

Influence of Calsination Temperature on Zeolite Synthesis from Non-Hazardous Wastes and Its Application as an Adsorbent of Mn Metal Ion

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Abstract

In this research, zeolite material was synthesized by hydrothermal method using non-hazardou waste of fly ash as the raw material at low crystallization temperatures of 70°C with seawater as solvent. The effect of calsination temperature on synthesis of zeolite was studied, and the ability of synthesized zeolite to adsorb Mn metal ion in aqueous solution was also investigated. The synthesized zeolite was characterized by Fourier Transform Infra-Red (FT-IR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM-EDS), and Surface Area Analyzer (SAA). Based on the results of XRD, the type of zeolites produced from the synthesized zeolite are zeolite X and sodalite. The synthesized zeolite was then tested for the adsorption of Mn metal ion, using atomic absorption spectrophotometer (AAS) and it was found that the zeolite material synthesized calcined 550°C had the highest adsorption capacity compared to other synthesized zeolite materials, where the maximum adsorption was 192,31 mg/g.

Keywords

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adsorbent
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1. Introduction

In this era, the use of heavy metals in the industry continues to increase along with the development of the economy and the process of making chemicals^[1]. Contamination of water and soil resources by heavy metals is a serious global environmental threat because heavy metals are highly toxic even at concentrations of one part per million, can't be degraded, and can accumulate in the food chain^[2]. One of the heavy metal which is dangerous in the environment with high concentrations is manganese^[3].

Manganese has a variety of important roles in human survival such as being used in making ceramics, dry battery cells, and electric coils; also used as an alloying element. However, many manganese wastes are thrown directly into the waters without being treated first, so that the concentration of manganese in the waters becomes high and can cause serious health problems in living things^[3]. This is very dangerous for human survival so that the heavy manganese in the water needs to be removed by various methods that have been developed, one of them is the adsorption method using fly ash and zeolites^[4-5].

Fly ash is a by-product (waste) from thermal power plants that have been used in making concrete and cement^[6]. Fly ash in the form of fine and mild powder when touched by hand. Fly ash compositions generally consist of amorphous aluminosilicate, small amounts of quartz (SiO₂), mullite (Al₆Si₂O₁₃), hematite (Fe₂O₃), magnetite (Fe₃O₄), and carbon. Silica and alumina oxides have a porous structure and are very numerous in fly ash, so fly ash is suitable as a raw material for synthesizing zeolites^[7].

Zeolite is a material composed of hydrated aluminosilicate crystals in the framework of tetrahedral (Si,Al)O₄ building unit forming a porous system connected by oxygen atoms^[7]. The aluminum (Al³⁺) ion present at the center of the tetrahedral unit produces a negative charge and is balanced by the presence of extra cations (Na⁺, K⁺, Ca²⁺, and Mg²⁺) within the framework^[8]. One of the properties of zeolite is the ability to exchange cations with heavy metals and has pores that can adsorb heavy metals^[9].

Zeolites can be divided into two that are natural zeolites and synthetic zeolites^[7]. Natural zeolites can be found directly in nature without having to make it first. However, natural zeolites still have weaknesses. In nat-

ural zeolites, there are so many impurities such as Na, K, Ca, Mg, and Fe and the crystallinity of natural zeolites is less good compared to synthetic zeolites^[10]. Zeolites can be synthesized from some raw materials, one of the most commonly used materials and is inexpensive in the synthesis of zeolites is fly ash because fly ash contain so many Si and Al content has enabled it to be used as raw material in zeolite synthesis^[11].

Fatiha et al. (2013) has synthesized zeolite from coal fly ash PLTU Ombilin using hydrothermal alkali method using seawater and distilled water as solvent at low temperatures (35°C, 45°C, and 60°C) for its hydrothermal process, the resulting zeolite was not calcined and the type of zeolite produced is sodalite. Belviso et al. (2014) also tested the ability of Mn metal ion adsorption by fly ash and zeolite X synthesized from fly ash hydrothermally and got the result that zeolite X better at adsorbing Mn metal ion than fly ash. In this study, the zeolite from the hydrothermal process will be calcined. To obtain the optimal calcination temperature, calcination was carried out at variations of low temperature 350 °C, medium temperature 450 °C and high temperature 550°C.

