



Scientific article

RESEARCH ON THE ION EXCHANGE PROPERTIES IN HYDROCHLORIC ACID-MODIFIED ZEOLITE FOR WATER TREATMENT

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ABSTRACT

Zeolite is a group of a few microporous, crystalline aluminosilicate materials which has attracted much attention due to its remarkable absorption, ion exchange, and catalytic behavior. Their uses in water treatment, particularly for heavy metal and ammonium ion removal, have been greatly augmented through chemical modifications. This study investigates the impact of HCl treatment on the physicochemical and structural properties of natural clinoptilolite zeolite. First, it was refluxed with 0.97 M HCl for acid modification and was extensively purified, characterized using FTIR, XRD, gas sorption, and DTG. Overall, compared with the raw material, the results clearly show that the treatment with HCl can increase the surface area and cation exchange capacity, and improve the pore distribution by removing impurities and reorganization of the aluminosilicate building unit. Gas sorption confirmed a 64.4 % increase in the BET surface area (280.3 m²/g), along with a decrease in pore diameter, indicating enhanced adsorption efficiency. Adsorption experiments also indicated that most of the HCl-treated zeolites outperformed the unmodified samples for Disperse Blue 26 (DB 26). Furthermore, although acid modification often improves adsorption characteristics, the research indicates the possibility of structural stability issues stemming from framework leaching. The results of this study revealed the potential of HCl-modified zeolites as efficient and environmentally friendly adsorption materials for water treatment, including wastewater.

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1. INTRODUCTION

Zeolite is a family of several microporous, crystalline aluminosilicate materials commonly used as commercial adsorbents and catalysts [1]. They mainly consist of silicon, aluminum, and oxygen, and have a general formula. $Mn+1/n(AlO_2)-(SiO_2) \cdot x \cdot yH_2O$. where $Mn+1/n$ is either a metal ion or H^+ . These positive ions can be exchanged for others in a contacting electrolyte solution. H^+ -exchanged zeolites are particularly useful as solid acid catalysts [2]. When Axel Friedrich Kronstadt noticed that metallic materials absorb a lot of water vapor when heated, he named them zeolites. The strongly charged particles are trapped in the pores of the zeolite structure, and normal zeolites are formed when volcanic debris reacts with antacid water. Over tens of thousands of years, zeolites crystallized [1].

Zeolites, a class of crystalline aluminosilicates, have garnered significant attention due to their different applications, specifically in water treatment processes. Defined by their exclusive microporous construction and cation exchange properties, zeolites are famous for their flexibility

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in adsorption, ion exchange, and catalysis [3]. Zeolites are hydrated aluminosilicates with a three-dimensional framework composed of interconnected $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ tetrahedra, which are linked through shared oxygen atoms. The negative charge of the framework is balanced by exchangeable cations (M^{n+}), located within the channels and cavities together with water molecules [4]

This sub-atomic development leads to a wide cluster of zeolite types, including clinoptilolite, mordenite, chabazite, and Fauja site, each showing particular pore sizes and compound pieces (Regular Zeolites: Event, Properties, Applications).

Hydrochloric acid (HCl) modification of natural zeolites has developed as a favorable technique to increase their adsorption effectiveness for water purification. Acid treatment modifies the physicochemical properties of zeolites by eliminating impurities, improving surface area, and refining ion exchange capacity [5]. This modification significantly develops the adsorption of heavy metals and early impurities, preparing it as a successful approach for water treatment applications [6]. These results indicate a substantial improvement in lead and ammonium ions removal performance with the HCl-treated zeolites [7...8]. The technique increases the cation-exchange property and porous nature of the material, which facilitates the adsorption of contaminants [9].

