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Synthesis of Zeolite-A from Oil Palm Shell Ash for Adsorption of Ferrous Metal Ions in

Borehole Water

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Abstract

Research has been conducted to adsorb Fe (III) ions in borehole water from Haruru Village, Central Maluku District, and artificial wastewater using A-type zeolite synthesized from oil palm shell ash. The oil palm shell ash was chemically activated by washing with 2 M HCl, serving as a source of SiO2. The synthesized zeolite A underwent characterization using X-ray diffraction (XRD) to determine its crystallinity. Batch adsorption of Fe (III) ions using zeolite A in borehole water and artificial wastewater was performed, involving variations in contact time and adsorbed iron concentration, and was analyzed using atomic absorption spectroscopy. The XRD analysis results indicated that the peaks of zeolite A were more pronounced at a NaOH mass of 93.75 grams, totalling ten peaks. The analysis of the initial concentration of Fe (III) ions in borehole water was 1.2151 ppm, while in artificial wastewater, it was 2.00 ppm. The optimum capacity and efficiency of zeolite A for Fe (III) ions in borehole water from Haruru Village were observed at a contact time of 40 minutes, with a capacity value of 0.1558 mg/g and an efficiency of 10.26%.

Keywords: Adsorption, Oil palm shell ash, Zeolite A, Fe (III) Ion Adsorption

INTRODUCTION

The provision of clean water services in Indonesia still needs to be improved. Regional Drinking Water Companies (PDAM) can only supply the water needs of urban areas, and the quantity provided still needs to be increased. As a result, a majority of the population, not covered by these water services, typically resorts to using groundwater or surface water, such as borehole water, for their daily necessities. However, both of these water sources often only meet quantitative requirements. Without proper treatment, the physical, chemical, and biological quality of surface water and groundwater in most regions of Indonesia does not comply with standards (Minister of Health Regulation No. 492/Menkes/Per/IV/2010), making it unsuitable for consumption (Purwoto & Sutrisno, 2016)

Iron is classified as an essential metal because it aids in the formation of blood cells in the body. However, it can be categorized as non-essential when its concentration is excessively high. Iron enters water through biological processes under reducing or anaerobic conditions (without oxygen). When water containing iron or manganese is exposed to air or oxygen, slow oxidation reactions of iron take place,

resulting in the formation of unwanted precipitates or colloidal clusters of iron oxides (Kurniawan, Wahyuningrum, & Anggraini, 2018). These colloid deposits can adhere to or accumulate in pipe walls, causing stains on laundered clothes. Moreover, colloid deposits can pose issues in pipe distribution systems as they may trigger the growth of microorganisms such as crenothrix and clonothrix, leading to pipe clogging and unpleasant color and odor (Kurniawan et al., 2018)

Iron in water, in the form of $Fe²⁺$ ions, reacts with free air and undergoes oxidation to become $Fe³⁺$ ions, resulting in a yellow color. Iron in water can impart a metallic taste or odor to drinking water at high concentrations. Therefore, for drinking water, the permissible iron content is 0.3 mg/L according to the Minister of Health's Decision No. 492 of 2010. Meanwhile, the maximum allowable iron content in the standard for clean water quality is 1.0 mg/L (Purwoto & Sutrisno, 2016).

To address the issue, a water treatment system capable of reducing the high iron content is required. One method that can be employed is adsorption. Adsorption is the adherence of one substance to another, either chemically or physically, and it can occur on the surface of a solid or liquid. One material that can be used to mitigate iron ion pollution in borehole water is zeolite. Zeolite is an aluminosilicate compound with framework structures and pores (cavities) and channels filled with exchangeable cations and water molecules, allowing it to easily adsorb ions (Agustina et al., 2015).

Based on its properties, zeolite is classified into natural and synthetic zeolite. Zeolite A is one of the zeolites that can be synthesized from materials containing $SiO₂$ and $Al₂O₃$, resulting in alumina and silica crystals forming four double rings connected by oxygen atoms (Nurhabibah & Mutaqin, 2017). Silica for the production of sodium silicate reactant in zeolite A synthesis can be obtained from the ash of oil palm shell ash. In contrast, alumina sources can be obtained by melting a mixture of $AI(OH)_{3}$ and NaOH (Muis, Permana, & Anwar, 2019). This type of zeolite can be utilized as a cation exchange agent for water purification, as a catalyst in the chemical industry, and also as an adsorbent for gas absorption and industrial wastewater treatment (Kurniawan & Widiastuti, 2017).