2. Materials and Methods

2.1. Materials

The materials used are coal fly ash PLTU Ombilin Sawah Lunto West Sumatra, NaOH (Merck), Padang seawater, and MnSO₄.H₂O (Merck).

2.2. Instruments

The instruments used are glassware, mortar, analytical balance, stirrer, furnace, autoclave, oven, Whatman No. 42 filter paper, Atomic Absorption Spectrophotometer (AAS Variant AA240), XRD (Philip X'pert Powder Type PW4030 / 60), FT-IR (Subtech Spectrum ASCII PEDS 4.00), SEM-EDS (JSM IT300), and SAA (Quantachrome NovaWin).

2.3. Synthesis

2.3.1. Seawater Preparation

The seawater used is Padang seawater taken at the seashore, then filtered with filter paper to separate impurities and then stored in closed containers.

2.3.2. Synthesis of Zeolite from Coal Fly Ash

Zeolites are synthesized from coal fly ash and are processed using hydrothermal alkali using seawater as solvent. The zeolite synthesis process is carried out by tak-

ing 10 g fly ash and 12 g NaOH and then crushed them using mortar. After that, It's calcined with furnace at 550°C for 1 hour. The furnace yield added 46 mL of seawater for the hydrothermal process at pH~14, left overnight while stirring with a stirrer. Followed by the process of hydrothermal alkali in the autoclave for 4 days at 70°C. The results of this hydrothermal alkali are then filtered, there are precipitate and filtrate. The precipitate was dried for 24 hours at 80°C. The drying product is again crushed and continued with calcination in the furnace at 550°C for 5 hours. The above experiments were also carried out for results that were not calcined, calcined 350°C, and calcined 450°C.

2.3.3. Sample Characterization

Zeolites synthesized from the coal fly ash PLTU Ombilin were characterized using FT-IR to see functional groups of chemical structures of zeolites at 4000 cm⁻¹ to 600 cm⁻¹ wavenumbers. After that, characterization with XRD was also carried out to determine the crystal structure, type, and size of zeolites. Furthermore, to determine the surface shape morphology, uniformity of particle size and content of the elements contained in zeolites were analyzed using SEM-EDS. Also carried out testing with SAA to determine the pore diameter and surface area of zeolites.

2.3.4. Making a Standard Solution of Mn Metal Ion

The main solution for the standard is made from Mn salts (calculated based on the molecular weight formula of metal). Then weighed 1.5364 g and put it into a 500 mL volumetric flask so that the main solution has a concentration of 1000 mg/L. The main solution used for making solutions whose concentrations are lower (50, 100, 150, 200, and 250) mg/L. To obtain a solution with a lower concentration, each 1000 mg/L main solution is taken 12.5; 25; 37.5; 50; and 62.5 mL respectively using a pipette and diluted in a 250 mL measuring flask.

2.3.5. Effect of Contact Time on Mn

To determine the optimum contact time of adsorption of Mn metal ions by fly ash and zeolite synthesized, in each Erlenmeyer 0.015 g of fly ash and zeolite were added and then 20 mL of Mn metal solution concentration of 50 mg/L was added. Stirring was carried out during contact time variations (15, 30, 45, 60, and 75) minutes at a shaking speed of 150 rpm. The mixture was filtered and the filtrate was analyzed with AAS.

2.3.6. Adsorption of Mn Metal Ions with Fly Ash and Zeolite Synthesized

To determine the adsorption capacity of fly ash and zeolite synthesized, in each Erlenmeyer fly ash and zeolite were added 0.015 g in 20 mL of solution (in different containers) containing Mn heavy metal ions with varying concentrations of 50, 100, 150, 200 and 250 mg/L. Shaking with optimum contact time at a shaking speed of 150 rpm. Then the solution containing heavy metal ions was filtered with filter paper and the filtrate was analyzed with AAS.

3. Results and Discussion

3.1. FT-IR Analysis of Zeolite Synthesis Results

FT-IR results in Figure 3.1. shows that all zeolite materials synthesized have absorption bands in the range of wavenumbers 1500-850 cm^{-1} indicating asymmetric strain of O-Si-O or O-Al-O bonds originating from tetrahedral $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$. These wavenumbers on the zeolite material synthesized are 1391, 1433, 1421, 1437, 948, 967, 970, and 966 cm^{-1} . While in the range of wave numbers 750-650 cm^{-1} indicates the symmetrical strain of O-Si-O or O-Al-O bonds in zeolite material where these wavenumbers on the zeolite material synthesized are at 737, 721, 718, and 702 cm^{-1} . The presence of absorption bands in the two ranges of wavenumbers indicates that all zeolite material synthesized has been successfully formed^[13-15].

