

# Ion Exchange of Na<sup>+</sup> Ions with H<sup>+</sup> Ions on ZSM-5 Zeolite Using Acetic Acid <sup>†</sup>

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<sup>†</sup> Presented at the IX International Congress “Engineering, Environment and Materials in Process Industry”—EEM2025, Bijeljina, Bosnia and Herzegovina, 2–4 April 2025.

## Abstract

ZSM-5 zeolites are primarily used in acid-catalyzed hydrocracking reactions in the petrochemical industry, and it is very important to ensure an adequate number of acidic sites for more efficient catalytic activity. This study investigated the possibility of exchanging sodium ions with hydrogen ions on ZSM-5 zeolite with a molar ratio of (SiO<sub>2</sub> / Al<sub>2</sub>O<sub>3</sub> = 1000) using an ion-exchange process with acetic acid. By employing the XRD and FT-IR methods, along with chemical analysis of ZSM-5 zeolite samples, the influence of hydrogen ion concentration on the chemical composition and structural characteristics of ZSM-5 zeolite was monitored at different acid concentrations and exchange times. It was shown that ion exchange with acetic acid leads to a significant reduction in sodium content even with less concentrated solutions while maintaining the stability of the crystal structure of ZSM-5 (SiO<sub>2</sub> / Al<sub>2</sub>O<sub>3</sub> = 1000) and a high degree of crystallinity.

**Keywords:** ion exchange; acetic acid; ZSM-5; zeolite



Academic Editors: Dragan

Vujadinović, Milorad Tomić, Vesna  
Gojković Cvjetković, Milan Vukić,  
Mitar Perušić and Slavko Smiljanić

Published: 20 June 2025

**Citation:** Došić, A.; Obrenović, M.;  
Obrenović, Z.; Vuković, J.; Savić, I.M.  
Ion Exchange of Na<sup>+</sup> Ions with H<sup>+</sup>  
Ions on ZSM-5 Zeolite Using Acetic  
Acid. *Eng. Proc.* **2025**, *99*, 17. <https://doi.org/10.3390/engproc2025099017>

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## 1. Introduction

Zeolites are aluminosilicate nanomaterials with wide applications in the industry due to their porous crystal structure, ion exchange and acid–base properties, and high specific surface area [1,2]. They are used as molecular sieves, in detergent production, as adsorbents, as catalyst carriers, and also as catalysts [3–7]. ZSM-5 (Zeolite Socony Mobile No. 5) zeolite has particular applications in the petrochemical industry, especially in hydrocracking reactions [8–10]. Specifically, ZSM-5 zeolite belongs to the group of high-silica MFI zeolites, which, due to their chemical composition, i.e., the presence of aluminum and a SiO<sub>2</sub> / Al<sub>2</sub>O<sub>3</sub> ratio > 50, exhibit pronounced acidic properties. The acidity of the zeolite decreases with an increase in the molar ratio of silicon to aluminum while hydrophobicity increases. The chemical composition of ZSM-5 zeolite is defined by the formula Na<sub>n</sub>Al<sub>n</sub>Si<sub>96-n</sub>O<sub>192</sub> · 16H<sub>2</sub>O, where n can range from 0 to 27. The aluminum content in the framework is relatively low compared to other zeolites and is proportional to the number of exchangeable cations, such as H<sup>+</sup> ions, which significantly affects its Lewis and Brønsted acidity [11–13].

The activity of ZSM-5 zeolite is related to its two main characteristics: selectivity and the presence of strong Brønsted acid sites within the pores. ZSM-5 zeolite exhibits high selectivity due to its pore system, with pore diameters of approximately 60 nm [14–16]. The

high selectivity of ZSM-5 zeolite is also a result of its specific crystal structure. The crystal lattice of ZSM-5 zeolite is a network composed of two types of intersecting channels at right angles, one of which is elliptical and straight (0.54–0.57 Å) and the other sinusoidal and circular in cross-section (0.51–0.54 Å). Each channel is formed by connecting ten pentagonal secondary building units (SBUs), known as pentasils, so that the channels have openings the size of 10-membered rings [13].

The application of ion exchange on ZSM-5 zeolite can influence the number of available Lewis and Brønsted acid sites, as well as their ratio, with the number of acid sites increasing as the acidity of the cations increases [17].

Based on previous research into the acid–base and ion-exchange properties of ZSM-5 zeolite, there is a need to explore the possibility of ion exchange on high-silica ZSM-5 zeolite with commercially available chemicals while simultaneously monitoring changes in the structural characteristics of the zeolite, as well as the stability of its crystal structure.

