 100-mesh sieve before being stored in a desiccator.

Activation with hydrochloric acid

In three separate 250 mL beakers, 10 grams of clay were added to each. Then, 100 mL of hydrochloric acid solution with concentrations of 2, 3, and 4 M were added to each beaker while stirring with a magnetic stirrer. The activation process was conducted over a 24-hour period, followed by filtration using a vacuum pump. The residue obtained was washed with distilled water until completely free from acid, confirmed by a negative test result with $AgNO_3$ solution. The clay was subsequently dried in an oven at 80 °C, ground, and sieved using a 100-mesh sieve. Finally, the activated clay was stored in a desiccator.

Characterization of Natural Clay Ouw

The natural clay samples, both before and after the addition of hydrochloric acid, underwent characterization. This process involved using a Surface Area Analyzer (SAA) to determine the specific surface area, pore volume, and pore size.

Determination of optimal concentration for adsorption of Pb metal ions

A total of 0.5 Ouw natural clay (LAO) before and after the addition of hydrochloric acid was put into a 100 mL Erlenmeyer flask and 50 mL of Pb(II) solution with a concentration of 50 ppm was added. then shaken with a shaker at a speed of 200 rpm for 24 hours. The mixture was then separated using filter paper and the filtrate was measured using an AAS instrument. From the AAS data, the final concentration of the sample (C_e) was obtained, then the percentage and adsorption capacity were calculated using the following formula Equation 1 and Equation 2.

$$Q = \frac{Co-Ce}{Co} x \; 100\% \quad (1)$$

$$q = (Co - Ce) \; x \; \frac{V_{metal\,ion}}{m_{LAO}} \quad (2)$$

Where Q represents the adsorption percentage and q represents the adsorption capacity. Co denotes the initial solution concentration, while C_e represents the final solution concentration. (C_o–C_e) indicates the concentration of the adsorbed solution, with V representing the volume.

Determination of the optimal concentration for adsorption of Cd metal ions

A total of 0.5 g of natural clay (LAO) was placed into a 100 mL Erlenmeyer flask before and after the addition of hydrochloric acid. Subsequently, 50 mL of Cd(II) solution with a concentration of 50 ppm was added, and the mixture was shaken at a speed of 200 rpm for 24 hours using a shaker. The resulting mixture was then separated using filter paper, and the filtrate was analyzed with an AAS instrument. Based on the AAS data, the final concentration of the sample (C_e) was determined. The percentage and adsorption capacity were subsequently calculated using the following formula Equation 1 and Equation 2.

RESULTS AND DISCUSSION

Sample preparation

The clay utilized in this study was sourced from Ouw Village, Saparua District, Central Maluku Regency. The study commenced by soaking and washing the clay multiple times with distilled water to separate impurities, including sand, gravel, and plant roots, from the clay. Subsequently, the clay was dried in an oven at a temperature of 80 ºC for a duration of 4 hours to eliminate water molecules present in the interlayer space of the clay. Following this step, the clay sample was finely ground and passed through a 100 mesh sieve to reduce its particle size. This process enhanced the clay sample's surface area, thereby improving the potential for interaction with hydrochloric acid (HCl) within the pores. The resulting clay can be observed in Figure 1.

Figure 1. Natural Clay Ouw (LAO) Preparation Results

Hydrochloric Acid (HCl) activation

The purpose of activation is to open up the interlayer space and eliminate impurities within the clay's internal structure. In this study, chemical activation was performed using HCl concentrations of 2, 3, and 4 M, which were stirred for 24 hours. Clay typically contains various cations from the alkali and alkaline earth groups. This aligns with the findings of Prasetiowati, (2014), who suggested that the addition of HCl facilitates the exchange of cations in the clay interlayer, such as Ca^{2+}, Mg^{2+}, and K^+, with H^+ cations from the HCl. Subsequently, the Al^{3+} ions and other metal ions, such as Fe^{3+}, are dissolved from the clay lattice layer, as depicted in Figure 2.