Zeolite-A is a type of synthetic zeolite with a low Si/Al ratio, specifically 1 or close to 1. This characteristic makes zeolite with a low Si/Al ratio often used in the treatment of wastewater, improvement of water quality by reducing hardness, and as an ion exchanger (Haryanto et al., 2016). reported that coal bottom ash from the Paiton power plant contains 49.73% SiO₂ and 19.51% Al₂O₃. The discussion of pure $Al(OH)$ ₃ as the source of Al_2O_3 and the melting process followed by hydrothermal treatment at 100°C for 3 hours successfully transformed the coal bottom ash into zeolite A.

One of the materials containing silica and alumina is ash derived from the husks and shells of palm oil. Data from XRF analysis by Reubun (2021) indicates that the highest metal oxide content in palm husk and shell ash is silica $(SiO₂)$ at 75.8%. This finding is more significant than the study reported by Sofyanto (2021), which stated that the silica content in palm shells was 70%.

The objectives of this research are to determine the crystallinity of synthesized zeolite A based on NaOH weight variations using XRD, to measure the initial concentration of Fe (III) ions in borehole water, and to assess the capacity and efficiency of synthetic zeolite A adsorption on Fe (III) ions in both borehole water and artificial wastewater.

METHODOLOGY

Materials and Instrumentals

The tools used in this study were: Erlenmeyer flask, Measuring glass, Glassware (Pyrex), Separatory funnel, Measuring flask, Desiccator, Aluminum foil, Oven, 100 mesh sieve, Mortar, Furnace, Analytical balance, XRD (Shimadzu XRD 6000), AAS (Shimadzu AA-7000).

The materials in this study were: Palm shell ash from PT. Nusa Ina Group, Borehole water from Haruru Village, HCl, NaOH, Al(OH)3, Aquades, Whatman 42 filter paper, Universal pH indicator.

Preparation and Activation of Oil Palm Shell Ash

The process begins with the activation step using 750 mL of 2 M HCl solution on 100 grams of palm shell ash that has been ground using a 100 mesh sieve, followed by filtration. The obtained residue is neutralized with equates until it reaches a neutral pH and then dried at 100°C until a constant weight is achieved. The palm shell ash was then analyzed using X-ray diffraction (XRD) (Sunarti at al., 2022).

Preparation of Sodium Silicate

Sodium silicate solution is made by grinding 25 grams of shell ash with 31.25, 62.5, and 93.75 grams of dry NaOH. The ground mixture is placed in a porcelain crucible and then melted at a temperature of 500°C in a furnace for 30 minutes. After cooling, the melt is ground and mixed with 75 mL of distilled water, placed in a Teflon vessel, and left for 24 hours to dissolve completely. The solution is then filtered and diluted to 250 mL (Sunarti at al., 2022).

Preparation of Sodium Aluminate

Sodium aluminate solution is prepared by dissolving 30.50 grams of NaOH in 100 mL of distilled water. Then, 21.65 grams of $Al(OH)_{3}$ is added to the solution while stirring. After all the Al(OH) $_3$ has dissolved, the solution is diluted to 250 mL (Sunarti, S, 2021).

Zeolite A Synthesis

The synthesis of zeolite is carried out by mixing 60 mL of sodium silicate with 40 mL of sodium aluminate in a beaker with continuous stirring for 3 hours. The mixture is then poured into a Teflon vessel and placed in an oven for crystallization at 60°C for 24 hours. The resulting crystallized solid is washed until the filtrate is clear and the residue's pH is neutral. The solid is then dried at 80°C for 4 hours. The obtained solid is analyzed using XRD (Sunarti at al., 2022).

Adsorption of Fe (III) Ion

An amount of 25 mL of bore well water, with a known concentration, was placed in an Erlenmeyer flask, and 1 gram of synthesized zeolite A was added. The mixture was then stirred using a magnetic stirrer with varying contact times of 10, 20, and 40 minutes. Afterward, the mixture was filtered using the Whatman 42 filter paper, and the filtrate was analyzed using an atomic absorption spectrophotometer. For comparison, an analysis was performed on artificial waste containing iron metal at a concentration of 2 ppm.