## 2. Materials and Methods

For the production of ZSM-5 zeolite ( $\text{SiO}_2 / \text{Al}_2\text{O}_3 = 1000$ ) at a facility for the production of special zeolites, amorphous silica with a particle size distribution up to 80 µm (Zeochem Ltd., Zvornik, Bosnia and Herzegovina) and sodium aluminate in powder form (Alumina Ltd., Zvornik, Bosnia and Herzegovina) are used as the primary raw materials, serving as sources of silicon and aluminum, respectively. In order to direct the formation of the crystal lattice during crystallization, stabilize the charge in the zeolite structure, and influence the synthesis rate of the zeolite itself, the so-called “templates” are often used in the synthesis of ZSM-5 zeolite. The empty space in the zeolite structure is occupied by these templates [18,19]. Tetrapropylammonium bromide (TPABr) (Sigma-Aldrich, St. Louis, MO, USA) was used as the template in this case. In addition to the primary raw materials, water glass rich in silicon content (Alumina Ltd., Zvornik, Bosnia and Herzegovina), caustic soda due to the basicity of the medium (Šiřecam Soda Ltd., Lukavac, Bosnia and Herzegovina), and a previously produced zeolite without specific characteristics (Alumina Ltd., Zvornik, Bosnia and Herzegovina) are used as the crystallization germ in the synthesis of ZSM-5 zeolite.

The experimental part was carried out in the Research Laboratory of Alumina Ltd. (Zvornik, Bosnia and Herzegovina). During the ion exchange process of ZSM-5 zeolite with acetic acid, a ZSM-5 zeolite sample calcined at 550 °C was used. The ion exchange process itself was performed in the Research Laboratory of Alumina, where a 20% suspension of calcined ZSM-5 zeolite and acetic acid was prepared, with acetic acid concentrations of 5%, 10%, and 15%. The suspension was then stirred on a magnetic stirrer for time intervals of 1, 3, and 6 h at a temperature of 90 °C. In the ion exchange reaction,  $\text{Na}^+$  ions were exchanged with  $\text{H}^+$  ions. After the ion exchange, vacuum filtration and washing were performed, and then the sample was dried at 105 °C in a drying oven (Fazos, Železniki, Slovenia) and used for further analysis. The loss on ignition was determined according to the standard method YS/T 575.19-2021 [20]. The determination of sodium and aluminum content in zeolite samples, before and after ion exchange, was performed according to the standard method ISO 11885 [21] on the ICP-OES SPECTROGENESIS device (Agilent Technologies, Inc., Santa Clara, CA, USA). Changes in the structural characteristics of the zeolite samples were shown using diffractograms collected on the X-ray diffractometer BRUKER D4 ENDEAVOR BRUKER D4 ENDEAVOR (Bruker AXS GmbH, Karlsruhe, Germany equipped with an X-ray tube with a copper anticathode, applying  $\text{CuK}\alpha$  ( $\lambda = 1.54060$  Å). The crystallinity degree of the zeolite samples was quantitatively determined according to the standard method ASTM D5758-1 [22] by comparing the peak areas characteristic of the standard ( $\text{SiO}_2 / \text{Al}_2\text{O}_3 = 400$ ) and the analyzed ZSM-5 zeolite samples ( $\text{SiO}_2 / \text{Al}_2\text{O}_3 = 1000$ ). For a

more detailed structural analysis of the samples, the FT-IR spectroscopy method (Fourier-transform infrared spectroscopy) was also applied, performed in the range of 500 to 4000  $\text{cm}^{-1}$  on the FT-IR spectrometer SHIMADZU IRAffinity-1S with a MIRacle 10 ATR accessory (Shimadzu, Kyoto, Japan).