Moreover, leaching of metals has an impact on the hydrophilicity and hydrophobicity of zeolites, affecting their selectivity between various contaminants [10]. Modified zeolites have been very efficient for different industrial and environmental applications [11]. especially in wastewater treatment. highlighted the sustainability and economic feasibility of natural zeolites as a long-term treatment for large quantities of water. Furthermore, [12] which showed that metal-modified zeolites are advantageous, supports this potential for acid-treated variants in selective adsorption. HCl pretreatment of zeolites can not only enhance their adsorption capacity but also play a role in stabilizing the structural integrity of zeolites in long-term processes. In spite of these merits, the leaching of structural components and crystalline changes require attention for effective performance [5]. Comprehending the aforementioned modifications is essential to fill the gap and design zeolite-based adsorbents with superior efficiency for sustainable water purification.

2. MATERIALS AND METHODS

The natural clinoptilolite zeolite (particle size: 0.7... 1 mm) used in this study was obtained from the Shanganai deposit (Zhetysu region, Kazakhstan).

The method of acid treatment of clinoptilolite has been adapted based on previously published studies [5...13]. Dirty zeolite was pre-ultrasonicated (Fisherbrand FB11201). Pre-ultrasonic treatment was used to remove surface contaminants, dust particles, and organic impurities. Then microwaved before being rinsed multiple times with deionized (DI) water to remove obvious loose debris. Washing with deionized water was carried out until the filtrate became transparent, which indicated the removal of free powdery and mechanical particles. The absence of impurities was additionally controlled by the neutral pH value of the rinsing water.

The zeolite was also treated with a NaCl solution for comparative purposes. The treatment conditions with NaCl (1 M, 90 °C, 12 h) were selected to be identical to those of the acid treatment in order to ensure a correct comparison of the reagents under the same regime.

The purified zeolite was placed in an oven at 100°C for 24 h of additional treatment. The process of modification was performed using hydrochloric acid (Hach ACS grade). A total of 20 g pre-treated zeolite was refluxed in a three-neck boiling flask with 115 mL deionized water and 10 mL concentrated HCl at around 90 °C for 12 h. A commonly used acid in this process was 0.97 M HCl. The concentration of 0.97 M was selected based on preliminary data and published studies [6...13], which showed that at concentrations above 1.0 M it can lead to a decrease in the ability to remove Pb^{2+} ions, and at concentrations below 0.5 M the effect of modification is weak. Thus, 0.97 M provided a balance between impurity removal efficiency and maintaining the structural integrity of the zeolite.

All reflux setups contained a Vigreux column, a thermometer, and foil to prevent evaporation. After the acid treatment, the zeolite was rinsed with absolute ethanol (Fisher Chemical denatured), followed by extensive washing with DI water.

Table 1

The raw zeolite chemical composition

Component	Percentage (%)
SiO ₂	68.26 %
Al ₂ O ₃	12.99 %
K ₂ O	4.11 %
CaO	2.09 %
Fe ₂ O ₃	1.37 %
MgO	0.83 %
Na ₂ O	0.64 %
TiO ₂	0.23 %

The end product was then placed into an oven (100 °C for 24 h). ATR table and Fourier Transform Infrared Spectroscopy (FTIR) were conducted with a Shimadzu IR Tracer 100. All the samples were ground into a fine powder prior to analysis. Subsequently, the surface area, pore volume, and pore diameter were determined by using a Quanta chrome Autosorb IQ2 gas sorption analyzer after degassing at 350 °C for approximately 17 h. The thermal constancy was estimated to be using the Difference Thermal Thermohydrometric (DTG) assessment by Shimadzu DTG-60 analyzer with dry grade airflow in alumina pans up to 600 °C.

The X-ray phase analysis of primary and activated samples from zeolite, which were carried out by copper cathode tubes (nickel filter) on diffractometer DRON-3M at an accelerator voltage of 35 kV. Shooting on speed 2/min, at segment measures from 5 to 50 °C The samples were cut to powder form and burnt in a glass cube Vaseline oil.