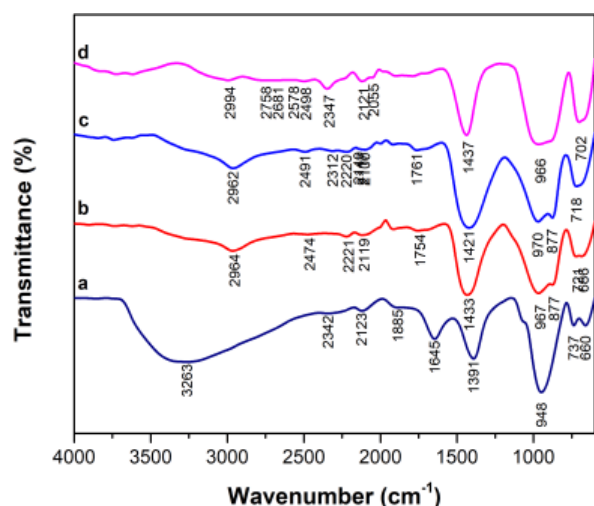


Figure 3.1. FT-IR spectrum of zeolite material synthesized with seawater as solvent: (a) without calcination (b) calcined 350°C (c) calcined 450°C (d) calcined 550°C

Figure 3.1. also shows the differences in the absorption peaks of zeolites synthesized without calcination and calcined in the range of wavenumbers

3400-2550 cm^{-1} and the range of wavenumbers 1650-1600 cm^{-1} . In the region of the wavenumber range 3400-2550 cm^{-1} indicating the presence of a hydroxyl group (-OH) from the H_2O molecules, this group may be derived from hydrates water that are still bound to zeolites. This hydroxyl group is only seen in zeolite material which is synthesized without calcination, that is in wavenumber 3263 cm^{-1} ^[13,16]. Whereas in the range of wavenumbers 1650-1600 cm^{-1} indicates the existence of O-H bending vibrations derived from water molecules (H_2O) attached to the zeolite framework. This wavenumber is only found in zeolite material which is synthesized without calcination, that is in wavenumber 1645 cm^{-1} ^[15,17].

3.2. XRD Analysis of Zeolite Synthesis Results

From the X-ray diffraction pattern in Figure 3.2. it appears that all the synthesized materials contain zeolite X and sodalite, but there are differences in the amount of zeolite produced based on the calcination temperature used. Zeolite materials which are synthesized without calcination have a higher amount of zeolite X compared to sodalite, whereas for Zeolite materials which are synthesized with calcination have a higher amount of sodalite compared to zeolite X. This is caused by the -OH bond present in the zeolite broken by the influence of calcination temperature and then form bonds with -Cl, so that the amount of sodalite produced is greater. From the XRD results, it was also seen that the calcination temperature of 550°C resulted in a higher sodalite peak than the other calcination temperatures. The differences in calcination temperature used show that the higher the calcination temperature used then the more sodalite formed^[18-19].

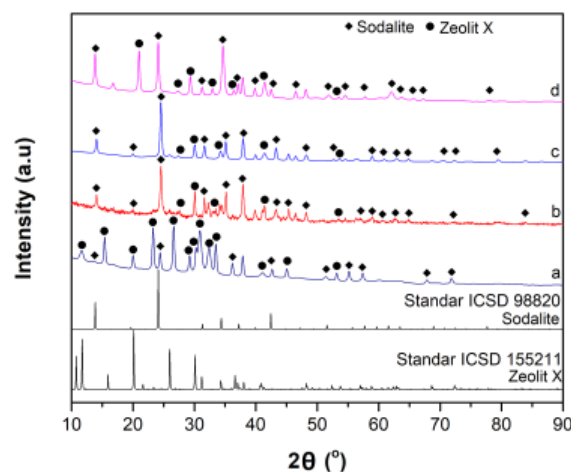


Figure 3.2. X-ray diffraction pattern of zeolite material synthesized with seawater as solvent: (a)