### 3. Results and Discussion

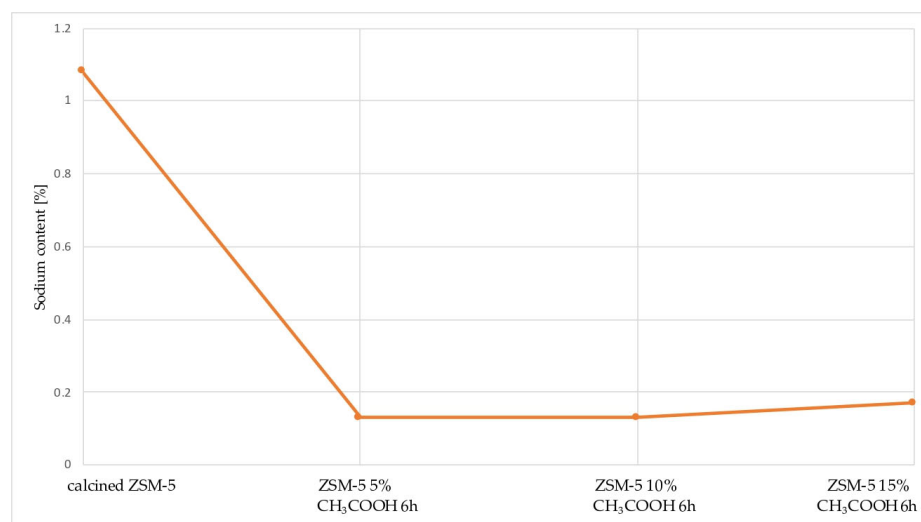
Based on the results presented in Table 1 and Figure 1, it can be observed that using 5%  $\text{CH}_3\text{COOH}$  already achieves a significant reduction in sodium content, around 88%, compared to the initial calcined zeolite sample. The reduction in sodium content during ion exchange with hydrochloric acid [23] is more significant compared to the reduction in sodium content achieved with acetic acid at the same acid concentration. The reason for this is that acetic acid belongs to weak organic acids, so the number of hydrogen ions produced by its dissociation will be significantly lower compared to the number of hydrogen ions produced by the dissociation of hydrochloric acid. Increasing the initial concentration of acetic acid during the treatment of ZSM-5 zeolite with this acid has a negative impact on ion exchange. This can be explained by the fact that increasing the concentration of the weak acetic acid reduces its dissociation degree. During ion exchange with 10% and 15%  $\text{CH}_3\text{COOH}$ , it can be observed that after just one hour of treatment, there is a significant reduction in sodium content, around 96%, compared to the initial calcined zeolite sample. By extending the ion exchange time beyond 3 h, the possibility of sodium ion exchange with hydrogen ions decreases. Furthermore, it can be observed that the ion exchange process practically ends within the first hour of treatment of ZSM-5 zeolite with acetic acid, and after that, a reverse reaction may occur, where sodium ions displace hydrogen ions from the zeolite.

**Table 1.** Effect of ion exchange time and  $\text{CH}_3\text{COOH}$  concentration on sodium content and  $\text{Al}_2\text{O}_3$ .

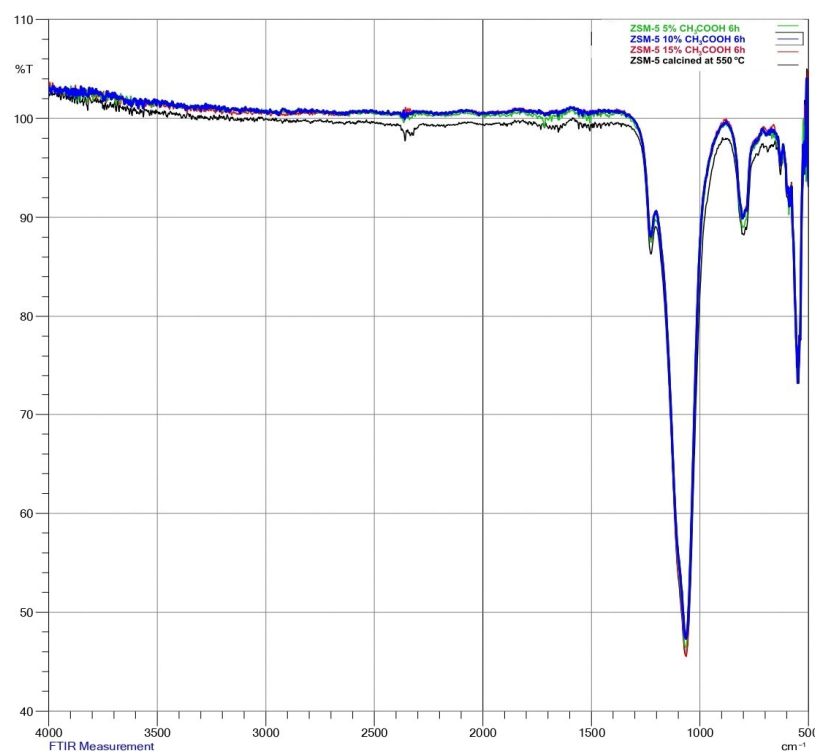
Sample	Ion Exchange Time [h]	Acid Concentration [%]	Loss on Ignition [%]	%Na	% $\text{Al}_2\text{O}_3$
calcined ZSM-5	/	/	4.01	1.08	0.18
ZSM-5 5% $\text{CH}_3\text{COOH}$ 6 h	6	5	1.64	0.13	0.17
ZSM-5 10% $\text{CH}_3\text{COOH}$ 1 h	1	10	1.67	0.044	0.17
ZSM-5 10% $\text{CH}_3\text{COOH}$ 3 h	3	10	4.69	0.048	0.16
ZSM-5 10% $\text{CH}_3\text{COOH}$ 6 h	6	10	1.57	0.13	0.16
ZSM-5 15% $\text{CH}_3\text{COOH}$ 1 h	1	15	3.26	0.041	0.16
ZSM-5 15% $\text{CH}_3\text{COOH}$ 3 h	3	15	2.89	0.046	0.15
ZSM-5 15% $\text{CH}_3\text{COOH}$ 6 h	6	15	0.04	0.17	0.15

