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Influence of process parameters in ion exchange on the properties of the obtained 5A zeolite powder

ABSTRACT

Zeolite5A is indeed highly regarded in the field of adsorption among zeolites, which makes it valuable for various commercial applications. It is most commonly obtained from 4Azeolite, where Na^+ ions in the crystal lattice of 4A zeoliteare replaced with Ca^{2+} or Mg^{2+} ions. This ion exchange process frees upmore space within the crystal pores, increases pore openings and enhances the adsorption capacity of the zeolite. From an industrial andcommercial production perspective, it is crucial to define the optimal process parameters for the production of 5A zeolite. This study examined the effects of theion exchange temperature, duration of ion exchange, and concentration of active components in the system on the characteristics of the obtained5A zeolite powder. The ion exchange was carried out using asolution of $MgCl_2{\cdot}6H2O$, resulting inthe Mg^{2+}form of5Azeolite,i.e. Na, Mg-A zeolite. The powders obtained after ion exchange were analyzed for their chemical composition and water adsorption capacity. In addition, the samples were further characterized through granulometry and particle size distribution analysis, X-ray diffraction, as well as SEM and FT-IR analysis.The results indicate dependencies between the process parameters of the system, the percentage of exchanged ions, crystallinity, and the adsorption properties of the material, which enables the determiningof the optimal conditions for 5A zeolite production.

Keywords: *5A, adsorption, ion exchange, process parameters, zeolite*

1. INTRODUCTION

The three-dimensional structure of zeolite is built of $[TO_4]$ tetrahedra where T represents Si or Al atom, or, in rare cases, another atom,depending on the type of material. In general, the chemical composition of zeolite can be presented by the formula $M_{x/n}[(Al_2O_3)_x{\cdot}(SiO_2)_y)]{\cdot}mH_2O$,where the negatively charged aluminosilicate framework is neutralized by the cation M with chargen, mrepresents the number of water molecules per unit cell, and x and y denote the total number of tetrahedra per cell [1-4].

Observing the tetrahedralgroups of Si and Al, it can be seen that the aluminum tetrahedral groups, $[AlO_4]^-$,is negatively charged. This necessitatesthe presence of cations within the zeolite crystal lattice to compensate for negative charge. Cationsare not directly fixed within the crystal latticestructure and can diffuse through it. As a result, they can be replaced by other cations throughthe process of ion exchange. The number of compensating cations is directly proportional to the proportion of $[AlO_4]$ tetrahedral groups in the composition of the crystal lattice. Therefore, zeolites with higher aluminum content also contain a greaterproportion of compensatingcations, resulting in more pronounced ion-exchange characteristics[5-7].

Zeolite A (with an LTA crystal structure) is one of the zeolites with the highest aluminum content, featuring a very low Si/Al molar ratio of approximately 1. This makes zeolites of this type particularly interesting for ion exchange applications. Combined with its other properties, this characteristic is why zeolite A has been one of the most commercially prevalent zeolite types on the market for decades [8,9].

Zeolite type A is most commonly synthesized in its sodium form, known as NaA or 4A zeolite. The regular cavities in the 4A zeolite structure have openings approximately 4 angstroms (4Å = 0.4 nm) in diameter, which is reflected in the designation. Through ion exchange, Na^+ ions can be replaced

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with other ions of suitable characteristics, thereby allowing the modification of the properties of A-type zeolite powders to meet specific application needs. The replacement of ions also changes the dimensions of the cavity openings in the crystal structure, which in turn alters the material designation. For example, replacing Na^+ ions with larger K^+ ions reducesthe cavity openings to approximately 3 angstroms (3A zeolite) [7,10]. In contrast, replacing sodium with divalent cations like Ca^{2+} or Mg^{2+} increases the cavity openings to 5 angstroms, and these zeolites are thus labeled as 5A zeolites, or Na,Ca-A and Na,Mg-A, respectively, since not all sodium is usually replaced [3,4,11,12].

Due to the larger cavity openings in the crystal, 5A zeolites exhibit slightly lower selectivity in comparison to 4A zeolites. However, they possess a significantly higher adsorption capacity and can adsorb larger molecules with diameters up to 0.5 nm. These characteristics make them suitablefor various adsorption and separation processes, such as water extraction from solutions and gases, separation of n- and iso-paraffins, CO_2 and H_2S adsorption from natural gas, nitrogen adsorption, and the production of concentrated N_2 and O_2 gases, among others [3-5,12-15].

Given the extensive commercial applicability of this material, defining the optimal conditions for producing 5A zeolites can be highly significant. This study aims to determine the effects of ion exchange temperature, ion exchange time, and the concentration of the ion exchange solution on the properties of the resulting 5A zeolite powders, using an ion exchange system containing $MgCl_2$ as the starting compound for the ion exchange process, in contrast to the more commonly studied calcium compounds. $MgCl_2$ was selected as the raw material due to the presence of one of the smallest divalent cations, which is expected to maximize the space within the crystal after ion exchange [3].

2. EXPERIMENTAL

The investigation into the effects of various process parameters during ion exchange on the properties of the resulting 5A zeolite powders was conducted at the Research Laboratory of Alumina as part of a broader study on the ion exchange process. This study presents 10 planned ion exchanges, with 4 ion exchanges each being used to examine the impact of different process parameters: the concentration of Mg^{2+} in the ion exchange solution, the suspension temperature during ion exchange, and the duration of the ion exchange. All other parameters, except those specifically investigated in this study, were kept constant. The relevant experimental conditions are listed in Table 1, along with the results of the analyses.

The ion exchanges were performed by preparing a suspension of 4A zeolite in a $MgCl_2$ solution, which was then mixed under the specified conditions. The volume of the suspension for each ion exchange was 250 mL, with the concentration of the solid phase 4A zeolite in the suspension being 210 g/L. The concentration of $MgCl_2$ in the liquid phase of the suspension was varied according to the experimental conditions. The obtained suspension was mixed at the specified temperature for the designated period as per the experimental conditions. The 4A zeolite used was from Alumina's standard production, intended for commercial use as a molecular sieve with high water adsorption capacity, and is labeled as 4A-MS zeolite. The $MgCl_2$ solution was prepared by dissolving $MgCl_2{\cdot}6H_2O$ in demineralized water. The $MgCl_2{\cdot}6H_2O$ used for the experiment was purchased from *Himedia*(India) and was of analytical grade purity.