Lyophilization of all prepared zeolites was carried out according to the standard procedure: where then adsorption of the modified zeolites was further assessed in column mode experiments. Disperse Blue 26 (DB 26) (Crescent Chemical, ASC12972019), a weakly cationic model compound, was used to evaluate adsorption efficiency. Experiments were performed in glass columns (length = 21 cm, internal diameter = 2 cm) filled with the modified zeolite to a height of 13 cm; a gauze filter was placed at the bottom. Before adsorption, the media were rinsed with 1 L of degassed DI water using a peristaltic chemical metering pump. Then, a 1 mL solution of 40 mg/L DB 26 was passed through the column with a flow rate of 5 mL/min. Before adsorption experiments, the concentration 0.1 M NaOH solution was used to bring the pH of the aqueous zeolite suspension to a neutral level (≈ 7.0). A similar approach was used in early studies on the preparation of zeolite adsorbents [14]. This concentration ensures sufficient control accuracy without the risk of excessive pH changes.

3. RESULTS AND DISCUSSION

Gas sorption analysis was used to study the effects of hydrochloric acid (HCl) and sodium chloride (NaCl) treatments on physicochemical and structural properties of the clinoptilolite.

Table 2

Showing the comparison of the BET surface area of HCl-treated, raw, and NaCl-treated zeolite.

Sample	BET Surface Area (m ² /g)	Pore Diameter (Å)
Raw Zeolite	170.5	18...20
HCl-Treated Zeolite	280.3	12...15
NaCl-Treated Zeolite	185.2	16...18

The results indicate that HCl treatment significantly increased the BET surface area (64.4% increase) compared to the raw zeolite, while NaCl treatment had only a moderate effect. The

decrease in pore diameter for HCl-treated samples suggests improved adsorption efficiency, likely due to impurity removal and framework reorganization.

Also, the surface areas and porosities are important properties that provide information about the changes caused by the treatments (Fig. 1). At the same time, the BET surface area of the sample treated with HCl (280.3 m²/g) was the largest among the other two samples, indicating no significant change, and NaCl did not enhance the surface area. The observed improvement indicates a drastic rearrangement of the zeolite structure, apparently leading to the elimination of impurities and a partial disaggregation of the aluminosilicate framework [15].

In contrast, the clinoptilolite NaCl sample showed moderate surface area increment with pore structure remaining stable in comparison to clinoptilolite HCl. The pore volumes obtained through this analysis show a marginal decrease in all treated samples, where HCl treatment has the strongest effect. This is possibly due to partial framework collapse and pores narrowing as a result of acid leaching [16]. In contrast, the NaCl treatment predominantly impacted on ion-exchange properties, without significant modifications to the pore architecture. The analysis of pore size distribution showed that HCl treatment reduced the pore diameter in a range of 12...15 Å, while NaCl treatment produced minor changes in pore size distribution, confirming its action as a modifier and not a restructuring agent [17].

These findings corroborate recent work on the effect of acid or salted modifications to zeolites. In overall, clinoptilolite is significantly modified with structural and textural characteristics when treated with HCl, with external area improving and absorbency reducing due to structure distortion. On the other hand, NaCl is a treatment that develops ion exchange without concerning the structural reliability of the material, thus it is more appropriate for applications that need to preserve stable porosity while improving ion-exchange capability.

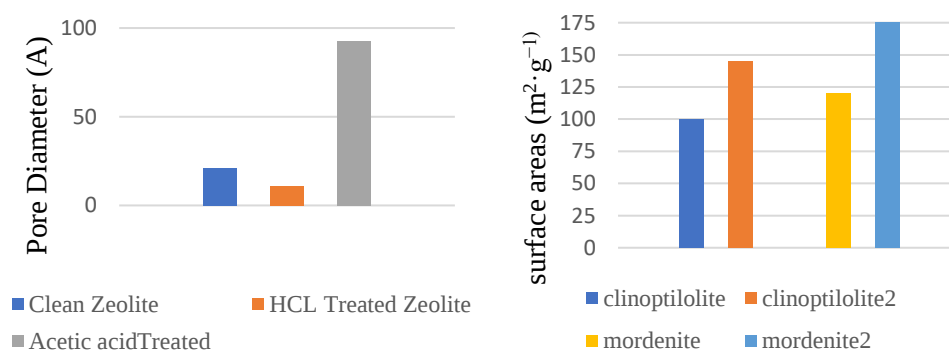


Figure. 1. In this figure (A) Surface area of zeolites and (B) the pore diameter. and HCl utilize the most Important modification to the physical features of the zeolite

