

Study of changes in surface properties of zeolite due to modifications

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Abstract

Zeolites are a mineral resource that is widely used mainly due to their specific properties. In the article, we investigated how the properties change - the internal capacity of zeolites under certain specific conditions, such as temperature change, time changes helium purification. All measurements of zeolite specific surfaces were performed on a Micromeritics Gemini VII - surface area and porosity analyzers.

The samples were measured (dust, various fractions, pellets) at various conditions: temperatures helium-purified (He), time changes.

The input and output parameters of the individual experimental samples were evaluated using descriptive statistics. All measured data were then analyzed by multidimensional ANOVA analysis and analysis, with our ambition to identify and quantify the dependence of the individual specimen treatments on the total output - measured values of the weight of the evaporated water, specific surface.

The results at the end are divided into small or insignificant influence of changes in other monitored parameters and on significant effects of parameters on the resulting parameters.

Small or insignificant influence of changes was stated by the amount of the water evaporated from the zeolite with respect to the time of heating of the samples prior to the evaluation of the zeolite specific surface. Similar conclusions can we come from the analysis of results where the different heating temperatures of zeolite samples were compared before determining their specific surfaces and the amount of evaporated water from the samples.

Significant effects of parameters on the resulting parameters: the different sampling times in their preparation for measuring the specific surfaces of the investigated zeolite samples significantly affect the measured specific surfaces. The weight of evaporated water is strongly dependent on the type of treatment - the values are significantly higher due to the effects of helium and vacuum.

Key words: zeolite, samples of zeolites, specific surface, ANOVA, numerical analyses, industrial rocks, temperature, helium, time

1. Introduction

Introduction in the current environmental problems with maintaining the purity and health acceptability of ingredients environment for nature and mankind, the modification of natural ones comes to the fore ion exchange materials to use them especially in cleaning or purification of various types of wastewater, including radioactive. By chemical treatment, zeolites acquire new, original properties, e.g. their surface increases, the sorption capacity increases, the physicochemical and mechanical ones improve properties, etc. For the chemical modification of zeolites, ion exchange is usually used, in which the zeolite is used to transform into a mono-cationic form, e.g. Na-zeolite, K-zeolite, etc. solutions of salts, hydroxides, or acids⁶.

Zeolite is a natural volcanic rock with unique physical properties. The uniqueness of zeolite is based on its extraordinary crystal structure, which enables it to capture substances in all: solid, liquid and gas states. Compositionally, zeolites are similar to clay minerals. More specifically, both are aluminosilicates. They differ, however, in their crystalline structure. Zeolites have a rigid, 3-dimensional crystalline structure (similar to a honeycomb) consisting of a network of interconnected tunnels and cages. The total volume of the cavity, also called porosity, is of 24 – 32%. Specific weight is around 2,200 – 2,440 kg.m⁻³, absorbability of used samples was as much as 39%. The properties of the zeolite come from its specific crystal structure. Special aspect of this structure is, that the pore and channel sizes are nearly uniform, allowing the crystal to act as a molecular sieve^{1,2}.

The tetrahedrons in space form a crystal lattice which differs from other types of similar lattices in that their cavities are interconnected by some channels which have an exact, always the same diameter for a given type of zeolite. The diameters of the channels of the natural zeolites of the collet from 0.2 nm to 0.7 nm form a micro-porous structure in which the volume and channels reach up to 50% of the total volume³.

These cavities and channels contain weakly bound water. Its amount is variable and depends on the chemical zeolite, the size of the channels and cavities as well as the external PCH e.g. temperature, the voltage of water vapor in the surrounding environment, and careful heating, this so-called zeolite water can be gradually removed from the grid without disturbing the mineral structure. The removed water can be re-absorbed by the mineral in the original amount, or in the absence of water it can be replaced by molecules of other substances (e.g. ammonia, ethyl alcohol, Hg, hydrogen sulfide, etc.) However only substances with a diameter less than or equal to such as the diameter of the inlet openings to the channels. Thus it is possible to separate e.g. argon from oxygen or molecules of paraffin with the same chain from paraffin branched - chained and the like. Thus, zeolites function as sieves or filters, by means of which we can selectively separate, "sieve" a certain type of molecules⁴.