The replacement of H^+ ions induces the release of Al^{3+}, Mg^{2+}, Fe^{3+} ions, and other impurities within the clay crystal structure lattice, thereby increasing the surface area and absorption properties through the opening of closed pores. Following the addition of HCl, LAO (Ouw Natural Clay) is subsequently filtered and thoroughly washed with distilled water until all chloride ions are completely removed, as confirmed by negative results when tested with $AgNO_3$. The chloride-free LAO is then dried in an oven at 80 ºC for 4 hours to eliminate water molecules present in the interlayer space of the clay, aligning with the findings of Sitanggang, Suarya, Simpen, & Putra, (2017)). Subsequently, the LAO sample is ground and sieved using a 100 mesh sieve to reduce particle size, further enhancing the clay sample's surface area and improving the potential interaction between hydrochloric acid and the pores.

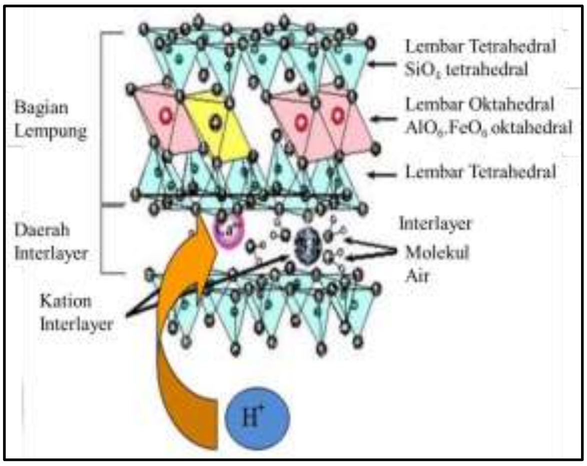

Figure 2. Interaction of technical plate with HCl

The original brown color of LAO prior to the addition of HCl fades, as depicted in Figure 9. This color change can be attributed to the reaction between the clay and HCl, resulting in the dissolution of Fe^{3+} ions within the clay, as observed in the research conducted by Nugraha, Mahatmanti, & Sulistyaningsih, (2017)

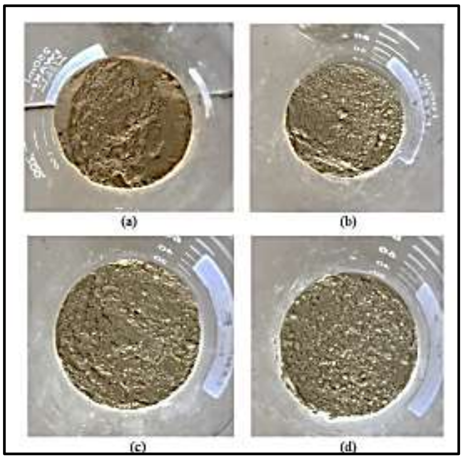

Figure 3. Natural Clay Ouw before and after the addition of HCl. (a) pure LAO, (b) LAO-HCl 2M, (c) LAO-HCl 3M, (d) LAO-HCl 4M.

Characterization of natural clay LAO

The LAO samples were subjected to characterization both before and after the addition of HCl. The characterization process involved the use of BET (Brunauer-Emmett-Teller) analysis, conducted using a Surface Area Analyzer (SAA), to determine specific surface area, total pore volume, and pore size. Surface area represents the extent of occupied space by a single molecule of adsorbate or solute, and is directly related to the sample's surface area (Chao, Deng, & Zhou, 2024; Saadat et al., 2024). Surface area plays a crucial role in adsorption and catalysis processes, as it determines the capacity of the adsorbent or catalyst to absorb adsorbate or reactant. According to Nugrahaningtyas, Widjonarko, Daryani, & Haryanti, (2016), surface area is influenced by factors such as particle/pore size, pore shape, and pore arrangement within the particles. Figures 4, 5, 6, and 7 depict the graphs illustrating the adsorption-desorption isotherm patterns for N_2 gas on pure LAO samples and after the addition of HCl.