A comparative study showing that treatment of zeolites with hydrochloric acid (HCl) resulted in a significant decrease in surface area. Possibly attributed to variations in either the acid treatment method or hydrochloric acid concentration used. The results indicate that the surface properties are strongly modified by reflux of the zeolite in hydrochloric acid. On the other hand, the untreated zeolites and the zeolites modified with phosphoric acid or citric acid presented relatively larger surface areas, but the difference was not significant in these treated samples [15].

Acid treatment may also fill smaller pores, which would prevent this reaction from occurring in the smaller pores and only in the larger ones. In polar solvents, it has been observed that HCl etching had little effect on the BET surface area and pore volume of synthetic ZSM-5 zeolites at 363 K [5]. Considering that the acid concentrations and treatment temperatures were very similar, the zeolite type and the treatment time were the only relevant differences. This implies that zeolite type-dependency needs to be accounted for in consideration of the use of acid to increase the zeolite surface area. These new findings, however, emphasize the need to adapt acid treatment procedures to the relevant zeolite architecture, allowing to improve the adsorption properties and surface properties of zeolites as a material for environmental remediation purposes [13].

The XRD analysis in (Fig.2) shows that there is a minor shift at around $2\theta = 26.5^\circ$ (characteristic of quartz) toward smaller values for the HCl leached clinoptilolite samples. Peaks at 22.3° and 30° are assigned to the clinoptilolite shape. Hydrochloric acid considerably influenced the crystallinity of clinoptilolite, assessed to untreated zeolite, and had a noticeable influence on the peak concentrations. This implies that HCl treatment affects zeolite construction more than other ineffective acids, such as acetic acid. Adsorption is commonly connected to surface area and chemistry; hence, the enhanced crystallinity imparted by the HCl treatment may cause a decrease in absorbency and lead to less defects, a smaller number of chemical places with lower energy, and thus weaker adsorption properties. At 22.5° , the maximum intensity decline is possibly related to lamellar deformation, which leads to partial structural changes.

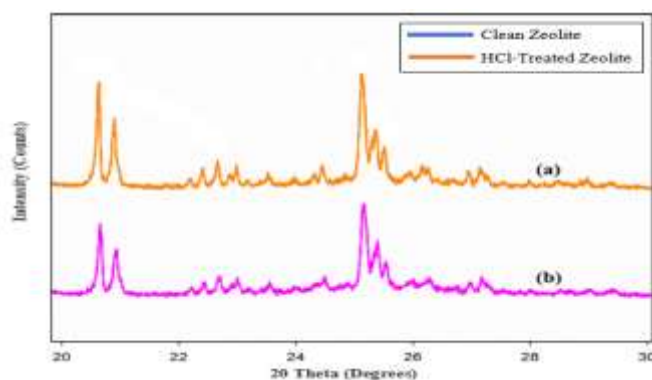


Figure. 2. XRD patterns of zeolites. Which shows the most intensity of HCl-treated Zeolite than the clean Zeolite

Decomposition of the structure while shifting is associated with a decrease in the interlayer region due to metals removed after acid leaching. Additionally, a significant change was observed in the quartz, mostly for hydrochloric acid. This is expected as hydrochloric acid is extensively recognized for its sanitization properties of quartz.

We used thermogravimetric analysis (TGA) to investigate the thermal decomposition behavior of clean and HCl-treated zeolite in our experiment. As seen in (Fig. 3), the mass loss trends, the loss of weight was greater on the HCl-treated zeolite than the air-activated zeolite at low temperatures (below 150°C), representing the loss of physically adsorbed water or volatile. Compared to clean zeolite that has a faster mass loss throughout the whole temperature spectrum, suggesting a stiffer framework. The mass loss observed after 200°C for zeolite treated with HCl suggests structural configuration modifications attributed to partial dealumination leading to an increased silica-to-alumina ratio. Besides, due to the different porosity and pore properties, its adsorption law also varies. The results described above indicate that the structure, thermal, and Adsorption properties of zeolite are successively modified by acid treatment, which is consistent with other zeolite modification studies.