Data Analysis

Adsorption capacity expresses the amount of contaminants absorbed from the sample solution, as indicated by the following equation (Widiastuti et al., 2011):

$$
q_t = \frac{(C_o - C_e)v}{m}
$$
 (1)

Where _{it} represents the adsorption capacity per unit weight of the adsorbent (mg/L), *Co* denotes the initial concentration of iron metal (mg/L), *C^e* represents the equilibrium concentration of iron metal, V denotes the volume (L), and m indicates the mass of the adsorbent (g).

Adsorption efficiency can be calculated using the following equation:

Adsorption efficiency $(\%) = \frac{(C_o - C_e)}{c}$ $\frac{1-e_e}{c_o}$ 100%) (2) (Abdou, 2013)

RESULTS AND DISCUSSION

Activation of Oil Palm Shell Ash

The ash sample used in this study is waste from oil palm shell ash obtained from PT. Nusa Ina Group, North East Seram sub-district, Kobi, Central Maluku Regency, Maluku Province. The oil palm shell ash was ground using a mortar and sieved with a 100 mesh sieve to homogenize the particle size of the ash. This was done to ensure that the adsorbent's absorption process of the adsorbate is faster because theoretically, the smaller the particle size, the larger the surface area of the adsorbent (Solika, 2017).

The leaching process was carried out in the initial stage of purification to separate impurities other than SiO2. HCl was used as a strong acid to dissolve some metal compounds contained in oil palm shell ash. Activation was performed by immersing the oil palm shell ash in HCl and stirring it using a shaker for 18 hours to maximize the separation of impurities in the oil palm shell ash that had been sieved with a 100 mesh sieve. The activated ash was then neutralized with distilled water and dried in an oven at 100°C

until a constant weight was achieved (Sunarti, S, 2021). The drying process aims to reduce the moisture content in the ash.

Making Sodium Silicate & Sodium Aluminate Solutions

In this research, a sodium silicate solution was prepared by grinding 25 grams of oil palm shell ash with varying amounts of NaOH: 31.25, 62.5, and 93.75 grams. Subsequently, the mixture underwent fusion in a furnace at a temperature of 500°C for an optimum duration of 30 minutes (Sunarti, Mariwy, & Laitupa, 2023). During the fusion stage, a dissolution process occurred, involving the release of Si and Al components due to the influence of adding the alkali base NaOH to the ash (Bukhari, Behin, Kazemian, & Rohani, 2015). The dissolution process is influenced by the activator, temperature, and fusion time (Mujiyanti, Ariyani, & Lisa, 2021). The fusion time affects the formation of zeolite, resulting in residues with different Si and Al compositions.

The fusion was carried out at a temperature of 500 °C, based on the melting point of NaOH at 318 °C. At this temperature, NaOH fully dissociates, forming Na⁺ and OH-ions, facilitating the formation of sodium silicate solution at a not excessively high temperature. Fusion and dissolution were performed to ensure the transformation of silica ash into sodium silicate (Trivana, Sugiarti, & Rohaeti, 2015).

The resulting fused mixture was added to 75 mL of distilled water and allowed to stand for 24 hours. Subsequently, filtration was carried out to separate the filtrate and residue. The filtrate was then diluted with distilled water to a volume of 250 mL, serving as the crystallization medium.

Figure 1. Sodium Silicate Solutions Extracted from Oil Palm Shell Ash with Various NaOH Variations. (a) 31.25 g, (b) 62.5 g, (c) 93.75 g.

The sodium silicate solution resulting from different NaOH variations exhibited color differences. In the image (a), with 31.25 g of NaOH, the solution appeared Dijon yellow; in the image (b), with 62.5 g of NaOH, it appeared light yellow; and in the final solution, with 93.75 g of NaOH, it appeared mustard yellow. The color change is likely due to a reduction in impurities present in the ash, allowing the Si and Al components to react more easily to form zeolite.