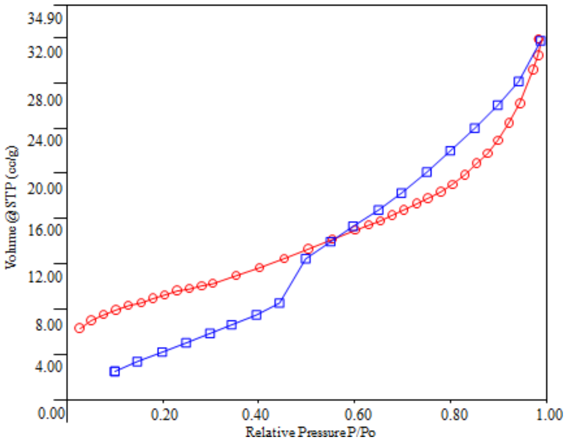

Figure 4. Adsorption-Desorption Isotherm Graph of Pure LAO (Red Line: Adsorption, Blue Line: Desorption)

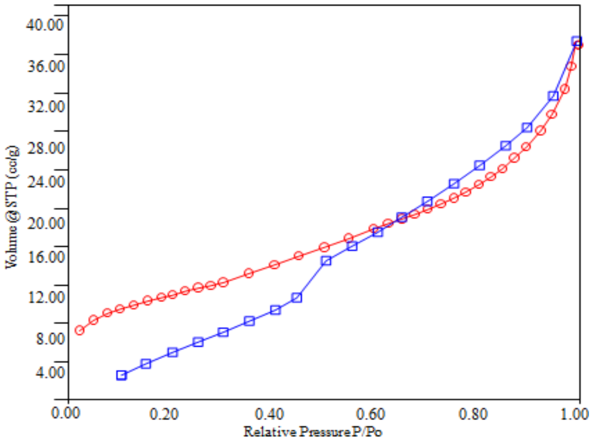

Figure 5. Adsorption-Desorption Isotherm Graph of LAO-HCl 2M (Red Line: Adsorption, Blue Line: Desorption)

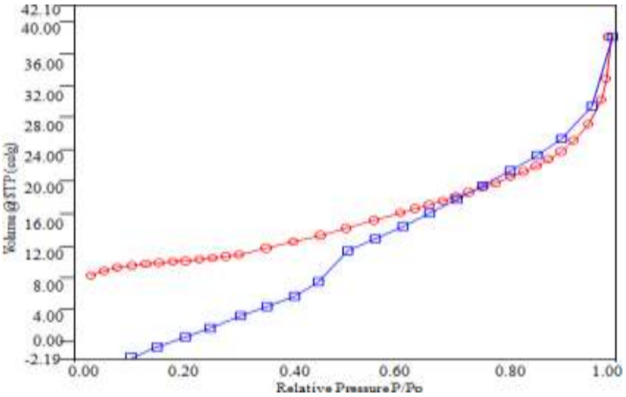

Figure 6. Adsorption-Desorption Isotherm Graph of LAO-HCl 3M (Red Line: Adsorption, Blue Line: Desorption)

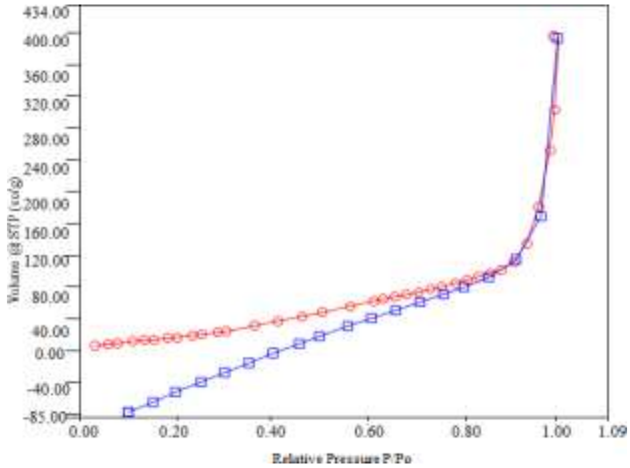

Figure 7. Adsorption-Desorption Isotherm Graph of LAO-HCl 4M (Red Line: Adsorption, Blue Line: Desorption)