In the Slovak Republic rock-forming zeolites represented by clinoptilolite, mordenite, and analcemia are found at the edge East Slovak Neogene basin between the villages of Vranov, Kučín, Nižný Hrabovec, Majerovce and Pusté Čemerné⁵.

Slovak zeolite from Nižný Hrabovec is gray-green and contains 40 – 70% clinoptilolite, 8 – 10% feldspar, 6 – 10% cristobalite, 2 – 5% quartz, and 13 – 30% volcanic glass. The primary cation in Slovak natural zeolite is potassium in other types it can be sodium, calcium, magnesium, iron, and volcanic glass. The Si / Al ratio for Slovak clinoptilolite ranges from 3.4 to 5.2. Zeolites clinoptilolite-type retain their structure over a wide pH range, from 1.0 to 11.5 and they are radiation stable to the generally stated absorbed radiation dose 107 Gy (gray). Slovak zeolite with primary cation K has very good thermal properties, withstands temperatures up to 600 °C without change¹⁵.

2. Literature review

The short review from literature was done, below are results. With Physical Adsorption Characterization of Mesoporous Zeolites deals authors, Thommes, M., Guillet-Nicolas, R., Cychosz, K.A.⁶ The authors of the book deal with the most important aspects of gas adsorption on the textural and structural internal structure of zeolites. The experiments were focused on the precise steps necessary to obtain accurate adsorption data and describe a detailed understanding of the basic adsorption mechanisms in surface and pores analysis. The BET (Brunauer, Emmett, and Teller) method is used to characterize the physical adsorption at surface evaluation. A more reliable analysis of the pore size of these materials is the method of Density functional theory (DFT)⁶.

Lecloux, A.J et al., discuss specific surface area. The specific surface analysis was used as a screening tool for the classification and identification of nanomaterials. The results are compared on particles with dimensions less than 100 nm. The research concludes that the experimental values of the analysis allow the identification of nanomaterials without any inaccurate negative identification (subject to certain conditions - particle size, relative pressure range, qualitative particle image). The proposed analysis model can be used to look for the potential effects of shape and size change for different nanoforms⁷.

An experimental study is focused on a new zeolite modification method (ZBHCM type) with an NH_4Cl solution and develops a new zeolite-based moisture control material. The type of zeolite for the study is determined by the morphological and physical characteristics of this material. Based on the experiments performed, it is proved that zeolite has an effect on the internal temperature and a significant influence on the humidity of the interior. These varies depending on the amount of zeolite used, with the issue deals Zhou, B et al. in the Experimental article study on moisture migration process of zeolite-based composite humidity control material⁸.

The research used the efficiency of MFIs, namely their synthesis by hydrothermal method. Subsequently, the results were evaluated by TGA analysis and BET analysis (coke storage utilization) for coke detection on MFI nanoparticles⁹.

The use of natural zeolites for environmental applications is gaining new research interests mainly due to their properties and significant worldwide occurrence. The present work describes the characterization of a natural Chilean zeolite and the results as adsorbent for ammonia from aqueous solutions. The zeolite-rich tuff sample, mainly composed of clinoptilolite and mordenite, consisted of 13 μm mean volumetric particle diameter, 55 $\text{m}^2 \text{g}^{-1}$ (methylene blue adsorption) and 177 $\text{m}^2 \text{g}^{-1}$ (nitrogen adsorption) of specific surface area. Particles were negatively charged over a broad pH range (with or without ammonia) and 1.02 meq $\text{NH}_4^+ \text{g}^{-1}$ cation-exchange capacity. The ammonia removal appears to proceed through ion-exchange and rapid kinetics (rate constant of 0.3 min^{-1}) at neutral pH value, with removal capacities up to 0.68 meq $\text{NH}_4^+ \text{g}^{-1}$. The Langmuir isotherm model provided excellent equilibrium data fitting ($R^2=0.97$). Results indicate a significant potential for the Chilean natural zeolite as an adsorbent/ion-exchange material for wastewater treatment and water reuse applications¹⁰.