The application of the ion exchange process with acetic acid of different concentrations leads to a slight decrease in the aluminum content in the zeolite (Table 1). Extending the ion exchange time with 10% and 15% acetic acid does not lead to significant changes in the aluminum content in the zeolite. Additionally, the reaction of aluminum from the zeolite with acetic acid primarily occurs during the first hour of the zeolite treatment with acid, in parallel with the ion exchange of sodium from the zeolite.

During the ion exchange of ZSM-5 zeolite with acetic acid, FT-IR spectra were obtained, as shown in Figure 2, which largely demonstrate the overlap of the characteristic peaks of the analyzed samples of ZSM-5 zeolite exchanged with acetic acid of different concentrations and the calcined sample of ZSM-5 zeolite.



**Figure 1.** Effect of CH<sub>3</sub>COOH concentration on sodium content.



**Figure 2.** FT-IR spectra of different ZSM-5 zeolite samples during ion exchange with CH<sub>3</sub>COOH.

The transmittance values for the zeolite samples exchanged with acetic acid are slightly higher compared to the transmittance of the initial calcined zeolite sample, which indicates the presence of greater stress in the structure of the exchanged zeolite samples. The increased stress in the crystal lattice is especially evident in the ZSM-5 zeolite exchanged with 15% CH<sub>3</sub>COOH for 6 h, as an intensified peak appears at 788 and 1084 cm<sup>−1</sup> for the ZSM-5 15% CH<sub>3</sub>COOH 6 h sample, compared to the initial calcined sample and the other ZSM-5 zeolite samples shown in Figure 2.

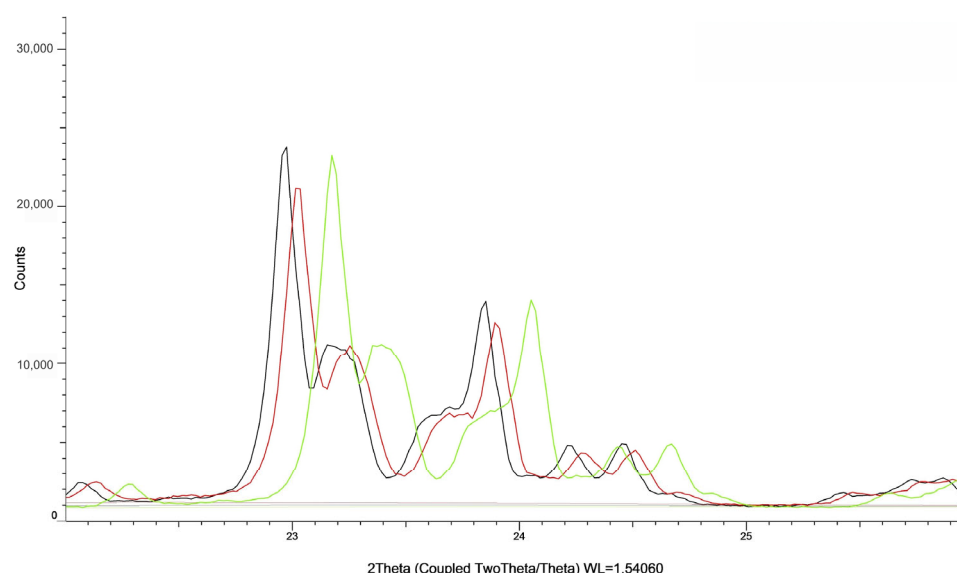
The values of the degree of crystallinity presented in Table 2 indicate that during ion exchange with 5% CH<sub>3</sub>COOH for 6 h, there is a decrease in the degree of crystallinity compared to the initial calcined ZSM-5 zeolite sample, which is a result of the ion exchange process occurring in the zeolite. Additionally, during ion exchange with 15% CH<sub>3</sub>COOH