The isotherm patterns of pure Ouw natural clay (LAO) before and after the addition of 2 M and 3 M HCl indicate type IV behavior. After the addition of 4 M HCl, LAO exhibits type V behavior. Both types indicate the presence of mesopores. The presence of a knee in the graph signifies the existence of pores in the material, suggesting irregular mesoporous characteristics. Seader, Henley, & Roper, (2010) explains that this type of isotherm pattern suggests the presence of mesoporous characteristics in the adsorbent, with a sequential adsorption process involving both monolayer and multilayer adsorption. Additionally, there are differences between the paths of adsorption and desorption, likely due to capillary condensation.

The pore size classification according to the International Union of Pure and Applied Chemistry (IUPAC) is as follows: micropores (d<20 Å), mesopores (20<d<500 Å), and macropores (d>500 Å). This classification is based on the adsorption of nitrogen gas at its normal boiling point by porous solids with a wide range of pore sizes. The determination of pore size distribution is a crucial factor in adsorption studies and is essential for evaluating porous media and membranes.

Analysis of specific surface area, total pore volume, and pore size measurements reveals that LAO, after the addition of HCl, exhibits a larger specific surface area and pore volume compared to pure LAO. It is worth noting that the pore size of pure LAO falls between that of LAO-HCl 2 M and LAO-HCl 3 M and 4 M, as indicated in Table 1.

Table 1. Data on Specific Surface Area, Total Pore Volume and Pore Size of LAO Before and After Addition of HCl

Samples	Sutface area spesific (m^2/g)	Total pore volume (cc/g)	Pore size (Å)
LAO_{pure}	31.37	$4.907x10^{-2}$	$3.129\ x10^{1}$
LAO-HCl 2M	37.52	$5.776x10^{-2}$	$3.079\ x10^{1}$
LAO-HCl 3M	31.79	$5.917x10^{-2}$	$3.722\ x10^{1}$
LAO-HCl 4M	99.68	$6.096x10^{-1}$	$1.223\ x10^{2}$

The research data presented in Table 1 indicates that the pore size of the sample is greater than 20 Å, thus falling within the mesoporous size range. The observed increase in specific surface area, total pore volume, and pore size in LAO following the addition of HCl can be attributed to the removal of octahedral cations from between the sample layers. This finding aligns with the view expressed by Bendou & Amrani, (2014), which posits a correlation between surface area and the combined content of Al_2O_3, Fe_2O_3, and MgO in the sample, as well as the development of microporosity during the activation process.

The increase in surface area and total pore volume in acid-activated bentonite occurs due to the release of Al^{3+}, Fe^{3+}, Mg^{2+} ions, and other impurities from the structure lattice. The pore volume increases because the impurities have been removed, resulting in a cleaner crystal lattice. Additionally, the radius increases as the impurities that narrow the pores diameter are eliminated during acid interaction (Chao et al., 2024).

Table 1 data shows that the specific surface area only increases in LAO added with 2 and 4

M HCl, while it decreases for LAO-HCl 3 M. This decrease in surface area is likely due to the formation of relatively larger pores. This finding aligns with the research conducted by Anawati, Suseno, & Taslimah, (2012) where samples with smaller pore sizes exhibited larger surface areas.

The observed decrease at a concentration of 3 M HCl can be explained by the passivation process. During this process, the destruction of octahedral sheets releases cations into the solution, while the silica produced by the tetrahedral sheets remains in the solid state, as it cannot be broken down. This finding is consistent with the research conducted by Bendou & Amrani, (2014), which states that free silica resulting from the destruction of tetrahedral sheets undergoes polymerization due to the high acid concentrations, ultimately protecting the undestroyed silicate fraction from further attacks and reducing the surface area. The measurements of total pore volume indicate that water content increases during the addition of HCl concentration due to the dissolution of impurities that occupy the pore system of the material during the acid dissolution process.