After the ion exchange, the suspension was filtered, and the resulting cake was washed, dried, and then analyzed. The obtained powders were analyzed for Na_2O and MgO content to determine the percentage of ion exchange, average particle diameter, and water adsorption capacity, a crucial quality characteristic for 5A zeolite. Additionally, the powders were further characterized using X-ray diffraction and IR spectroscopy, and examined with electron microscopy to investigate the impact of the ion exchange process on the morphology of the resulting 5A zeolite. All analyses, except for scanning electron microscopy, were performed at *Alumina ResearchLaboratory*.

Chemical composition analysis of the zeolite and the determination of Na_2O and MgO content were conducted using an atomic absorption spectrometer Shimadzu AA-7000. The determination of Na_2O content in the zeolite samples was perfomed according to the standard method*Na-MA.ZIB-P.012*, while the determination of MgO content was carried out according to the standard method*Mg-MA.ZIB-P.024*.

Particle size distribution and average diameter (d_{50}%) were determined using a CILAS 1090 liquid particle size analyzer, with a range of 0.02 to 500 μm, and 30 seconds of ultrasonic sonication, following the internal particle size distribution method, MA.GX 032a.

X-ray diffraction (XRD) analysis and determination of crystallinity were performed using a Bruker D8 ENDEAVOR diffractometer with a cobalt anode X-ray tube, employing CoKα radiation with a wavelength of λ=1.78897Å.

Water adsorption capacity (WAC) was measured using a Kambič KK-105-CH climate chamber at 20°C, relative humidity rH = 50±2, for 24 hours, according to the internal method *Determination of the water adsorption capacity of zeolite*. The method requires prior sample preparation through thermal activation in an oven at t=350-550˚C.

Zeolite crystal imaging using scanning electron microscopy (SEM method) was conducted at the Faculty of Technology and Metallurgy in Belgrade using a MIRA3 TESCAN device with magnifications up to 10000 times. SEM analysis of zeolite samples was carried out according to the standard method BAS ISO 1287.

FTIR(eng. *Fourier Transform Infrared spectroscopy*) analysis was performed on a Shimadzu IRAffinity-1S spectrophotometer with a MIRacle 10 ATR accessory at *Alumina ResearchLaboratory*. FTIR analysis of zeolite samples was perfomed according to the standard method BAS ISO 2345.

Table 1. Investigated process parameters of ion exchange and analysis results of the obtained zeolite powders after ion exchange

Synthesis number	T	τ	m/m	Na_2O_{4A}	D50	Na_2O	MgO	Exchange with Na	XRD	WAC
	°C	min		%	[µm]	[%]	[%]	[%]	[%]	[%]
5A1	80	120	0.2	19.52	4.05	11.26	6.52	42.32	98.75	28.66
5A2	80	120	0.3	19.52	4.05	9.35	7.97	52.10	97.58	29.51
5A3	80	120	0.57	19.52	4.13	8.63	9.42	55.79	96.92	30.18
5A4	80	120	1	19.52	4.22	7.65	10.82	60.81	94.61	29.37
5A5	70	120	0.3	19.52	3.96	9.53	7.49	51.18	96.97	29.42
5A6	60	120	0.3	19.52	3.81	9.97	6.92	48.92	98.22	29.21
5A7	50	120	0.3	19.52	4.01	10.75	6.01	44.93	98.59	28.86
5A8	80	30	0.3	19.52	3.92	10.88	5.77	44.26	98.91	28.74
5A9	80	60	0.3	19.52	3.91	9.42	6.93	51.74	98.17	29.32
5A10	80	180	0.3	19.52	3.90	8.87	7.71	54.56	96.11	29.28

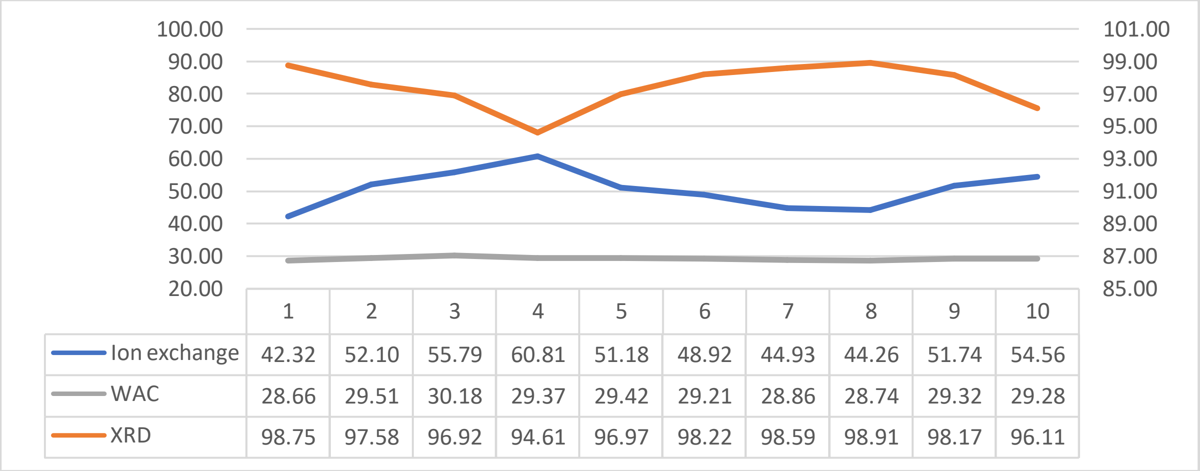
3. RESULTS AND DISCUSSION

The analysis results of the sample analysis from all the displayed ion exchanges showed a significant proportion of Mg^{2+} ions, indicating a significant percentage of ion exchange. Thus, for practically all samples, it can be said that a 5A zeolite was obtained, and the ion exchanges were relatively successful. Therefore, it is even more interesting to observe the dependencies and differences in the quality of the obtained powders, as these details are crucial for optimizing the ion exchange process.