for 6 h, the degree of crystallinity decreases compared to the calcined sample, which may be a result of the stresses occurring in the crystal structure of the zeolite, and these are not solely due to the ion exchange process of sodium with hydrogen ions.

**Table 2.** Comparison of the degree of crystallinity of ZSM-5 exchanged with acetic acid.

Sample	Degree of Crystallinity [%]
calcined ZSM-5	94.83
ZSM-5 5% CH <sub>3</sub> COOH 6 h	87.11
ZSM-5 15% CH <sub>3</sub> COOH 1 h	94.74
ZSM-5 15% CH <sub>3</sub> COOH 3 h	95.56
ZSM-5 15% CH <sub>3</sub> COOH 6 h	92.07

Figures 3 and 4 show the XRD diffractograms for ZSM-5 zeolite samples during ion exchange with acetic acid. The obtained peaks in the  $2\theta$  range of  $22.7\text{--}25.1^\circ$  confirm the crystal structure of all the analyzed ZSM-5 zeolite samples [13,24].

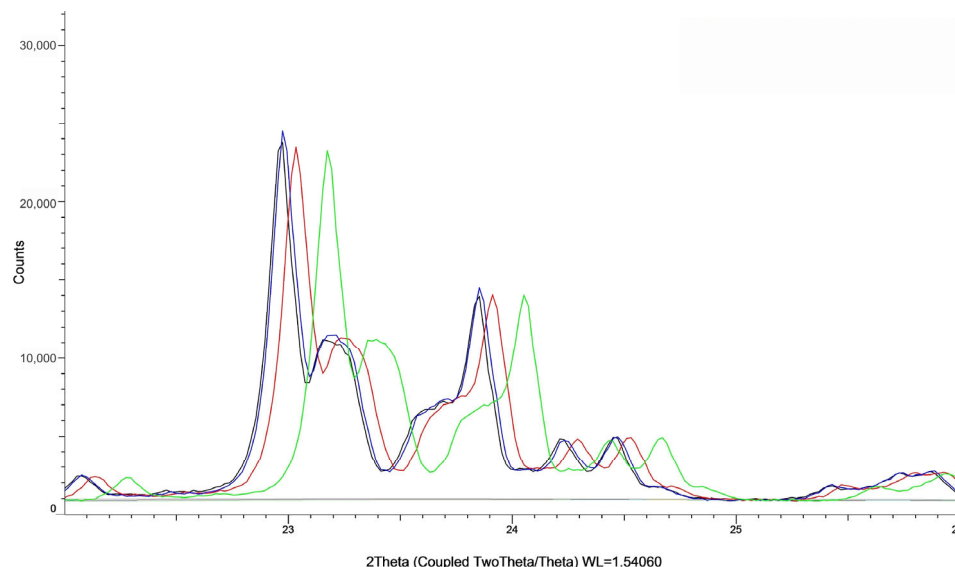


**Figure 3.** Comparison of XRD diffractograms: calcined ZSM-5 (—), ZSM-5 5%CH<sub>3</sub>COOH 6 h (—), and ZSM-5 15% CH<sub>3</sub>COOH 6 h (—).

By comparing the XRD diffractograms shown in Figure 3, a shift of the peaks to the left on the  $2\theta$  axis can be observed in relation to the initial calcined ZSM-5 zeolite sample, which confirms the dominance of the ion exchange process, as well as the results obtained from the chemical analysis, which indicate that the treatment of ZSM-5 zeolite with acetic acid does not significantly affect the reduction in aluminum content nor the destruction of the crystal structure of the zeolite (Table 2). For ZSM-5 5% CH<sub>3</sub>COOH 6 h, the intensity of the peaks in the  $2\theta = 22.7\text{--}25.1^\circ$  region is lower compared to the peak intensity of the initial calcined zeolite sample and the ZSM-5 15% CH<sub>3</sub>COOH 6 h zeolite, as ZSM-5 5% CH<sub>3</sub>COOH 6 h has the lowest degree of crystallinity (Table 2). In this case, the lower concentration of acetic acid, due to a higher degree of dissociation, likely accelerates the ion exchange process, creating stresses in the crystal lattice and reducing the degree of crystallinity of the ZSM-5 zeolite.

