



Synthesis of Ni²⁺/Natural Zeolite by Electro-Ion Exchange

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ABSTRACT. Electro-ion exchange was carried out to synthesize Ni²⁺/natural zeolite at room temperature using various concentrations of Ni(NO₃)₂ of 0.05, 0.1, 0.15, and 2.0 M, zeolite wetting times of 0 and 24 hours, electrolysis times of 1, 2, and 3 hours, and voltages of 7.5, 15, and 28.4 V. The amount of Ni²⁺ that can be impregnated in the zeolite was monitored with Atomic Absorption Spectroscopy (AAS). The Ni²⁺/zeolite formed was characterized for its total acidity. The optimum conditions for this process are 28.4 V, 3 hours, and 0.1 M Ni(NO₃)₂. Under these optimum conditions, the Ni²⁺ content of the natural zeolite was 0.04 mmol/g, with a total acidity of 2.5 mmol/g. The increasing trend in the amount of Ni²⁺ content matched the trend in the total acidity. The synthesized Ni²⁺/natural zeolite is expected to be applied as a catalyst in reactions such as hydrogenation and hydrocracking.

INTRODUCTION

Metal impregnation of zeolites has been widely used for certain purposes. Preparation of M^{x+}/natural zeolite usually requires the formation of H/zeolite first as an initial process. However, this is rather laborious because it usually uses HCl and NH₄Cl (Fatimah and Utami, 2017; Windarti and Suseno, 2004). This study aims to offer a novel approach to the ion exchange process by preparing Ni²⁺/natural zeolite via electro-ion exchange, thereby simplifying the process of metal impregnation in zeolite. The use of this electro-ion exchange method is expected to improve green chemistry parameters by eliminating HCl and NH₄Cl in the synthesis process. Compared to the conventional ion exchange method, the electro-ion exchange method is faster because it involves only a single electrolysis step. In contrast, the preparation of Ni²⁺/natural zeolite using the conventional ion-exchange method requires acid activation, replacement of H⁺ with NH₄⁺, and subsequent replacement of NH₄⁺ with Ni²⁺.

Zeolite is a negatively charged solid that is balanced by cations. It must be placed in the salt-bridge position in an electrolysis process, where its balancing cations migrate toward the cathode and are replaced by Ni²⁺. The major balancing metal ions in natural zeolite are K⁺ and Ca²⁺, while heavy metal ions are minor ones (Putri and Side, 2020).

This method is similar to the Electro-deionization method in the water desalination process, which uses ion exchange membranes (Khan *et al.*, 2023), in which zeolite was used as a membrane. The migration of cations in zeolite requires an activation energy depending on the position of the cations (Ramsahye and Bell, 2008). This requires variations in electrical voltage. The ion exchange process in zeolite is also influenced by the concentration of the exchange ions (Ermiyati, 2002). As the optimum concentration has not been determined yet, varying the concentration of Ni²⁺ is needed.

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RESEARCH METHODS

The research materials used include natural zeolite from Klaten, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Merck), HCl (38%, Merck), HNO_3 (65%, Merck), NH_3 (25%, Merck), deionized water, and 99% pure Ni electrode. The electro-ion exchange apparatus was arranged as shown in [Figure 1](#). The container on the cathode was filled with deionized water, while the anode was filled with $\text{Ni}(\text{NO}_3)_2$ solution. A total of 5 g of zeolite was placed in the sample container completely, allowing the cations to migrate through the zeolite towards the cathode. Before the electrolysis process, wetting, namely allowing the zeolite to be immersed in the reactor by the electrolyte, was carried out for 0 and 24 hours. The electrolysis process was carried out at various concentrations of 0.5, 0.1, 0.15, and 2.0 M. The electrolysis time was varied to 1, 2, and 3 hours. The time variation was conducted because ion migration from the initial to the final end took time, and the optimal time was not yet known. The voltage was varied to 7.5, 15, and 28.4 V because each value could affect the electric field, which, in turn, determines the rate of ion mobility ([Goodenough and Park, 2013](#); [Maier, 2004](#); [Holyst and Poniewierski, 2012](#)). The material yielded was then washed with deionized water to remove $\text{Ni}(\text{NO}_3)_2$ that was not bound by the zeolite. Ni^{2+} ions impregnated in zeolite were detected by Atomic Absorption Spectroscopy (AAS) with $\text{Ni}(\text{NO}_3)_2$ as the standard. The Ni^{2+} /zeolite material was characterized for its total acidity gravimetrically by ammonia adsorption. The effect of voltage on the framework structure of natural zeolite was studied using XRD data.

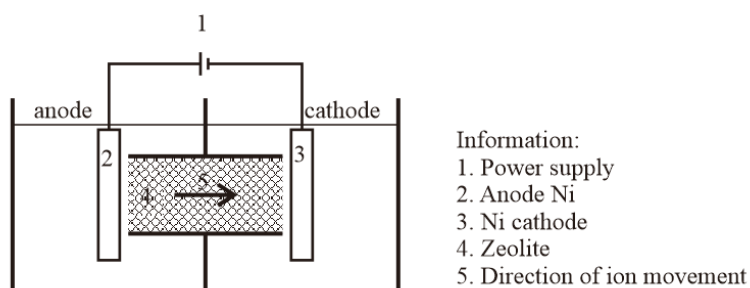


Figure 1. Electro-ion exchange device circuit.