Examining the results of the analysis of 5A zeolite powders obtained from varying concentrations of $MgCl_2$ in the ion-exchange system, we observe that increasing the concentration of Mg^{2+} ions in the system significantly effects the percentage of exchanged ions. This is expected because as the concentration of Mg^{2+}ions in the solution increases, the equilibrium of the ion exchange reaction shifts towards the formation of 5A zeolite, which is richer in magnesium. The reaction accelerates, and the diffusion of ions through the crystal lattice is enhanced.

It is also noticeable that increasing the amount of $MgCl_2$ from 0.2 to 0.3 g/g of 4A zeolite increased the percentage of ion exchange from 42.32% to 52.10%. On the other hand, increasing the amount to 1 g/g of 4A zeolite, an increase of five times, resulted in an ion exchange percentage of 60.81%. In other words, increasing the concentration, i.e. the amount of Mg^{2+} ions in the solution relative to the amount of zeolite, promotes ion exchange, but the efficiency of the process decreases. As the percentage of exchanged ions within the zeolite increases, the concentration of Na^+ ions in the ion-exchange solution also rises, which slows down the ion exchange reaction and hinders the diffusion of new magnesium ions that need to find their place in the zeolite crystal. Therefore, a substantial increase in the amount of $MgCl_2$ is necessary to achieve the kinetics of the ion exchange reaction for higher percentages of exchanged ions.

Furthermore, it can be observed that as the percentage of ion exchange increases from 42.32% to 60.81%, the water adsorption capacity showed an increase from 28.66% at 42.32% ion exchange to 30.18% at 55.79% ion exchange, and then decreases to 29.37% at 60.81% ion exchange. These results indicate that the sorption capacity of 5A zeolite increases with the number of exchanged ions, but only up to a certain upper limit, after which the increase stops or even declines.



	1	2	3	4	5	6	7	8	9	10
Ion exchange	42.32	52.10	55.79	60.81	51.18	48.92	44.93	44.26	51.74	54.56
WAC	28.66	29.51	30.18	29.37	29.42	29.21	28.86	28.74	29.32	29.28
XRD	98.75	97.58	96.92	94.61	96.97	98.22	98.59	98.91	98.17	96.11

Figure 1. Comparative trend of changes in water adsorption capacity (WAC) and percentage of ion exchange

Comparing the results of powders obtained at different ion exchange temperatures, it can be concluded that a temperature of 50°C is significantly less favorable for the ion exchange process than the higher tested temperatures. Conversely, samples exchanged at temperatures of 60, 70, and 80°C had similar ion exchange percentages, suggesting that within this temperature range, the effect of temperature on ion exchange was minimal. There is a slight increase in the percentage of exchange with rising temperature. These values were obtained for an ion exchange duration of 2 hours, and it is possible that with a shorter ion exchange time, the relationship between the percentage of achieved exchange and temperature might be different.

The sample with the lowest percentage of exchange also has the lowest adsorption capacity, below 29%. The other three samples have similar WAC values with a slight increase corresponding to the increase in temperature, which aligns with the ion exchange percentages for these samples.

Samples with ion exchange performed over different durations showed a similar trend to that observed with varying temperatures during ion exchange. The shortest duration of 30 minutes resulted in the lowest percentage of ion exchange, while the values for the other three samples were significantly closer to each other, with the change from 60 to 180 minutes being much less significant. This indicates that the system approached an equilibrium state after just 60 minutes of ion exchangeat the given concentrations.

Here as well, the sample with the lowest percentage of ion exchange has the lowest water adsorption capacity, below 29%, while the water adsorption capacities for the other samples are quite similar, which again corresponds to the similar ion exchange percentages of these samples. It can be noted that the sample with the highest percentage of ion exchange, obtained after 180 minutes of ion exchange, has a slightly lower water adsorption capacity compared to samples exchanged for 60 and 120 minutes. This may be related to a similar observation with samples where ion exchange was performed with solutions of different $MgCl_2$ concentrations, where the sample with the highest percentage of exchange showed a decrease in WAC. This phenomenon might be related to changes in the crystallinity of the material during ion exchange. Figure 1 illustrates the trends in water adsorption capacity (WAC) and ion exchange percentage for all the samples.

The XRD analysis results for samples obtained through ion exchange from solutions with different concentration of $MgCl_2$ are shown in Table 1 and Figure 2. As the proportion of Mg^{2+} ions increases, it is likely that changes in crystal lattice occur, which XRD detects as changes in the intensity of characteristic peaks on the diffractogram. Comparison of the diffractograms of 5A zeolites with the initial 4A form sample shows that some characteristic peaks are lower for the 5A zeolite samples, while the intensity of some peaks has increased. However, the calculated values of crystallinity show that, although relatively high crystallinity is retained, there is a decrease in material crystallinity with an increasing percentage of ion exchange, with the lowest crystallinity value recorded for the sample with the highest percentage of exchanged ions. This may also explain why this sample shows a decrease in water adsorption capacity relative to the overall trend observed in this series of samples.