without calcination (b) calcined 350°C (c) calcined 450°C (d) calcined 550°C

3.3.SEM-EDS Analysis of Zeolite Synthesis Results

It can be seen that the surface morphology of the zeolite materials synthesized without calcination have more open pore than the zeolite synthesized calcined 550°C. From the XRD result in Figure 3.2. seen that the zeolite materials synthesized without calcination and calcined 550°C have the type of zeolite X and sodalite so that in Figure 3.3. can be seen the particle shape of zeolite X and sodalite that is rod-shaped. This is in accordance with previous research Belviso et al. (2010) who has succeeded in forming zeolite X and sodalite in which rod morphology is obtained. From the SEM-EDS data, it is obtained the EDS data contained in Table 3.1.

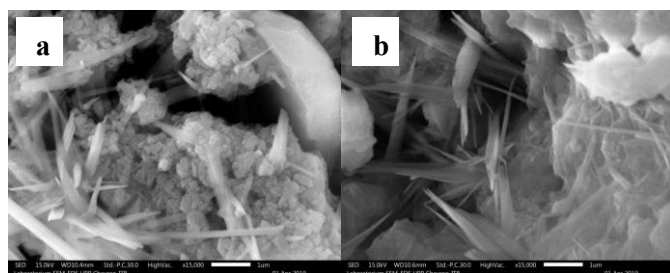


Figure 3.3.Surface morphology of zeolite material synthesized with seawater as solvent: (a) without calcination magnification 15,000x (b) calcined 550°C magnification 15,000x

Table 3.1.EDS Zeolite Data Synthesis Results with Seawater Solvent Without Calcination and Calcined temperature of 550°C

Without Calcination		Calcined 550°C	
Component	%Mass	Component	%Mass
O K	39,75	O K	36,50
Na K	30,24	Na K	27,85
Si K	16,45	Si K	14,87
Al K	10,49	Al K	6,19
Fe K	1,03	Fe K	8,34
Ca K	1,01	Ca K	2,58
Mg K	0,55	Mg K	0,43
K K	0,50	K K	3,24
	100		100

From EDS data it is seen that the zeolite materials synthesized without calcination and calcined 550°C are composed of sodium, silica, and alumina which are the main components of zeolite. The zeolite material synthesized without calcination has a Si/Al ratio = 1.57. This is consistent with the XRD result that the zeolite material synthesized has a large amount of zeolite X and zeolite X belongs to the type of zeolite synthesized with

low Si content, wherein zeolite synthesized with low Si content has Si/Al ratio $1 < 2$ [10].

3.4.SAA Analysis of Zeolite Synthesis Results

Figure 3.4. shows the adsorption isotherm curve of zeolite material synthesized without calcination is type-IV, while for zeolite material synthesized calcined 550°C is type-II. The Type II isotherm represents unrestricted monolayer-multilayer adsorption. From the two curves, it can be seen that adsorption occurs at the middle relative pressure ($0.1 < P/P_0 < 0.9$) which shows that the dominant structure of the zeolite material synthesized without calcination and calcined 550°C are mesoporous. However, the curve also shows growth at relatively low pressures at P/P_0 pressures < 0.1 , which indicates the presence of a small micropore structure [21].

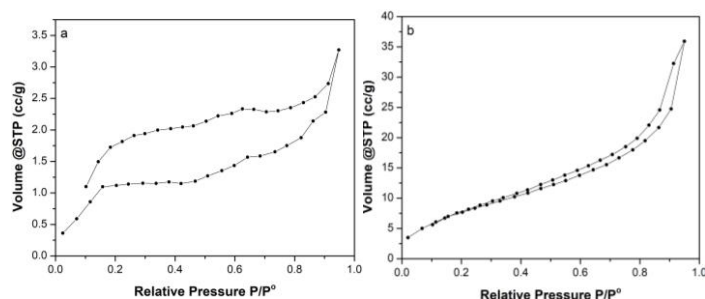


Figure 3.4.Graph of BET Isotherm from zeolite material synthesized with seawater as solvent: (a) without calcination (b) calcined 550°C