The diagrams in Figure 4 show that the XRD pattern of the zeolite sample treated with 15% CH<sub>3</sub>COOH for 1 h is shifted to the left along the  $2\theta$  axis compared to the calcined untreated zeolite sample, which confirms that within the first hour of treatment of the

zeolite with acetic acid, the ion exchange process of sodium ions with hydrogen ions occurred intensively. The diffraction patterns of the zeolite samples treated with 15% for 3 h and 6 h are practically identical, which is certainly the result of a reverse reaction and the exchange of hydrogen ions in the zeolite with sodium ions. All the obtained peaks in the  $2\theta$  range of  $22.7\text{--}25.1^\circ$  are shifted to the left compared to the initial calcined ZSM-5 zeolite, which confirms the fact that during the treatment of the zeolite with acetic acid, the dominant process is ion exchange, while the reaction of aluminum from the zeolite with acetic acid occurs with very low intensity.



**Figure 4.** Comparison of XRD diffractograms: calcined ZSM-5 (—), ZSM-5 15%  $\text{CH}_3\text{COOH}$  1 h (—), ZSM-5 15%  $\text{CH}_3\text{COOH}$  3 h (—), and ZSM-5 15%  $\text{CH}_3\text{COOH}$  6 h (—).

#### 4. Conclusions

Based on the obtained research results and a review of the literature references, it can be concluded that in the reaction of ZSM-5 zeolite with a  $\text{SiO}_2/\text{Al}_2\text{O}_3$  molar ratio of 1000 and acetic acid, the sodium content in the zeolite during ion exchange with 5%  $\text{CH}_3\text{COOH}$  for 6 h decreases by about 88%. Further increasing the concentration of acetic acid negatively affects the ion exchange process, as the increase in the concentration of the weak acid raises the possibility of a reverse process in which sodium ions displace hydrogen ions from the zeolite.

On the other hand, during the ion exchange process, reactions of aluminum ions with the acid also occur, which reduces the aluminum content. During the ion exchange of sodium with acetic acid, there are no significant changes in the reduction in aluminum content, so the process of dealumination does not practically occur. In the reaction of ZSM-5 zeolite with acetic acid, the dominant process is ion exchange, as all the obtained XRD diffraction patterns show a shift of the peaks to the left along the  $2\theta$  axis compared to the initial calcined zeolite sample.

During ion exchange with the acetic acid solution, the degree of crystallinity changes. The reduction in crystallinity during ion exchange with 5%  $\text{CH}_3\text{COOH}$  for 6 h occurs due to the presence of stresses in the crystal lattice of the zeolite caused by the increase in the number of hydrogen ions formed by acid dissociation at its lower concentration. With the action of more concentrated acetic acid solutions and with ion exchange times longer than one hour, the degree of crystallinity decreases compared to the calcined zeolite sample due to greater stresses in the zeolite structure, which are not exclusively the result of exchange between hydrogen ions and sodium ions in the zeolite. Results obtained



from FT-IR analysis, in combination with X-ray diffraction, confirm the possibility of ion exchange with acetic acid on ZSM-5 zeolite ( $\text{SiO}_2/\text{Al}_2\text{O}_3 = 1000$ ).

Based on the previously mentioned observations, it can be concluded that ion exchange with acetic acid has several advantages, as it leads to a significant reduction in sodium content even with less concentrated solutions while maintaining the stability of the crystal structure of ZSM-5 zeolite ( $\text{SiO}_2/\text{Al}_2\text{O}_3 = 1000$ ) and a higher degree of crystallinity. In addition, the degree of ion exchange with acetic acid is very high (around 90%) even after an ion exchange time of 1 h.

**Author Contributions:** Conceptualization, A.D. and Z.O.; methodology, A.D.; validation, A.D., Z.O. and M.O.; formal analysis, M.O.; investigation, M.O.; resources, A.D. and J.V.; data curation, A.D.; writing—original draft preparation, A.D., M.O. and I.M.S.; writing—review and editing, Z.O., I.M.S. and J.V.; visualization, M.O.; supervision, Z.O. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** Data are contained within the article.

**Conflicts of Interest:** Author Zoran Obrenović was employed by the company Alumina Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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