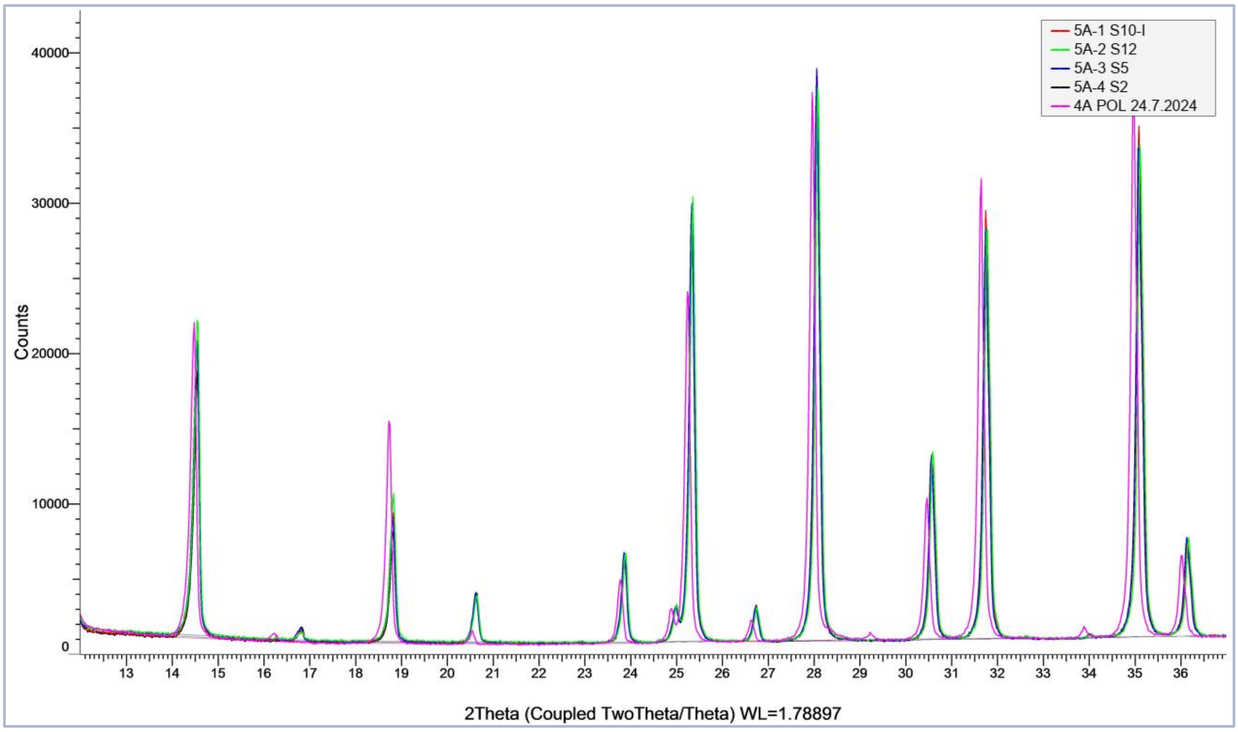


Figure 2.Diffractograms of 5A zeolite powders obtained through ion exchange from solutions with different $MgCl_2$ concentrations

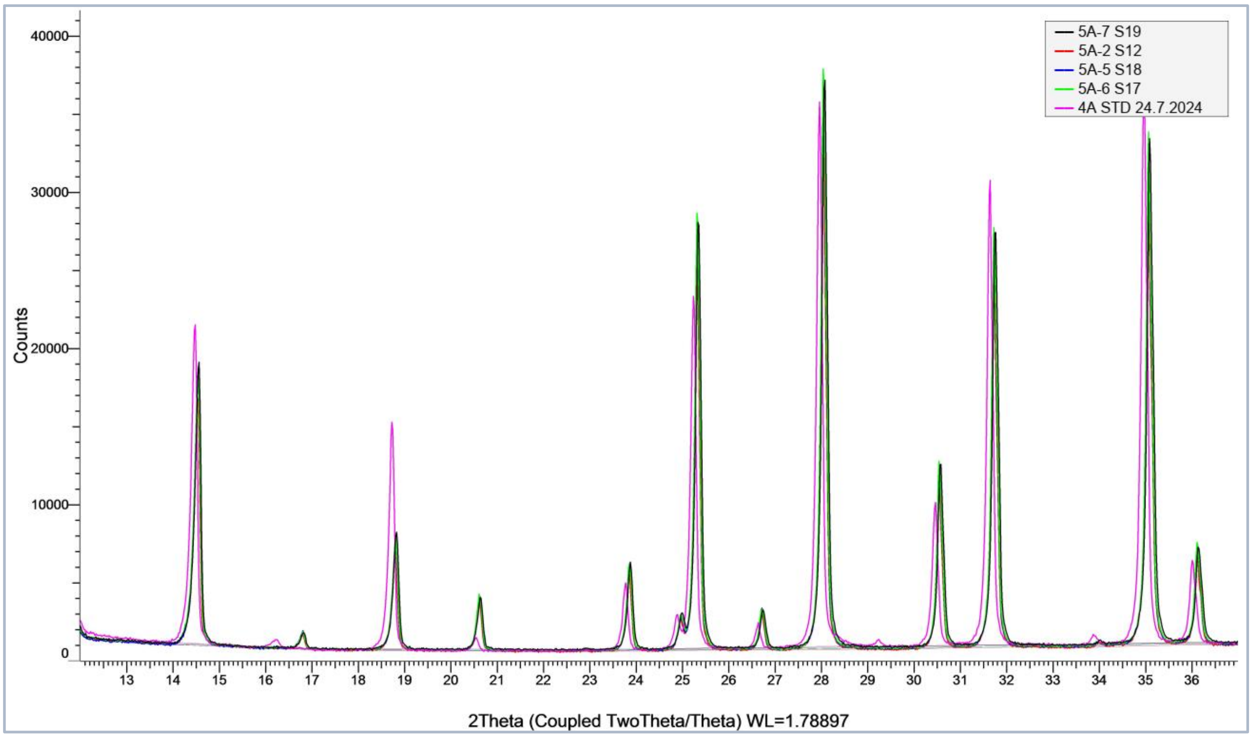


Figure 3.Diffractograms of 5A zeolite powders obtained at different ion exchange temperatures

XRD analysis of samples from the second series, where ion exchange was performed at different temperatures, confirms the effect of the percentage of exchanged ions on the measured relative crystallinity of the material. The obtained crystallinity values are even more precise in this case than in the previous example, but higher crystallinity is observed in samples with a lower percentage of exchange.