The fusion reaction between $SiO₂$ and NaOH:

$$
SiO_{2(s)} + 2NaOH_{(s)} \rightarrow Na_2SiO_{3(s)} + H_2O_{(g)}
$$
(1)

(Trivana et al., 2015)

In this study, the preparation of sodium aluminate solution was carried out by dissolving 30.50 grams of NaOH in 100 mL of distilled water. Subsequently, 21.65 grams of

 $Al(OH)$ ₃ was added to the solution and stirred until dissolved. Distilled water was then added to reach a volume of 250 mL (Sunarti 2021). The use of $Al(OH)_{3}$ as a source of alumina is because $Al(OH)$ ₃ dissolves in sodium hydroxide (NaOH). The reaction between NaOH and $AI(OH)$ ₃ is as follows:

 $\text{Al(OH)}_{3(s)} + \text{NaOH}_{(aq)} \rightarrow \text{NaAlO}_{2(aq)} + 2\text{H}_2\text{O}_{(l)}$ (2)

(Faradina et al., 2016)

The sodium silicate and sodium aluminate solutions are subsequently utilized for the synthesis of zeolite A.

Zeolite A Synthesis

In this study, the zeolite crystallization process was carried out through a hydrothermal process to transform the amorphous phase into crystalline solids. The hydrothermal process was conducted in an oven at an optimum temperature of 60°C for 24 hours. Nurhabibah & Mutaqin, 2017 explain that zeolite formation occurs through the following process:

Figure 2. Scheme of the general mechanism of zeolite formation

From Figure 2, it can be explained that when the alumina solution and silica solution are mixed, two phases

are formed: the gel phase and the saturated solution phase. These two phases are in equilibrium as the initial stage of crystal formation. In the crystal formation stage, the amorphous gel undergoes a rearrangement of its structure through heating, forming crystal nucleus embryos. There is an equilibrium between crystal nucleus embryos, the remaining amorphous gel, and the saturated solution. This process is in a metastable state. If the remaining amorphous gel dissolves again, crystal growth will occur from these nucleus embryos until the remaining amorphous gel is depleted, and crystals are formed in a stable state.

The synthesis reaction of zeolite A is as follows: $NaOH_(aq) + Na₂SiO_{3(aq)} + NaAl(OH)_{4(aq)} \rightarrow$ $[Na_x(AIO_2)_y(SIO_2)_zNaOH.H_2O]_{(gel)} \rightarrow$ $Na_{12}[(SiO_2)_{12}(AlO_2)_{12}]$.27H₂O (zeolite-A crystal) (3)

(Kurniawan & Widiastuti, 2017)

The produced zeolite is in the form of a white solid, and further analysis is conducted using XRD to determine its concentration level.

Analysis of the Crystallinity of Activated Ash Using XRD (X-Ray Diffraction)

XRD analysis aims to determine the crystallinity of zeolite A and to obtain information about the microstructure of a crystal, including its phase purity and lattice changes based on the 2θ angle (Bukhari et al., 2015). The diffractogram image provides an illustration of the crystallinity level of palm shell ash containing silica, with the SiO2 diffractogram pattern showing characteristic sharp and high-intensity peaks (Faradina et al., 2016).

Figure 3. XRD (X-Ray Diffraction) Crystallinity Analysis Diffractogram Data of Activated Ash with Various NaOH Variations. (Figure 1) 31.25 g, (Figure 2.) 62.5 g, (Figure 3.) 93.75 g.

This diffractogram data refers to the zeolite A standard data from Treacy and Higgins (2007). Based on the diffractogram data, for Zeolite A with a NaOH mass of 93.75 g, 10 peaks were formed with the highest peak and a relative intensity value of 100% at an angle of $2\theta = 29.85$. Several other zeolite peaks appear. Meanwhile, at a NaOH mass of 65.2 g, Zeolite A is formed at angles of $2\theta = 23.17$, 26.53, and 29.14, with the highest peak owned by the Faujasite-type zeolite at an angle of $2\theta = 6.05$ with a relative intensity of 100%. In the diffractogram data of NaOH mass of 31.25 g, Zeolite A is formed at angles of $2\theta = 23.23$, and 26.73, with the highest peak owned by the Analcime-type zeolite at an angle of 2θ $= 18.13$ with a relative intensity of 100%.