Determination of optimum hydrochloric acid concentration for adsorption of Pb metal ions and Cd metal ions

The optimization of HCl concentration aims to determine the best adsorption capacity between pure LAO and LAO after the addition of 2 M, 3 M, and 4 M HCl. The obtained results from the SSA test are the residual concentrations of Pb(II) solution and Cd(II) solution, or the final concentrations of the solutions (C_e). These results are then used to calculate the adsorption percentage and adsorption capacity. The adsorption results for Pb metal ions and Cd metal ions are presented in Tables 2 and 3.

Table 2. Data on adsorption results of Pb metal ions by Ouw Natural Clay (LAO)

Sample	C_e (ppm)	Co-C_e (ppm)	Q (%)	q (mg/g)
LAO pure	0.0631	49.9369	99.8738	4.99369
LAO-HCl 2M	4.2255	45.7745	91.5490	4.57745
LAO-HCl 3M	3.5870	46.4130	92.8260	4.64130
LAO-HCl 4M	2.1526	47.8474	95.6948	4.78474

Table 3. Adsorption results data of Cd metal ions by Natural Clay Ouw (LAO)

Sample	C_e (ppm)	Co-C_e (ppm)	Q (%)	q (mg/g)
LAO pure	4.6501	45.3499	90.6998	4.53499
LAO-HCl 2M	5.2671	44.7329	89.4658	4.47329
LAO-HCl 3M	5.4755	44.5245	89.049	4.45245
LAO-HCl 4M	5.5443	44.4557	88.9114	4.44557

Based on the data in Tables 2 and 3, it can be seen that pure LAO has a better ability to adsorb Pb metal ions and Cd metal ions compared to LAO after the addition of HCl. This is possible because in pure LAO, the pores and active sites are more open for adsorption compared to LAO after the addition of HCl, this is in accordance with research conducted by Prasetiowati, (2014).

LAO after the addition of HCl for the adsorption of Pb metal ions, has an adsorption capacity that increases directly proportional to the increase in acid concentration. From the research data in Table 2, it can be seen that LAO-HCl 4 M has a better ability than LAO-HCl 2 M and 3 M in adsorbing Pb metal ions. This is in accordance with the characteristics of the pore volume and pore size of LAO-HCl 4 M which are larger than the other samples.

The results of Cd metal ion adsorption in the research data in Table 3 show that Cd^{2+} with a smaller ion radius than Pb^{2+} can accumulate better in LAO-HCl 2 M, where the pore size is the smallest, namely 3.079 x 101 Å and the pore volume is quite large. LAO-HCl 3 M and 4 M with large pore sizes show lower absorption performance, this is likely due to the presence of larger pore sizes which can cause many Cd metal ions to escape during absorption. In addition, the addition of HCl at high concentrations can cause dealumination. This occurs because the interlayer of clay is saturated with H^+ ions, causing H^+ ions to replace Al^{3+}. The dealumination process causes damage to the octahedral layer which results in the collapse of the Si-Al framework in the clay, thus affecting the adsorption power. In addition, at high concentrations, the H^+ ions exchanged to the interlayer and clay lattice layers increase, so that more Al^{3+} ions dissolve (Prasetiowati, 2014).

This results in damage to the clay lattice structure which has an impact on decreasing absorption capacity. LAO adsorbs more Pb metal ions than Cd metal ions, this is in accordance with Prasetiowati's research (2014), which states that there is a strong interaction between medium acids and hard bases compared to soft acids and hard bases, where Cd^{2+} ions are a group of soft acids, Pb^{2+} ions are a group of medium acids, and the most dominant active side in clay is the type of hard base. In addition, the size of the Pb^{2+} ion radius, which is 1.32 Å, is also larger than the Cd^{2+} ion, which is 0.97 Å, so that Pb metal ions can accumulate more in LAO-HCl 4 M than Cd metal ions.

CONCLUSION

The characterization results of Ouw natural clay (LAO) before and after the addition of hydrochloric acid showed an increase in the specific surface area from 31.37 m^2/g for pure LAO to 99.68 m^2/g for LAO-HCl 4 M. The addition of hydrochloric acid to Ouw natural clay (LAO) reduced the adsorption capacity for Pb metal ions and Cd metal ions.

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