Specific surface area of zeolite synthesized without calcination and calcined respectively is 1.35836 m²/g and 16.976 m²/g. The average pore size of zeolites synthesized without calcination and calcined respectively were 2.4426 nm and 3.7787 nm. The total pore volume of zeolites synthesized without calcination and calcined sequentially is 0.0050709 cm³/g and 0.055677 cm³/g. From the adsorption-desorption N₂ Data, zeolite synthesized by the calcination process has a wider specific surface area, larger pore size and larger total pore volume, this is because the calcination process can (1). remove water and impurities contained in the zeolite, thereby increasing the specific surface area and pore size. (2). develop the zeolite pore structure, thereby increasing the total pore volume and pore size. (3). increase the crystallinity of zeolite, thereby increasing the specific surface area and adsorption properties. (4). reduce the agglomeration of zeolite particles, thereby increasing the specific surface area and pore size. The pore size obtained in this study is classified as mesoporous zeolite because it has a pore size in the mesoporous zeo-

lite range of 2–50 nm^[22]. Based on the description above, it can be seen in Table 3.2.9999999999999999`1

Table 3.2. Adsorption-Desorption N₂ Data

	Without Calcination	Calcined 550°C
Specific Surface Area	1,35836 m ² /g	16,976 m ² /g
Average Pore Size	2,4426 nm	3,7787 nm
Total Pore Volume	0,0050709 cc/g	0,055677 cc/g

3.5.Application of Adsorbent (Fly Ash and Zeolite Material Synthesis) to Adsorption Mn Metal Ion

3.5.1.Determination of Optimum Contact Time of Adsorbent (Fly Ash and Zeolite Material Synthesized) to Mn Metal Ion Adsorption

Figure 3.5. shows the optimum contact time obtained to adsorb the most Mn metal ions obtained by each different adsorbent. For the fly ash and zeolite material synthesized without calcination, the optimum time is 15 minutes, while for zeolite material synthesized calcined 550oC, the optimum time is 30 minutes. So that the contact time used for concentration variations later is 30 minutes.

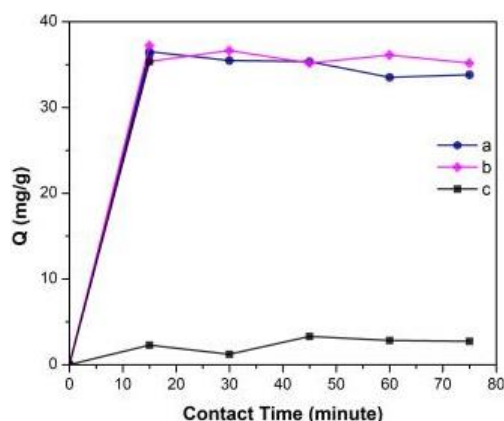


Figure 3.5. Adsorption capacity for contact time of adsorbents; zeolite material synthesized by seawater as solvent: (a) without calcination (b) calcined 550°C, and (c) fly ash

3.5.2. Adsorbent Adsorption Capacity (Fly Ash and Zeolite Material Synthesized) with Variations in Mn Metal Ion Concentration

From Figure 3.6. It is seen that the zeolite material synthesized with seawater solvent has a greater adsorption capacity compared to fly ash. From Figure 3.6. it is also seen that the higher concentration of Mn metal ions adsorbed, the higher the adsorption capacity is obtained.

This is due to the higher concentration of Mn metal ions, the strength of mass transfer will also be greater so that the zeolite material will have greater adsorption ability. Adsorption of Mn metal ions is caused by the transfer of Mn metal ions from the surface of zeolite material into the pores of zeolites and the exchange of cations with Na^+ . With the increasing concentration of Mn metal ions which will be adsorbed by zeolite material, the Mn metal ions which will undergo cation exchange with Na^+ are also increasing^[5,23].

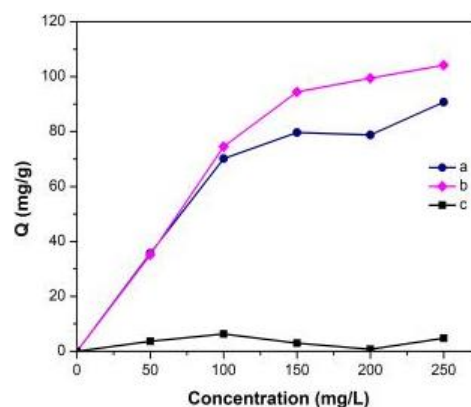


Figure 3.6. Adsorption capacity of Mn metal ion concentration of the adsorbent; zeolite material synthesized by seawater as solvent: (a) without calcination (b) calcined 550°C, and (c) fly ash.