Földesová, M., Hudec, P. made study of surfaces of natural and chemically modified zeolites of clinoptilolite type by physical adsorption of nitrogen at the temperature of liquid nitrogen by the volumetric method was done on Slovak zeolite clinoptilolite with very good results¹⁵.

3. Experiment and equipment

3.1 Equipment Gemini VII - measurement of specific surface

Before measuring the internal surfaces on zeolite samples (dust, various fractions, pellets), they were subjected to various drying, helium-purified and vacuum processes, under defined conditions – temperatures in a range of 120 – 300 °C respectively, and time from 0.5 to 2 hours.

Precise measurements of the internal zeolite structure can be carried out using the Micromeritics Gemini VII Surface Area and Porosity Analyzers (See the picture Figure 1). The device works on principles of material analyses and reliably produces accurate results on the surface but also in the internal pores of the measured material. The device reliably and repeatedly focuses on the total pore volume (not only surface) material and distributes the data directly through outputs to the digital form for further processing. The unique ability of the device is to analyze not only the surface of the material under investigation but also its internal specific surfaces (porous spaces in the specific structure)^{12,13}. The device can avoid some source mistakes associated with traditional static volume instruments by rejecting a common (general) mode. This feature provides much more accurate measurements. The device also includes software (Windows, Windows XP, or Windows Vista), that includes a video installation and verification system, including data processing and evaluation.



Figure 1: Equipment Gemini VII Surface Area and Porosity Analyzers

Specific device parameters:

The applicability: Surface area: From 0.1 m² (full), from 0.01 m². g⁻¹ (specific),

Pore volume: 4x10⁻⁶ cm³. g⁻¹.

Pressure measurements: Measured pressure range: 0 – 950 mm Hg, (0 – 126655,6 Pa)

P / P₀ resolution: <10⁻⁴,

Relative pressure range: 0 – 1.0 P / P₀ (adsorption only),

Pressure resolution: <0.1 mmHg, (13,332 Pa)

Accuracy and linearity: more than +/- 0.5%.

Environment: Temperature: Operating 10 – 35° C (50 – 96° F), room temperature 0 – 50° C (32 – 122° F),

Humidity: 20% – 80% relative, not condensation.

Adsorption: Optimal for the use of liquid nitrogen - Nitrogen analysis. The device is used for non-corrosive adsorption of gases under certain pressure at room temperature (and higher). Other gases used: oxygen, argon, carbon dioxide, butane, methane, other hydrocarbons. The helium is needed.

Vacuum system: The vacuum source is 20 x 10⁻³ mm Hg (even higher) than the supply device (it is recommended to connect the oil pair reduction device). The system includes an oil crossover device (as a preventive part of the device).

Sample tube: 0.95 cm, outside length 15.5 cm, internal volume: 6.5 cm³, maximum sample volume in the tube is approximately 2.0 cm³.

3.2 Device VacPrep 061 Degasser

Sample drying takes place on the VacPrep 061 Degasser laboratory equipment, which is a surface monitoring device and pore structure analysis. VacPrep offers two methods for removing pollutants from samples such as - water vapor and adsorbed sample gases, to avoid interference with surface measurements. The device ensures minimization of sample contamination during transfer from the device to the output analysis. It markedly increases process reliability and accuracy of results. VacPrep allows the removal of contaminants by heating samples up to 400° C using a vacuum system or flowing gas method, or a combination of both methods. The device includes six sample heating stations and six cooling stations. The temperature is set simply on the digital control panel located at the front of the device (See the picture Figure 2).