Based on the XRD results of the three NaOH masses, more Zeolite A peaks are formed at a NaOH mass of 93.75 g. This is because Zeolite A is a lowsilica synthesis zeolite with a cubic crystal structure with eight tetrahedra (four fused rings) and an octahedron with 24 tetrahedra that easily undergoes a structural change into zeolite with a more complex structure, such as faujasite zeolite. Faujasite zeolite has a framework similar to zeolite A (LTA) and sodalite, consisting of 24 T atoms (six four-membered rings or four six-membered rings) (Xu et al., 2009). This indicates that the addition of NaOH in the production of sodium silicate causes the formed zeolite to undergo a structural change into another type of zeolite. This aligns with the statement by (Faradina et al., 2016) that NaOH significantly influences the type, purity, and homogeneity of the produced zeolite.

Analysis of Initial Fe Concentration in Borehole Water from Haruru Village, Central Maluku Regency

The concentration of Fe in the borehole water of Haruru Village, Central Maluku Regency, was analyzed using AAS. The initial step involved establishing a standard curve for the iron solution. The standard curve was created to obtain a linear regression equation that would be used to determine the iron concentration in borehole water. The standard curve was prepared by diluting the Fe (III) stock solution of 1000 ppm to 100 ppm and further diluting it to 10 ppm. Subsequently, a series of standard Fe (III) solutions with concentrations of 0.0, 2.0, 4.0, 6.0, and 8.0 ppm were prepared and their absorbance was measured using AAS. The absorbance data for the series of standard solutions are presented in Table 1.

Table 1. Absorbance Measurement Data of Standard

Based on the data in Table 1, it can be observed that the higher the concentration of the standard solution, the higher the absorbance. This is by Lambert-Beer's law, which states that absorbance is directly proportional to the concentration of a substance.

Figure 4. Standard Fe (III) Solution Curve

Based on the analysis results, the iron concentration in the borehole water is determined to be 1.2151 mg/L. This concentration has exceeded the clean water quality standard, where the permissible standard is 1.0 mg/L. Therefore, adsorption using zeolite synthesized from oil palm shell ash with a NaOH weight of 93.75 g is conducted to reduce the iron concentration.

Adsorption of Fe (III) Metal Ion Using Zeolite A Synthesized from Borehole Water and Artificial Wastewater with Variation in Contact Time.

Adsorption time is one of the parameters in the adsorption process, as time is a factor that can describe the kinetics of an adsorbent in interacting with an adsorbate. Time can also be used as an indicator to determine the efficiency level of adsorbent utilization (Haryanto et al., 2016). In this study, various contact times between the adsorbent and adsorbate were applied to determine the equilibrium time in the adsorption process. Equilibrium time is the optimum time required for the adsorbent to adsorb the adsorbate in a balanced state.

After the adsorption process at various contact times, the mixture was filtered using filter paper, the filtrate was collected, and its absorbance was measured using SSA. Adsorption processes were conducted using zeolite A (resulting from the variation in NaOH mass of 93.75 g) with a quantity of 0.02 g, borehole water volume of 25 mL, and contact time variations of 10, 20, and 40 minutes. The data on the measured concentrations of borehole water and artificial wastewater after the adsorption process are presented as follows. The results obtained from the conducted research are displayed as shown in Table 2.

Adsorption of Fe (III) Ion in Borehole Water

The measurement results for borehole water, including the concentration after the adsorption process, capacity, and adsorption efficiency, are presented in Table 2.

Table 2. Adsorption Data Based on Contact Time

Variations					
Conta _{ct}	Initial Concent	After Adsorption		Adsorp tion	Adsor ption
Time (Minu tes)	ration of Borehol e Water (ppm)	Absorb ance	Concen tration (ppm)	Capaci ty (mg/g)	Efficie ncy (%)
10	1,2151	0.1143	1,1070	0.1351	8,89
20	1,2151	0,1176	1,1413	0,0922	6,07
40	1,2151	0,1127	1.0904	0,1558	10,26

The determination of adsorption capacity aims to understand the optimal capability of zeolite A adsorbent to adsorb Fe (III) ions in borehole water. Meanwhile, adsorption efficiency aims to assess the effectiveness of zeolite A adsorption. The calculated results of adsorption capacity and adsorption efficiency using zeolite A show that the highest adsorption capacity and efficiency for Fe (III) ions occur at a contact time of 40 minutes, with values of 0.1558 mg/g and 10.26%, respectively, based on a mass of adsorbent of 0.02 grams.