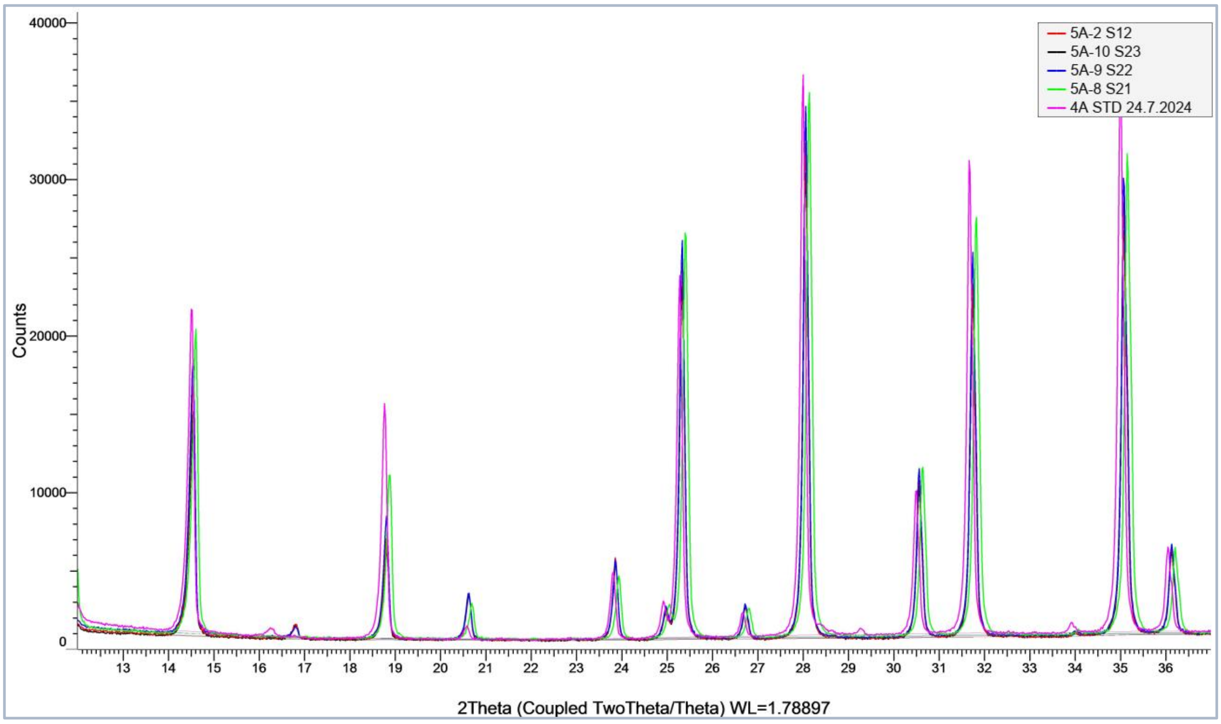


Figure 4.Diffractograms of 5A zeolite powders obtained at different ion exchange duration

The same trend is observed in the third series of samples. The sample with the shortest retention time in the ion-exchange system has the lowest percentage of exchanged ions and thus retains a crystallinity and water adsorption capacity closest to the initial sample. A noticeable decrease in crystallinity is also observed in the sample with the longest ion exchange duration, which most likely caused a slight reduction in water adsorption capacity compared to samples exchanged for 60 and 120 minutes. The poorer crystallinity of this sample can again be attributed to the higher ion exchange, but the crystallinity percentage is also somewhat lower than in samples with very similar ion exchange percentages, suggesting that even longer retention times in the ion-exchange system may have a slight effect on crystallinity.

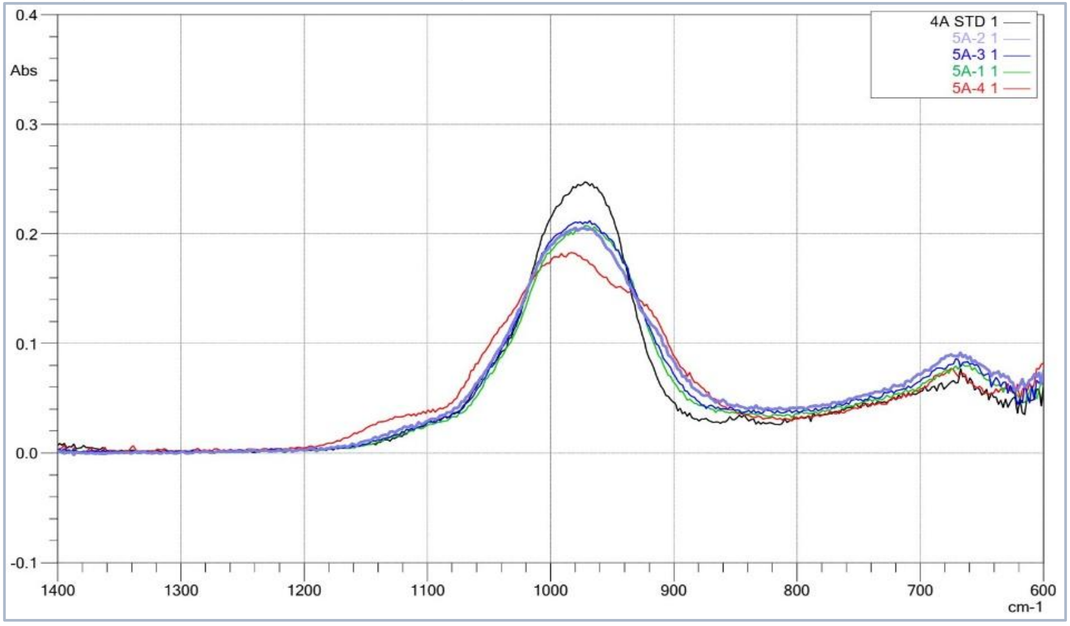


Figure 5. FT-IRspectra of 5A zeolite powders obtained through ion exchange from solutions with different $MgCl_2$ concentrations