Figure 2: Equipment VacPrep 061 Degasser

4. Preparation of samples, measurement and analysis

4.1 Preparation of samples

All measurements of zeolite specific surfaces were performed on a Micromeritics Gemini VII - surface area and porosity analyzers.

The individual measurements differed from each other by the preparation of samples before the process of measuring specific surfaces, the drying times of the samples, the degrees of drying, the use of a vacuum system and the passage of helium through the sample varied.

The samples were measured (dust, various fractions, pellets) at various conditions: temperatures (120 ° C, 2 hours, 300 ° C, 0.5 hours, 300 ° C, 2 hours), helium-purified (He). Sample weight: 0.491g – 0.565g (before drying).

Samples of zeolites (of different grain size) were prepared at a precision of 0.01g on a metrological verified laboratory scale of the WTC 06 / B1 type before the drying process. The weight of each sample was as much as 0.491 – 0.539 g. The individual weight of each instrument tube is in the range 10.325 – 10.404 g (for this reason the weight of each tube was individually measured and matched to the weight of each prepared zeolite sample).

After measuring the sample weights, the samples proceeded to the drying process with the possibility of a vacuum and gas connection (helium) - individually used in all processes. Drying was carried out on VacPrep 061 Degasser at various temperatures (max 300° C when the crystalline lattice of the zeolite occurs). The drying time was from 0.5 to 2.0 hours. During the drying, the water present in the zeolite was evaporated from the samples. Following the drying process, the samples were again weighed to determine the amount of evaporated water (%)¹⁴.

The dried samples were then placed in a Gemini VII - Surface Area and Porosity instrument in the instrumentation tubes to measure their specific surface area ($\text{m}^2 \cdot \text{g}^{-1}$). Before the start of the measurement, the weight of the measured sample, the weight of the test tube and the sample density were entered into the software. Subsequently, the program started the measurement process and evaluated the results in the required format (text, graph - isotherms, table). The results were then analyzed and evaluated. Data were collected by the experiment. Values need to be adjusted for statistical analyzes in the JMP statistical software. Columns that gave a range of values and were perceived as categorical data were adjusted to the average values we achieved by a numeric indicator.

4.3 Numerical analysis

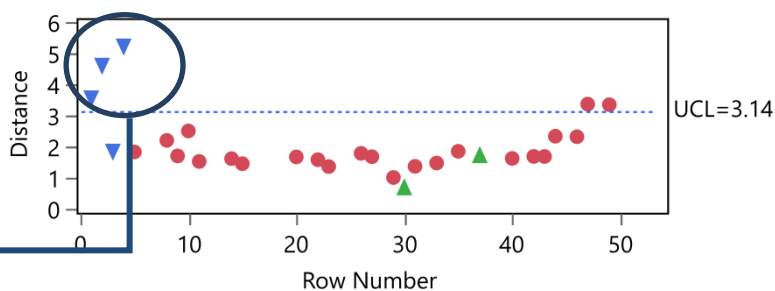
The input and output parameters of the individual experimental samples (See the picture Figure 3) were evaluated using descriptive statistics.

Samples with a fraction of 0.05 – 3 mm were included in the study, which was subsequently exposed to a temperature of 120 – 300 °C, with a heating time of 0.5 – 2.5 hours resp., in some cases the treated samples were exposed to He and Vac. With the samples thus prepared, the mass of evaporated water was measured in the range of 0.96 – 25.7% and the specific surface area in $\text{m}^2 \cdot \text{g}^{-1}$. Where the values ranged from 19.78 – 40.36 $\text{m}^2 \cdot \text{g}^{-1}$.