From the data in Table 2, it is also observed that there is an increase in the concentration of Fe (III) ions after contact with zeolite A. The increase in the concentration of Fe (III) ions in the borehole water sample ranges from 8.89% to 10.26%.

According to Lee et al. (2013), adsorption is a phenomenon closely associated with the surface, involving interactions between liquid or gas molecules and solid molecules. These interactions occur due to the attractive forces of atoms or molecules covering the surface.

Figure 5 shows that the optimal adsorption efficiency of Fe (III) ions in borehole water samples is achieved at a contact time of 40 minutes, reaching 10.26%. Adequate contact time is necessary for zeolite A to adsorb Fe (III) ions effectively. As the contact time increases, zeolite A particles have more opportunities to interact with the Fe (III) ions within their pores. However, after a certain point, the adsorption efficiency declines as the active sites of the adsorbent become saturated.

During the 20-minute contact time, there is a decrease in adsorption capacity. This may be due to the possibility that the adsorbate concentration is insufficient to interact with the adsorbent. As a result, not all adsorbate molecules bind to the active sites of the adsorbent due to saturation. Additionally, some of the adsorbates may be released from the adsorbent, leading to a decrease after reaching optimal conditions (Syafrianda, Yenie, & Daud, 2017).

Adsorption of Fe (III) Ion in Artificial Wastewater

Subsequent research was conducted using artificial wastewater containing Fe (III) at a concentration of 2 ppm with an adsorbent mass of 0.02 g and a wastewater volume of 25 mL. The contact time was varied at 10, 20, and 40 minutes.

The data from absorbance measurements of Fe (III) artificial wastewater after the adsorption process are presented in Table 3.

Table 3. Concentration and Absorbance of Fe (III) Artificial Wastewater After Adsorption Process with Variation in Contact Time, as well as Adsorption

Based on the analysis results presented in Table 3, it can be observed that there is an increase in the concentration of Fe (III) ions with longer adsorption time, indicating high adsorption of Fe (III) ions by zeolite A. This is in line with the statement (Purwoto & Sutrisno, 2016) that the adsorption capacity is directly proportional to time until a certain point.

Based on Table 3, it can be observed that Zeolite A successfully adsorbed Fe (III) ions in the artificial wastewater sample at a contact time of 40 minutes, achieving 80.25% efficiency. The determination of the adsorption capacity and efficiency of Zeolite A for Fe (III) ion content in artificial iron wastewater is considered maximum. This indicates that the produced Zeolite A is capable of adsorbing Fe (III) ions in the artificial wastewater itself (Ngapa & Ika, 2020).

A different outcome is observed in the borehole water sample used, where the optimum adsorption result is 10.36% at the same contact time of 40 minutes. This may occur due to the competition among other ions present in borehole water to bind with the active sites of Zeolite A. The reaction mechanism of Zeolite A with Fe ions is illustrated in Figure 7.

Figure 7. Reaction Mechanism of Zeolite A with Fe (III) Ion

In the mechanism depicted in Figure 7, ion exchange can be observed, where the exchange of $Fe³⁺$ ions with Al ions takes place. The undesired $Fe³⁺$ ions will be exchanged with Na⁺ ions. This phenomenon is possible because metal ions and water molecules can move freely, allowing reversible ion exchange without any change in form (Purwoto & Sutrisno, 2016).

CONCLUSION

Based on the research findings, it can be concluded that the crystallinity results of synthetic Zeolite A, based on the XRD analysis data, show the highest values for the NaOH mass variation of 93.75 g, with 10 peaks at angles 2θ = 7.05; 10.04; 12.28; 21.55; 23.08; 26.44; 26.94; 29.85; and 34.10, with relative intensity (%) at the angle $2\theta = 26.94$. The initial concentration of Fe (III) ions in the borehole water of Haruru Village, Central Maluku Regency, and artificial wastewater are 1.1251 ppm and 2.00 ppm, respectively. The adsorption capacity and efficiency of Zeolite A for Fe (III) ions in borehole water from Haruru Village are 0.1558 mg/g and 10.26%, respectively. Meanwhile, the adsorption capacity and efficiency of Zeolite A for Fe (II) ions in artificial wastewater are 2.4427 mg/g and 80.25%, respectively.

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