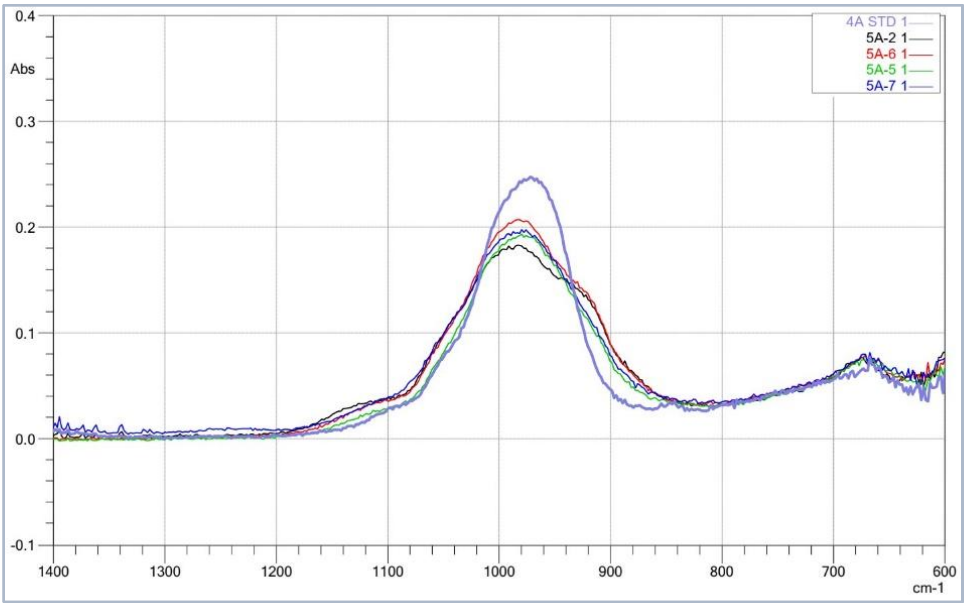


Figure 6. FT-IRspectra of 5A zeolite powders obtained at different ion exchange temperatures

In the FT-IR spectra, the difference between the initial 4A zeolite and the 5A zeolite samples is most pronounced in the range of 900 to 1100 cm^{-1}, which corresponds to the characteristic stretching of T-O-T bonds in the zeolite's crystal structure. It is evident that the intensity of this peak drastically decreases with the ion exchange process, with nearly all 5A zeolite samples clearly differing from the initial 4A zeolite sample in this part of the diagram. This also confirms the hypothesis that the difference in material crystallinity, which increases with the percentage of ion exchange, primarily arises from different bond strain within the zeolite's crystal lattice. Additionally, this phenomenon is particularly noticeable in samples with the greatest reduction in relative crystallinity, which belong to the first and third series of samples, indicating a correlationwith the XRD analysis results. The difference in the FT-IR spectra is also observed in the 650-700 cm^{-1} range, where slightly higher activity is noted in the exchanged samples, especially those with exchange percentages around 40 to 55%, compared to the initial sample.

Overall, given the relatively simple sample preparation for this analytical method and the speed of the analysis, the patterns observed in the FT-IR diagrams suggest that this analysis can quickly determine whether a sample is 4A or 5A zeolite.

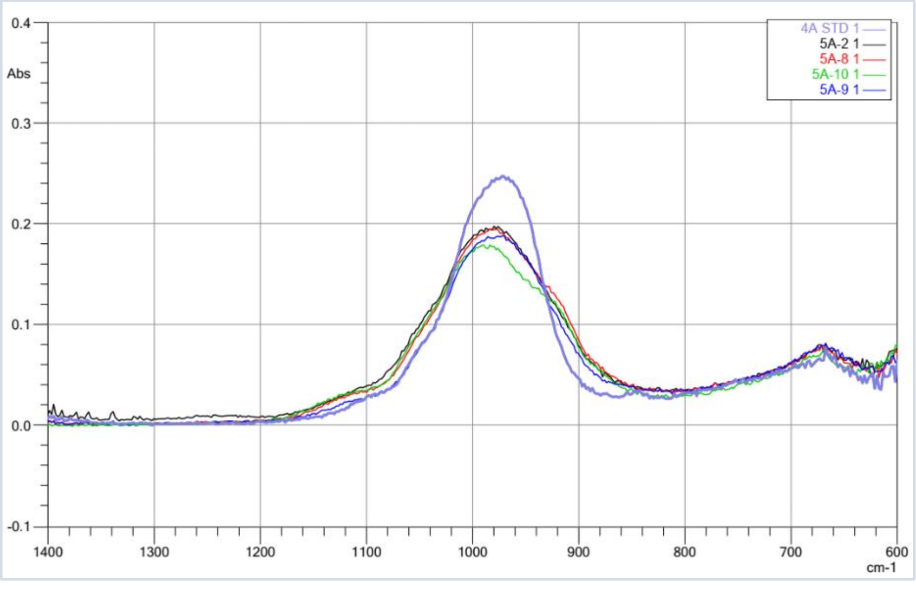


Figure 7. FT-IRspectra of 5A zeolite powders obtained at different ion exchange durations