Multivariate Simple Statistics							
Column	N	DF	Mean	Std Dev	Sum	Minimum	Maximum
The grain sized - mean	49	48.00	0.7936	0.7536	38.8600	0.0500	3.0000
The heating °C	53	52.00	241.887	83.1370	12820.0	120.000	300.000
The time [hour]	53	52.00	1.4528	0.7290	77.0000	0.5000	2.5000
The weight of evaporated water [%]	35	34.00	6.3723	4.3069	244.217	0.9600	25.7000
The inner surface [cm ² /g]	51	50.00	25.3650	3.4642	1295.01	19.7798	40.3362

Figure 3: Multivariate Simple statistics of inputs and outputs

The basic analysis has been found to be extensions - measurements that are significantly different from the average (See the picture Figure 4), they are samples of tiny fractions (dust) that were also exposed to all treatments (temperature He and Vac).



The grain size [mm]	The grain size - mean	The heating °C	The time [hour]	Gas. He	Vac.	The weight of evaporated water [%]	The inner surface [cm ² /g]
<0.1	0.05	120	2	A	A	8.35	34.01
<0.1	0.05	300	2	A	A	19.06	40.33
0.2	0.2	300	2	A	A	25.7	28.01

Figure 4: Analysis of divergent values

All measured data were then analysed by multidimensional ANOVA analysis and analysis, with our ambition to identify and quantify the dependence of the individual specimen treatments on the total output - measured values of the weight of the evaporated water, specific surface.

4.4 Analysis of multivariate

Multidimensional analysis of the dependencies of individual numerical variables is limited to examining columns characterizing inputs (fraction, heating temperature, heating time) and characterizing outputs (evaporated mass, the inner surface). The result of the analysis pointed to only a slight dependence (See the picture Figure 5):

- determining the entry conditions: (inverse relationships - lower temperatures have been allowed at lower temperatures and shorter at higher temperatures),
- the dependence of the outputs: (direct relationship - increasing the weight of evaporated water, increase the inner surface of the sample),

Input-Output Dependence: (the direct relationship between exposure time and inner surface sample), this dependence was subjected to further investigation using ANOVA analysis.