RESULTS AND DISCUSSION

Optimization of Immersing (Wetting) Time and Voltage

This optimization aimed to ensure the continuity of the electrolysis process. The immersion process aimed to completely wet the zeolite with water, which served as a medium for cation migration. Meanwhile, voltage played a role as a driving force for cation migration in the zeolite solid, which functioned as a salt bridge between the electrodes. The effects of immersion and voltage variations are shown in [Tables 1](#) and [2](#), respectively.

Table 1. Effect of wetting natural zeolite before electrolysis^a.

Optimization	Observation results
Without wetting	No electric current was observed.
With 24-hour wetting	An electric current was observed.

^awith 28,4 V and 0.1 M $\text{Ni}(\text{NO}_3)_2$

Table 2. Optimization of voltage and direct current^a.

Voltage (V)	Electric current (A)	Observation results
7.5	0	No reaction at the electrode was observed.
15	0.01	A reaction at the electrode occurred, as marked by the appearance of bubbles at the cathode and slight dissolution of Ni at the anode.
28.4	0.01	A reaction at the electrode occurred, as marked by the appearance of bubbles and a shiny appearance at the cathode, and more Ni dissolved at the anode.

^aThe process was carried out after a 24-hour wetting, with a concentration of 0.1 M $\text{Ni}(\text{NO}_3)_2$

[Table 1](#) shows that without prior wetting of the zeolite, no electric current was observed, and the electrolysis process did not occur. This indicates that cation migration on the zeolite surface requires a medium. The process

of wetting the zeolite with the electrolyte took time. After 24 hours of wetting, the electrolysis process was observed, as indicated by the presence of an electric current.

Table 2 shows that the voltage affects the occurrence of electrolysis. Electrolysis with a zeolite-based salt bridge requires a relatively high voltage. At a voltage of 7.5 V, no electrolysis process was observed, while at a voltage of 15 – 28.4 V, an electrolysis process was observed. The higher the voltage, the more the anode dissolved, and the more bubbles formed on both electrodes. This indicates the breakdown of water into hydrogen and oxygen gas (El-Shafie, 2023). The continuation of this process allows the migration of balancing cations in the zeolite to the cathode, and Ni^{2+} ions migrated from the anode to replace the position of the balancing cations in the zeolite. The use of the highest voltage of 28.4 V is predicted not to damage the zeolite framework structure. This is because at 28.4 V, with a current of 0.01 A and an electrode distance of approximately 2 cm, the system generates an electric field of 1420 V/m and an electrical power of 0.284 W. For an aqueous electrochemical system, this electric field is considered moderate, and the electrical power is relatively low. The absence of structural damage is evidenced by the diffractogram shown in Figure 2. The diffractograms of natural zeolite and Ni^{2+} /natural zeolite are very similar.

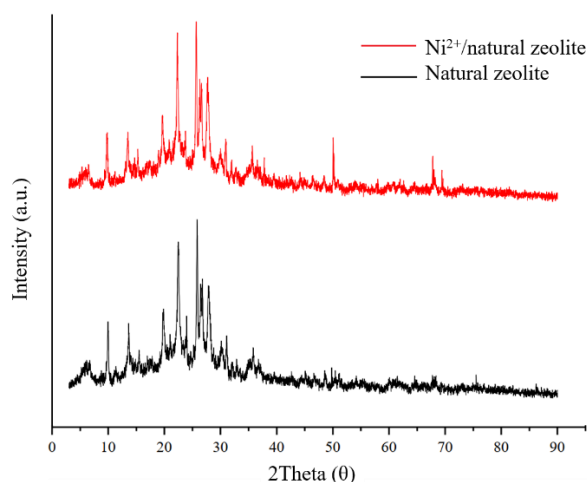


Figure 2. The diffractograms of natural zeolite and Ni^{2+} /natural zeolite.

Electro-ion Exchange

Electro-ion exchange was carried out in various concentrations of $\text{Ni}(\text{NO}_3)_2$ solution and at various times of the electrolysis process. The concentration of the electrolyte solution corresponds to the number of substituting ions being higher than that of the replaced ones. The time of the electrolysis process is related to the rate of ion exchange. The effect of $\text{Ni}(\text{NO}_3)_2$ concentration on the amount of Ni^{2+} impregnated is shown in Figure 3a.