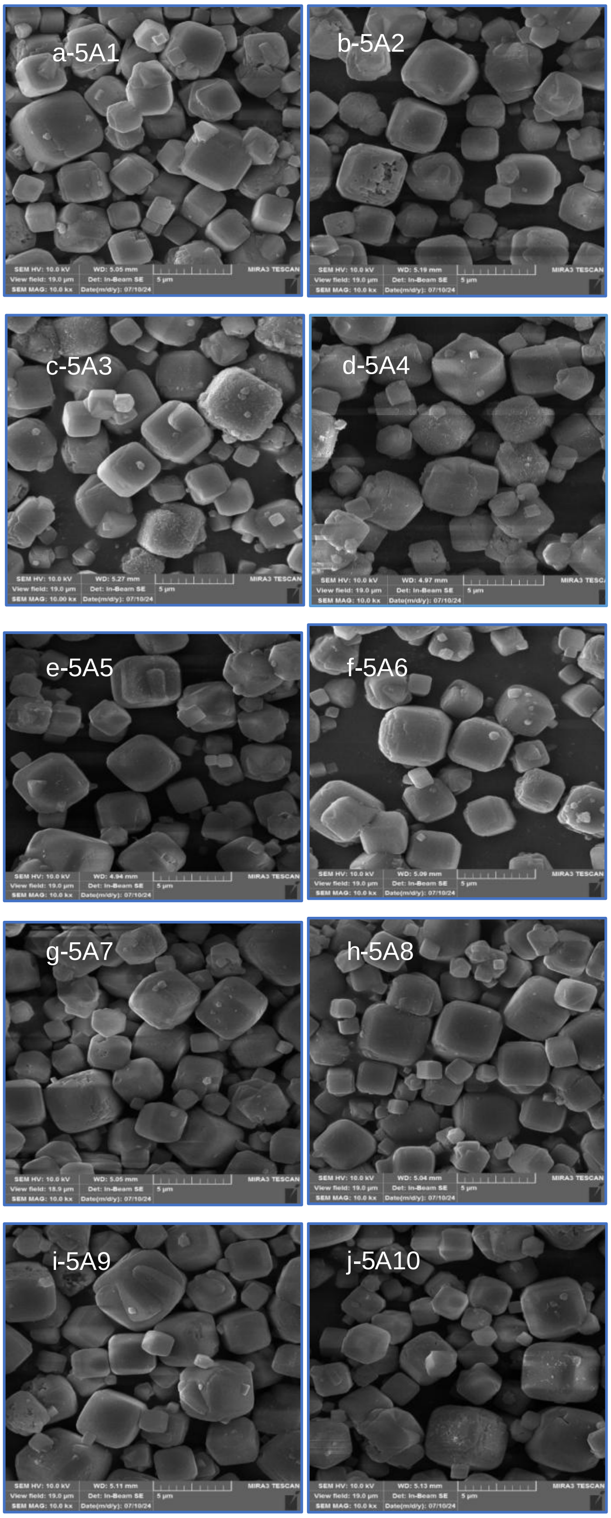


Figure 8 a-j. SEM images of 5A zeolite powders obtained under different ion exchange conditions

SEM images of samples 5A1 – 5A4 (Figure 8a-8d) show materials with well-formed crystal shapes and a relatively uniform particle distribution. There is no significant presence of agglomerates. These favorable characteristics of the observed crystals are largely attributed to the properties of the initial 4A zeolite material. It is important to note that the crystals of powders obtained at higher concentrations of the ion-exchange solution exhibit noticeable fine contamination. This contamination may originate from MgO, encouraged by a higher concentration of Mg^{2+} ions in the system. The distribution of these contaminants is not uniform. It is assumed that a higher amount of formed contamination requires more intensive washing of the material after ion exchange, which may also affect the reduction of water adsorption capacity.

Furthermore, examining samples where ion exchange was conducted at different process temperatures 5A2, 5A5 – 5A7 (Figure 8b, 8e-g), the SEM images show that temperature does not have a significant impact. The SEM images of samples 5A1 – 5A4 (Figure 8a-8d) reveal no significant presence of agglomerates, and these favorable characteristics of the observed crystals are largely due to the properties of the initial 4A zeolite material.

Samples obtained from ion exchange with lower tested concentrations of $MgCl_2$ (Table 1) also show no presence of the previous mentioned impurities, which can be associated to the formation of MgO at higher concentrations of Mg^{2+} ions in the solution.

For samples obtained with different ion exchange durations, 5A2, 5A8 – 5A10 (Figure 8b, 8f-h), there is once again the presence of fine contamination on the surface of certain particles, particularly in the sample exposed for the longest time in the ion-exchange solution, for 180 minutes. This contamination was not observed in the other samples from this series. Based on the previous assumption that contamination originates from separated MgO on the particle surfaces, it can be said that prolonged retention of zeolite in the ion-exchange system favors the formation of this impurity. This phenomenon and its effects on the properties of zeolite powders could certainly be the subject of further analytical investigation.

Based on the data presented in Table 1 and in Figures 9a-9j, it can be observed that there are almost negligible changes in the average particle diameter of the obtained 5A zeolite powders when varying the process parameters: concentration of $MgCl_2$ in the ion-exchange solution, temperature, and duration of ion exchange. The largest average particle diameter, at 4.22 µm, is observed in the zeolite sample with the highest percentage of exchanged ions, 5A4 zeolite, which is consistent with the previously mentioned interpretations of the XRD analysis, as it is often the case that a decrease in crystallinity leads to an increase in the average particle diameter of zeolites [16]. Based on the obtained histograms (Figures 9a – 9j), it can be noted that the particles of all analyzed samples are uniform, given the narrow size distribution of the particles.