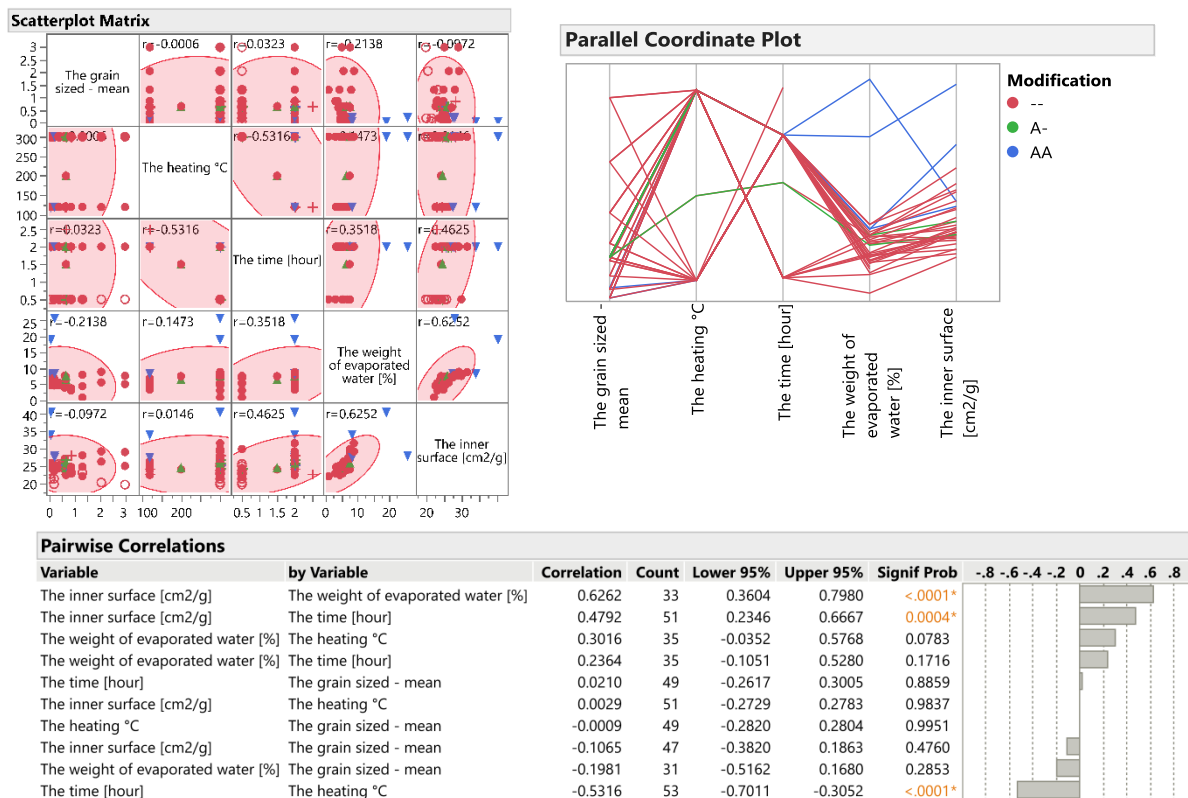


Figure 5: Multivariate analysis of numerical experiment data

4.5 Analysis of ANOVA

Data were examined using the variability analysis principles, for the difference in outputs (evaporated water mass, the inner surface) of the treatment method, which was considered in this case as categorical data:

- A. Temperature time (0.5 / 1.5 / 2)
- B. Operating temperature (120/200/300)
- C. Type of additional adjustments (without additional modifications / He / Vac + He)

A. HEAT RATE TIME

The analysis of the measured sized - mean values of the mass of evaporated water about the different time of temperature has not shown statistically significant variability so that it can be argued, that the time does not play an important role in the effort to achieve higher values of evaporated water (See the picture Figure 6).

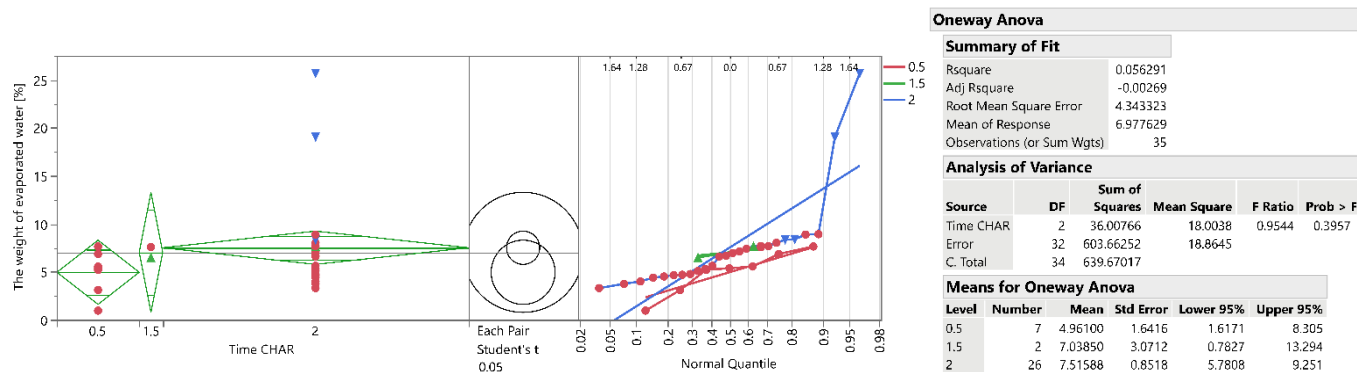


Figure 6: Analysis of ANOVA - the mass of evaporated water versus the time of heat

The analysis of the time influence on the inner surface of the samples showed a statistically significant difference in the values for the different time categories - 0.5 / 1.5 / 2 hours (See the picture Figure 7). On this basis, it can be argued that time play a statistically significant role in trying to increase and decrease the inner surface of the samples.