Figure 3.6. also showed that the zeolite material synthesized which had the best adsorption was zeolite material synthesized calcined 550°C. This can be seen from the results of SAA that zeolite material synthesized by seawater solvent and calcined 550°C from Table 3.2. has a large surface area and total pore volume. So the greater the surface area and total pore volume of an adsorbent, the better the adsorption.

3.6. Mn Metal Ion Adsorption Isotherms by Synthesized Zeolite Material

From the graph in Figure 3.7. and Figure 3.8. obtained data as in Table 3.3.

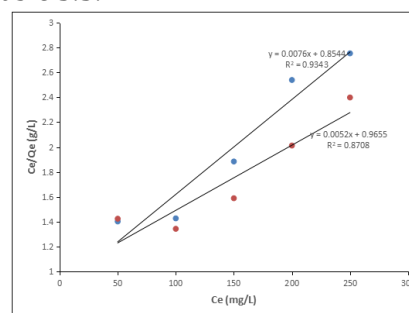


Figure 3.7. Langmuir adsorption isotherm from zeolite material synthesized with seawater as solvent: (a) without calcination (b) calcined 550°C

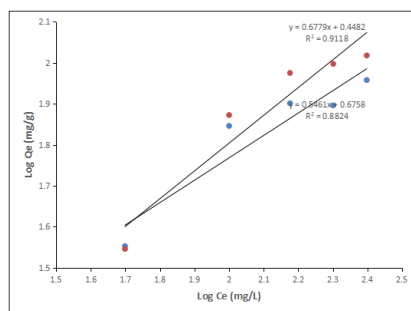


Figure 3.8. Freundlich adsorption isotherm from zeolite material synthesized with seawater as solvent: (a) without calcination (b) calcined 550°C

Table 3.3. Isotherm Adsorption Langmuir and Freundlich

Adsorbent	Langmuir		
	R ²	K _L	Q _{max}
Fly Ash	0,233	-0,034	1.467
Zeolit without calcination	0,9343	0.009	131,58
Zeolit calcined 550°C	0,8708	0,005	192,31

Adsorbent	Freundlich		
	R ²	n	K _f
Fly Ash	0,1047	-2,320	24,328
Zeolit without calcination	0,8824	1,831	4,740
Zeolit calcined 550°C	0,9118	1,475	3,078

From Table 3.3. obtained R² (coefficient of determination) from the Langmuir and Freundlich adsorption isotherm. It was seen that the zeolite material synthesized without calcination followed the Langmuir adsorption isotherm because it was seen from the value of $R \geq 9$ (close to 1). This indicates that the zeolite material synthesized without calcination performs a homogeneous adsorption on the surface to form a single layer (monolayer) and chemical bonds occur between Mn metal ions with zeolite material synthesized. From table 3.3. It was also seen that the zeolite material synthesized calcined 550°C following the Freundlich adsorption isotherm, which indicates that the zeolite material carries out heterogeneous adsorption on the surface to form more than one surface layer (multilayer) and Van der Waals interactions occur between the Mn metal ions with this zeolite material (adsorption of physics)^[17].

Based on Table 3.3. above is obtained the maximum adsorption capacity of each adsorbent (fly ash, zeolite material synthesized with seawater solvent without calcination and calcined 550°C), where the maximum capacity (Q_{max}) is owned by the material zeolite synthesized with seawater solvent and calcined 550°C that is equal to 192,31 mg/g.

4. Conclusions

Based on the research that has been done, it can be concluded that the PLTU Ombilin fly ash can be used as a raw material in synthesizing zeolites at 70°C hydrothermal temperatures and the types of zeolites produced are zeolite X and sodalite. The amount of zeolite material produced varies depending on the hydrothermal temperature and the presence or absence of calcination. All zeolite synthesized materials have a greater adsorption capacity than fly ash, so it can be said that all zeolite synthesized materials can be used as adsorbents in Mn heavy metal adsorption. The highest adsorption capacity is obtained by the zeolite material synthesized by seawater solvent and calcined 550°C so that it can be seen that the zeolite material synthesized by seawater solvent and calcined 550°C has a good Mn metal adsorption capacity.

Conflict of interest

The authors declare no conflict of interest.

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