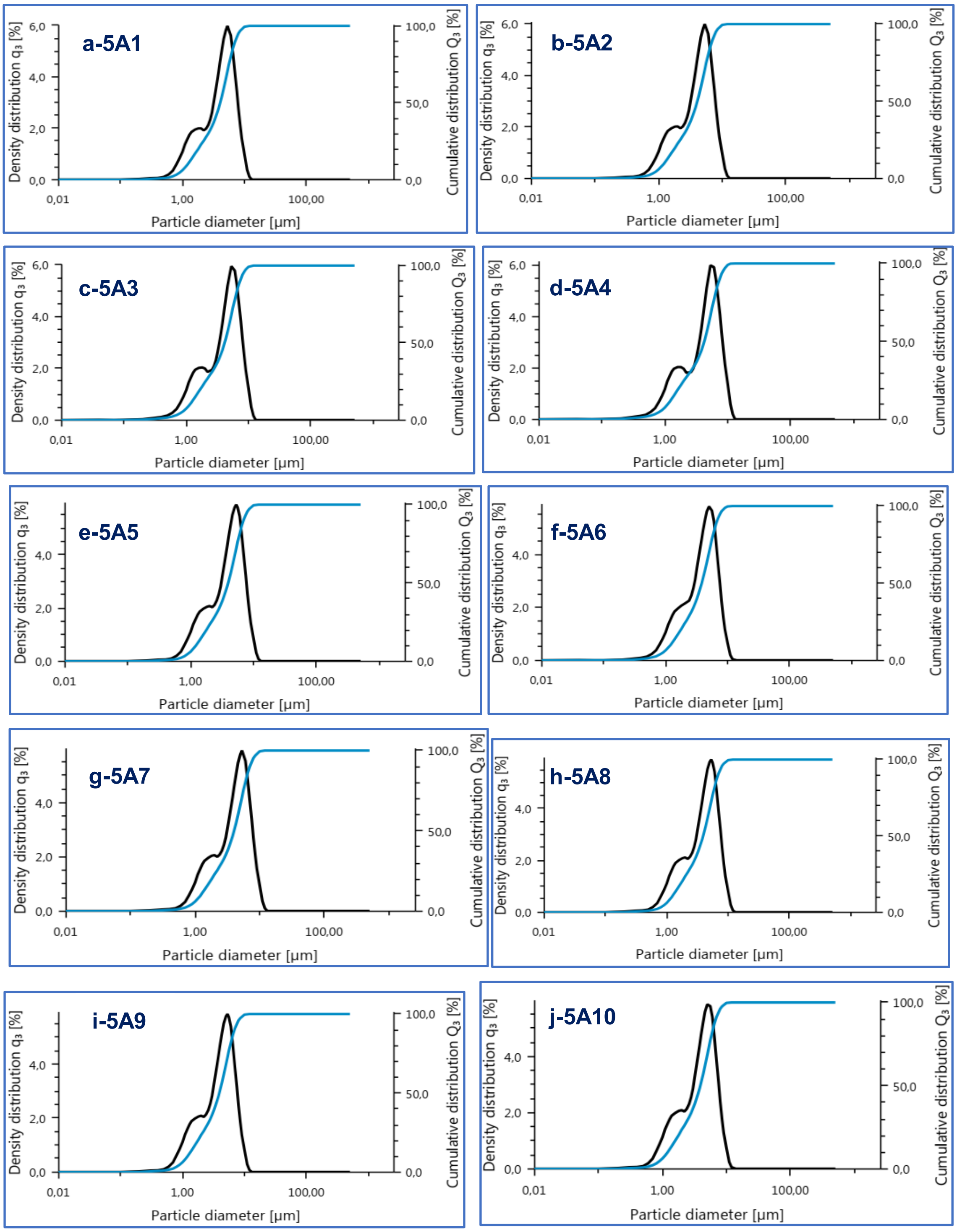


Figure 9 a-j. Particle size distribution of 5A zeolite powders obtained under different ion exchange conditions

4. CONCLUSION

Considering water adsorption capacity as a key quality parameter for 5A zeolite, it can be observed that the highest values for this parameter are achieved with approximately 50-55% ion exchange, which corresponds to about half of the exchanged ions. Further increases in the ion exchange percentage lead to decreased crystallinity of the material, which limits the increase in adsorption capacity. Additionally, within the given range, it is evident that increasing the ion exchange percentage from around 40% to 60% requires a significant increase in the amount of $MgCl_2$ used as a raw material, while this same increase only results in about a 1.5-2% rise in water adsorption capacity. Therefore, considering the substantial impact of raw material consumption on the cost-effectiveness of the process, it is very important to determine the optimal ion exchange value in 5A zeolite according to the material's intended application. Based on the results of this study, the optimal value is around 50% ion exchange.

Furthermore, higher concentrations of the ion exchange solution and longer retention times of the material in the solution promote the formation of impurities on the surface of 5A zeolite crystals, necessitating more intensive washing of the zeolite. An ion exchange duration between 1 and 2 hours can be considered optimal for the process.

XRD and FT-IR analyses provided interesting results regarding the differentiation of 5A zeolite from the starting 4A zeolite. Notably, FT-IR analysis distinctly demonstrates these differences in its spectra, and this analytical method requires relatively minimal sample preparation and analysis time.

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IZVOD

UTICAJ PROCESNIH PARAMETARA JONSKE IZMJENE NA OSOBINE DOBIJENIH PRAHOVA 5A ZEOLITA

Zeolit 5A predstavlja jedan od najboljih adsorbenata među zeolitima što ga čini veoma interesantim proizvodom sa komercijalne strane. Najčešće se dobija iz 4A zeolita, tako što se Na^+ joni prisutni unutar kristalne rešetke 4A zeolita izmenjuju sa jonima Ca^{2+} ili Mg^{2+}, pri čemu se oslobađa više prostora unutar pora kristala, povećava otvor pora i raste adsorpcioni kapacitet zeolita. Sa stanovišta industrijske, odnosno komercijalne proizvodnje 5A zeolita, vrlo je bitno definisati optimalne procesne parametre. Stoga je u ovom radu ispitan uticaj temperature jonske izmene, trajanja jonske izmene i koncentracije aktivnih komponenti u sistemu na karakteristike dobijenog praha 5A zeolita. Jonska izmena je izvođena iz rastvora $MgCl_2 \cdot 6H_2O$, tako da je nastala Mg^{2+} forma 5A zeolita, odnosno Na,Mg-A zeolit. Prahovi dobijeni nakon jonske izmene su analizirani u pogledu hemijskog sastava i kapaciteta adsorpcije vode. Pored toga urađena je dodatna karakterizacija uzoraka: određivanje granulometrije i raspodele veličine čestica, difrakcija X-zraka, te SEM i FT-IR analize. Dobijeni rezultati ukazuju na zavisnosti između procesnih parametara sistema, procenta izmenjenih jona, kristaličnosti i adsorpcionih osobina materijala omogućavajući odabir optimalnih uslova proizvodnje 5A zeolita.

Ključne reči: *5A, adsorpcija, jonska izmena, procesni parametri, zeolit*

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