Optical microscopy indicates the morphological changes caused by HCl modification of natural zeolite. As shown in (Fig. 1), the untreated zeolite exhibits a relatively rough and heterogeneous surface with irregularly shaped particles and visible agglomerations. The overall texture suggests a porous structure beneficial for adsorption applications.

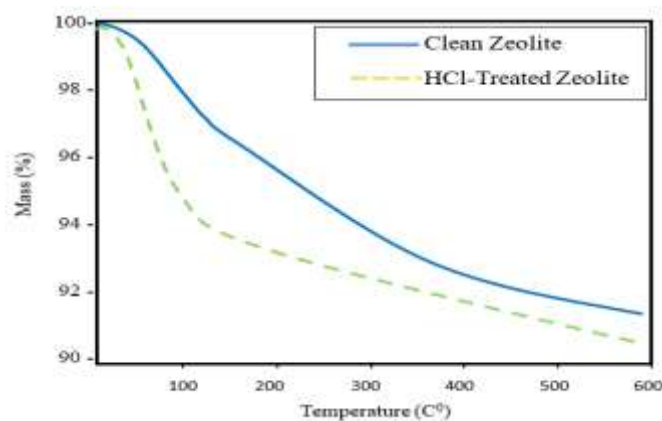


Figure. 3. Information from DTG showing the amount of mass lost as the treated materials reach 600 °C. There is essentially no change in thermal stability

A major change in surface appearance is observed after HCl treatment. Thus, acid treatment is beneficial for obtaining a cleaner surface, eliminating amorphous phases and other impurities. These findings are in line with reported literature, where acid leaching improves the textural characteristics of zeolites by increasing the number of available adsorption sites [5...15].

In addition, the treated zeolite shows a clearer particle arrangement, suggesting better accessibility of adsorption sites [8]. However, excessive acid exposure and repeated washing can cause partial structural degradation, lowering the overall crystallinity of the material [18].

HCl-modified zeolite demonstrates improved surface characteristics that are advantageous in water purification, as the removal of non-framework components (mainly metal oxides) increases the ion-exchange capacity, thereby facilitating contaminant removal. Overall, optical microscopy analysis supports that HCl modification enhances the morphology of natural zeolite, producing a cleaner structure suitable as an adsorbent medium in water purification.

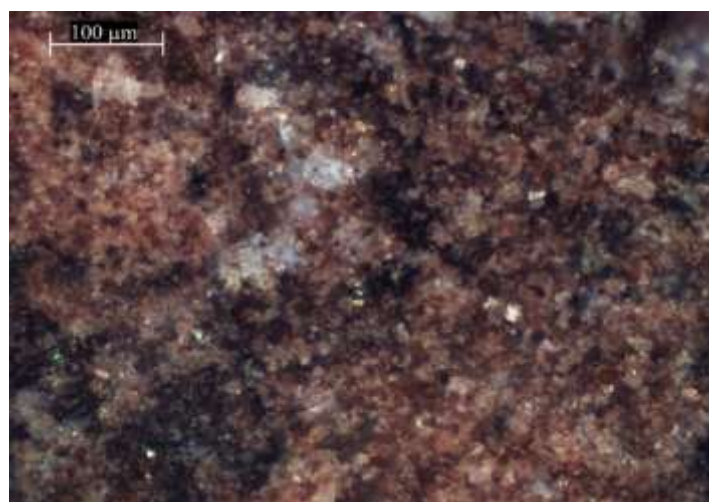


Figure. 4. The Optical microscopy image of the Surface morphology of the treated zeolite

A comparative study showing that treatment of zeolites with hydrochloric acid (HCl) resulted in a significant decrease in surface area. Possibly attributed to variations in either the acid treatment method or hydrochloric acid concentration used. The results indicate that the surface properties are strongly modified by reflux of the zeolite in hydrochloric acid. On the other hand, the untreated zeolites and the zeolites modified with phosphoric acid or citric acid presented relatively larger surface areas, but the difference was not significant in these treated samples [15].