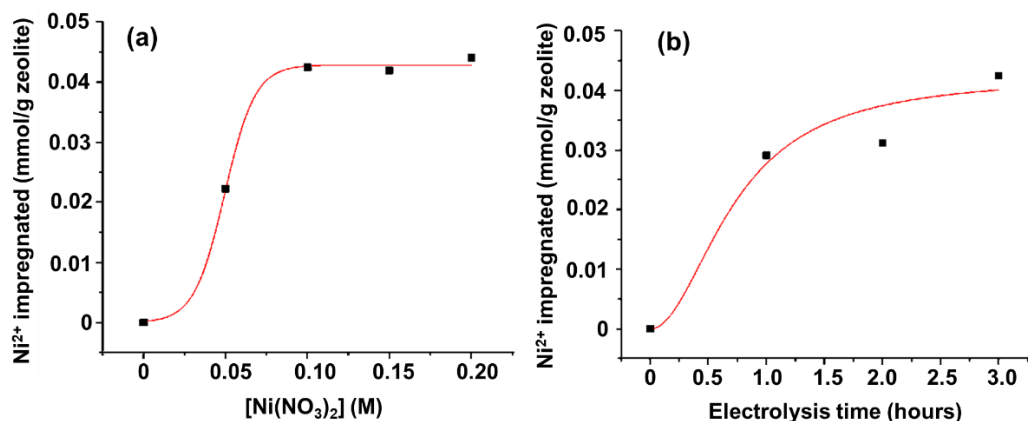


Figure 3. The amount of Ni^{2+} impregnated, (a) effect of $[\text{Ni}(\text{NO}_3)_2]$ on 3-hour electrolysis, (b) electrolysis time on 0.1 M $\text{Ni}(\text{NO}_3)_2$.

Figure 3a shows that the concentration of $\text{Ni}(\text{NO}_3)_2$ is directly proportional to ion exchange. The greater the number of cations, the greater the ability to replace the balancing cations in the zeolite. The optimal ion exchange

condition was found to be 0.1 M $\text{Ni}(\text{NO}_3)_2$ when the electrolysis was carried out for 3 hours. Figure 3b shows that the longer the electrolysis time, the more Ni^{2+} ions were impregnated.

Total Acidity of Ni^{2+} /Natural Zeolite

Total acidity is related to the nature of the solid for catalytic activity. It can describe the acidity level of the catalyst. In addition to providing information on the number of active sites, it is a combination of weak, strong, and physisorption sites. The effects of the $\text{Ni}(\text{NO}_3)_2$ concentration and electrolysis time are shown in Figure 4. As in Figure 4a, the optimum total acidity was found at 0.05 M $\text{Ni}(\text{NO}_3)_2$, in contrast to the amount of impregnated Ni^{2+} , which reached the optimum at 0.1 M $\text{Ni}(\text{NO}_3)_2$. This occurred possibly due to the amount of NH_3 bound by physisorption. The highest total acidity value in this experimental range was obtained after 3 hours of electrolysis. The relationship between the amount of Ni^{2+} impregnated and total acidity shows that greater Ni^{2+} impregnation results in higher total acidity. This is because Ni^{2+} acts as a Lewis acid that interacts with the Lewis base NH_3 (Mccaig, 2022).

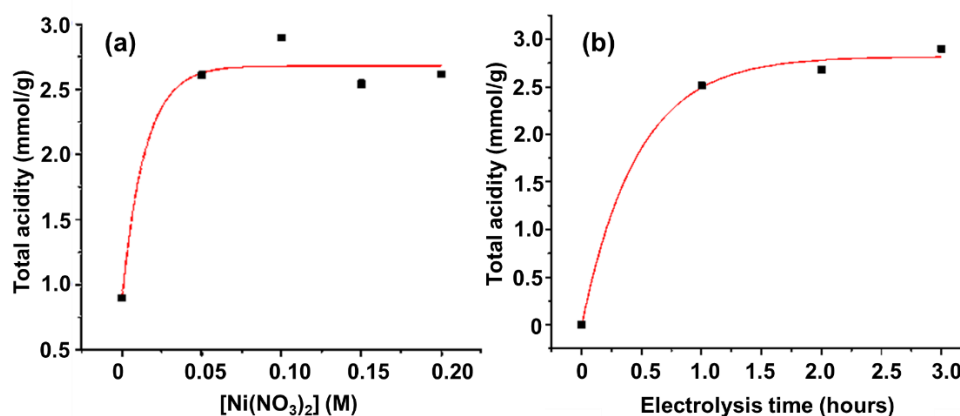


Figure 4. Total acidity of Ni^{2+} /natural zeolite: (a) effect of $\text{Ni}(\text{NO}_3)_2$ concentration on 3-hour electrolysis and (b) effect of electrolysis time on 0.1 M $\text{Ni}(\text{NO}_3)_2$.

CONCLUSION

This electro-ion exchange method offers a rapid alternative for the preparation of metal-supported natural zeolites that better aligns with green chemistry principles. The resulting material also has strong potential for hydrocracking applications.

CONFLICT OF INTEREST

There is no conflict of interest in this article.

AUTHOR CONTRIBUTION

IFN: Conceptualization and Data Analysis; FR: Manuscript Review and Editing; LAN: Experiment and Investigation; YH: Analysis, Writing, and Review; EH: Supervision and Analysis; KDN: Manuscript Review and Editing; TYH: Manuscript Review and Editing.

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