Moreover, these findings are consistent with those observed in treatment with glacial acetic acid. Acetic acid does not seem to have any effect on the modification of zeolite. Acid treatment may also fill smaller pores, which would prevent this reaction from occurring in the smaller pores

and only in the larger ones. In polar solvents, it has been observed that HCl etching had little effect on the BET surface area and pore volume of synthetic ZSM-5 zeolites at 363 K [5]. Considering that the acid concentrations and treatment temperatures were very similar, the zeolite type and the treatment time were the only relevant differences. This implies that zeolite type-dependency needs to be accounted for in consideration of the use of acid to increase the zeolite surface area. These new findings, however, emphasize the need to adapt acid treatment procedures to the relevant zeolite architecture, allowing to improve the adsorption properties and surface properties of zeolites as a material for environmental remediation purposes [13].

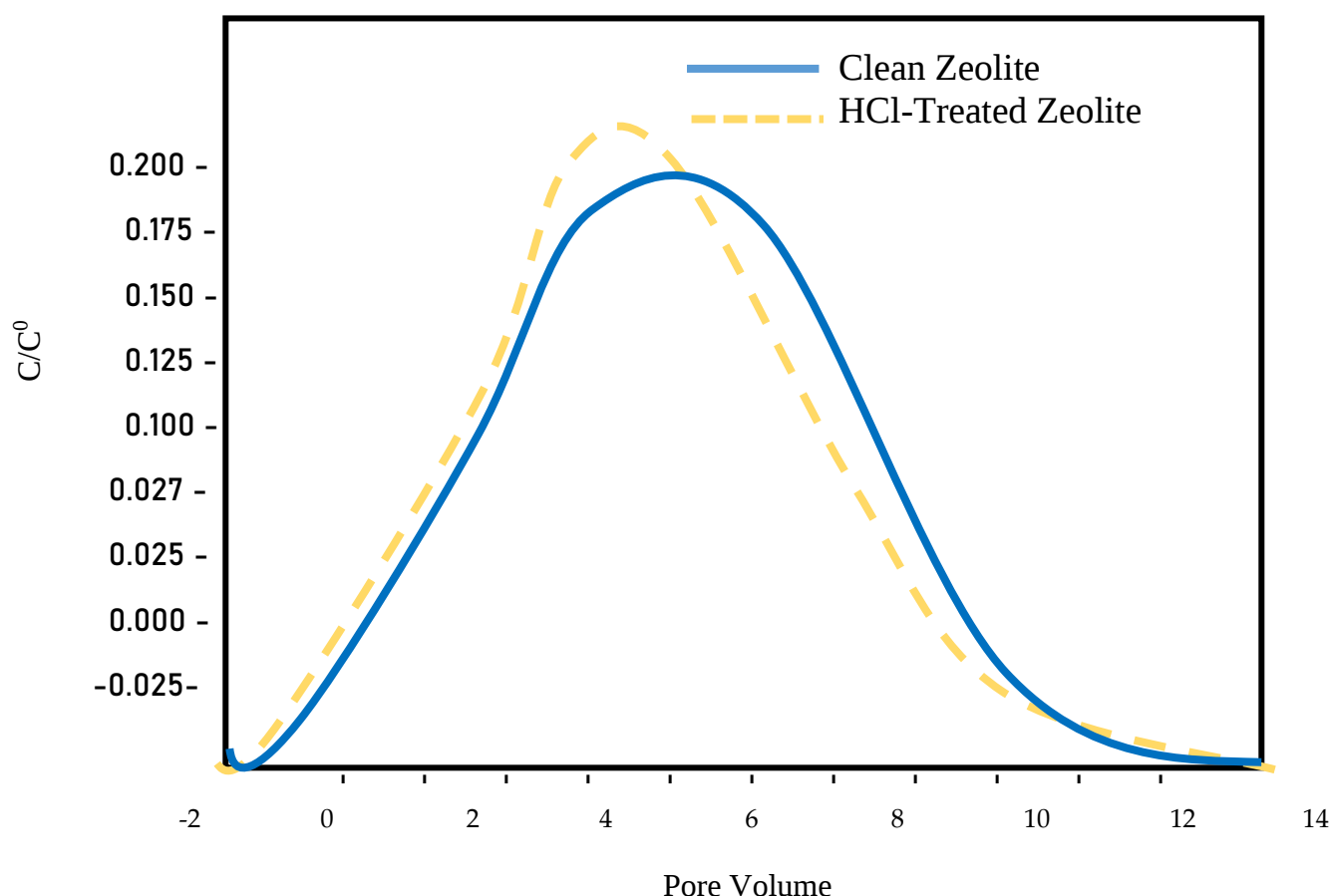


Figure 5. Breakthrough curve for adsorption of DB26 in column mode experiment

The breakthrough curves of the graph show the effect of the treatment of zeolite (HCl) on its pore volume and adsorption behavior. The overall trends for the clean zeolite and the HCl-treated zeolite are similar; however, the curve for the HCl-treated zeolite peaks slightly earlier at a lower pore volume, indicating that acidity treatment altered the pore structure. This shift suggests that treating with HCl may have changed the accessibility of adsorption sites, improved the rate of diffusion, yet decreased the overall available sites for adsorption. However, the lower tailing effects indicate a more homogeneous adsorption process in the acid-modified zeolite of the present study, which is consistent with previous reports where strong acids such as sulfuric and nitric acid were shown to enhance the adsorption performance of zeolites even more effectively than HCl [19...20].

4. CONCLUSION

This study demonstrates that acid treatment, particularly with HCl, modifies the structure of natural zeolites and improves their adsorption efficiency. Zeolites, due to their high surface area, tunable porosity, and ion-exchange capacity, remain highly promising materials for water purification and environmental applications. The results highlight the potential of HCl-modified

zeolites as effective adsorbents, while future work should include comparative studies with other strong acids and real wastewater samples to fully evaluate their long-term applicability.

Although acid treatment improves adsorption properties, its effect on long-term structural stability remains controversial. Some studies report stabilization of zeolite frameworks after acid leaching [13], while others note possible framework degradation. Therefore, further investigation of durability and reusability is required before confirming long-term stability.

The results were in line with prior research, suggesting modified zeolites as a preferred component in sustainable water treatment options. Further investigation is required to improve the modification conditions in order to strike a reasonable balance between the properties of structure reliability and high adsorption performance. Consideration of durable strength and reusability of modified zeolites will be critical for their practical application in water treatment as well as their environmental applications. Investigating zeolite synthesis and functionalization will improve our efficacy and cost-efficiency in resolving the trouble of global water contamination.

DATA AVAILABILITY

The data used in this study were obtained by the authors from public and paid sources.

AUTHORS' CONTRIBUTION

Conceptualization - YeD, MZ, AS, AK; resources - NA, DA, RI; formal analysis - AZh, KS; methodology - YeD, MZ; software - NB, AA; supervision - OD; visualization - AS, AK; writing - original draft preparation - MZ, YeD; writing - review and editing - AZh.

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АУЫЗ СУДЫ ТАЗARTY YШIN TYZ KYШKЫЛЫМЕН ӨНДЕЛГЕН ЦЕОЛИТТИҢ ИОН АЛМАСУ ҚАСИЕТТЕРІН ЗЕРТТЕУ

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ТҮЙІН СӨЗДЕР

қышқылмен өңдеу,
цеолитті модификациялау,
адсорбция,
суды тазарту,
кеуек құрылымы,
клиноптилолит.

АБСТРАКТ

Цеолит – ерекше адсорбциялық, ион алмасу және катализикалық қасиеттерімен танымал микрокеуекті кристалды алюмосиликат материалдар тобы. Олар ағынды суды тазартуда, әсіресе ауыр металдар мен аммоний иондарын жоюда кеңінен қолданылады. Бұл зерттеу табиғи клиноптилолит цеолитінің физика-химиялық және құрылымдық қасиеттеріне HCl (тұз қышқылы) өңдеудің әсерін зерттейді. Бірінші кезеңде 0.97 М HCl ерітіндісімен қайнату арқылы қышқылмен өңдеу жүргізілді, содан кейін ол мұқият тазартылып, FTIR, XRD, газ адсорбциясы және DTG әдістерімен сипатталды. Алынған нәтижелер бастапқы материалмен салыстырғанда HCl өңдеуі цеолиттің меншікті бетін 64.4%-ға (BET әдісі бойынша 280.3 м²/г дейін) ұлғайтатынын, катион алмасу қабілетін арттыратынын және алюмосиликат құрылымдық бірліктерін қайта ұйымдастыру арқылы қоспаларды жою арқылы кеуек құрылымын жақсартатынын көрсетеді. Газ адсорбциясы кеуек диаметрінің төмендеуі есебінен адсорбция тиімділігінің артқанын растады. Сонымен қатар, адсорбция эксперименттері HCl өңделген цеолиттердің Disperse Blue 26 (DB 26) бояғышын кетіруде өңделмеген үлгілерге қарағанда жақсы нәтиже көрсеткенін дәлелдеді. Дегенмен, қышқылмен өңдеу адсорбциялық қасиеттерді жақсартқанымен, құрылымдық тұрақтылықтың бұзылу қаупі болуы мүмкін екені анықталды. Бұл зерттеу нәтижелері HCl модификацияланған цеолиттердің суды, соның ішінде ағынды суларды тазарту үшін тиімді және экологиялық таза адсорбенттер ретіндегі әлеуетін көрсетеді.

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ИССЛЕДОВАНИЕ ИОНООБМЕННЫХ СВОЙСТВ ЦЕОЛИТА, МОДИФИЦИРОВАННОГО СОЛЯНОЙ КИСЛОТОЙ, ДЛЯ ОЧИСТКИ ВОДЫ

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КЛЮЧЕВЫЕ СЛОВА

кислотная обработка,
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адсорбция,
очистка вод,
пористая структура,
клиноптилолит.

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АБСТРАКТ

Цеолит – это группа микропористых кристаллических алюмосиликатных материалов, которые привлекли значительное внимание благодаря своим выдающимся сорбционным, ионообменным и каталитическим свойствам. Их применение в очистке сточных вод, особенно для удаления тяжелых металлов и ионов аммония, значительно расширилось благодаря химическим модификациям. В данном исследовании изучается влияние обработки соляной кислотой (HCl) на физико-химические и структурные свойства природного клиноптилолитового цеолита. Сначала его подвергли рециркуляции с 0.97 М HCl для кислотной модификации, затем тщательно очистили и охарактеризовали с помощью FTIR, XRD, газовой сорбции и DTG. В целом, по сравнению с исходным материалом, результаты ясно показывают, что обработка HCl увеличивает удельную поверхность и катионообменную способность, а также улучшает распределение пор за счет удаления примесей и реорганизации алюмосиликатных структурных единиц. Газовая сорбция подтвердила увеличение удельной поверхности по BET на 64.4 % (до 280.3 м²/г) при одновременном уменьшении диаметра пор, что свидетельствует о повышенной эффективности сорбции. Эксперименты по адсорбции также показали, что большинство цеолитов, обработанных HCl, превосходили немодифицированные образцы в удалении красителя Disperse Blue 26 (DB 26). Кроме того, хотя кислотная модификация часто улучшает адсорбционные свойства, исследование указывает на возможные проблемы со структурной стабильностью из-за выщелачивания каркаса. Результаты данного исследования подчеркивают потенциал цеолитов, модифицированных HCl, как эффективных и экологически чистых адсорбентов для очистки вод, в том числе сточных.

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