B. POSITION TEMPERATURE

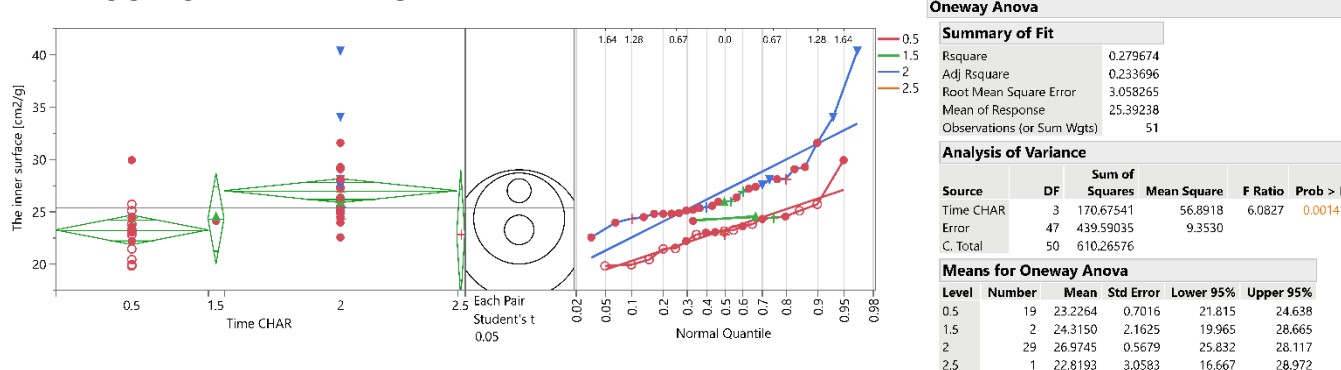


Figure 7: Analysis ANOVA – inner surface versus time of temperature activity

The analysis of the measured evaporated water weight values about the different temperature of 120/200/300 °C, respectively, did not show statistically significant variability, so it can be argued that the temperature not play a significant role in trying to achieve higher values of evaporated water (See the picture Figure 8).

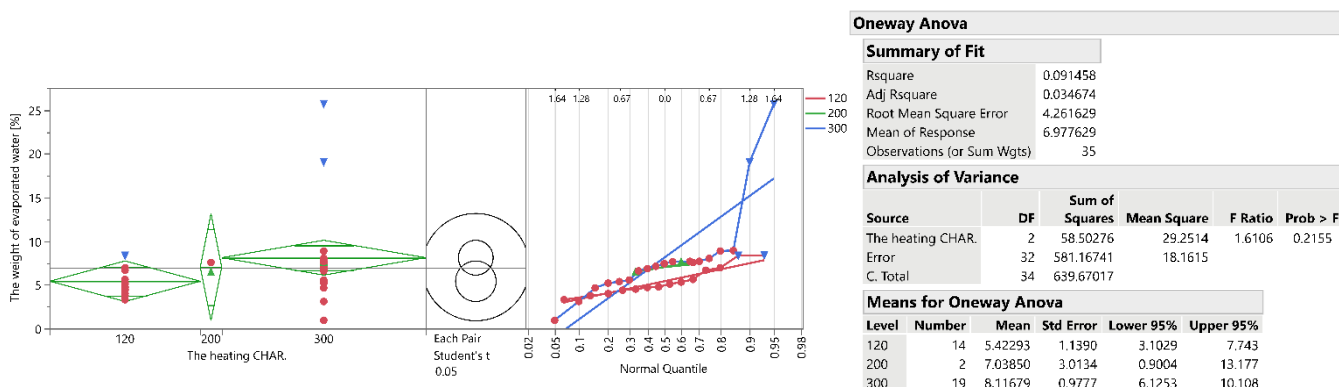


Figure 8: ANOVA analysis - Evaporated water weight versus heating temperature

Even the analysis of the temperature effect on the inner surface of the samples did not show a statistically significant difference in the values for the individual heat categories - 120/200/300 °C (See the picture Figure 9). On this basis, it can be stated that increasing / decreasing heating temperature cannot achieve statistically significantly higher / lower values of the inner surface of the individual samples.

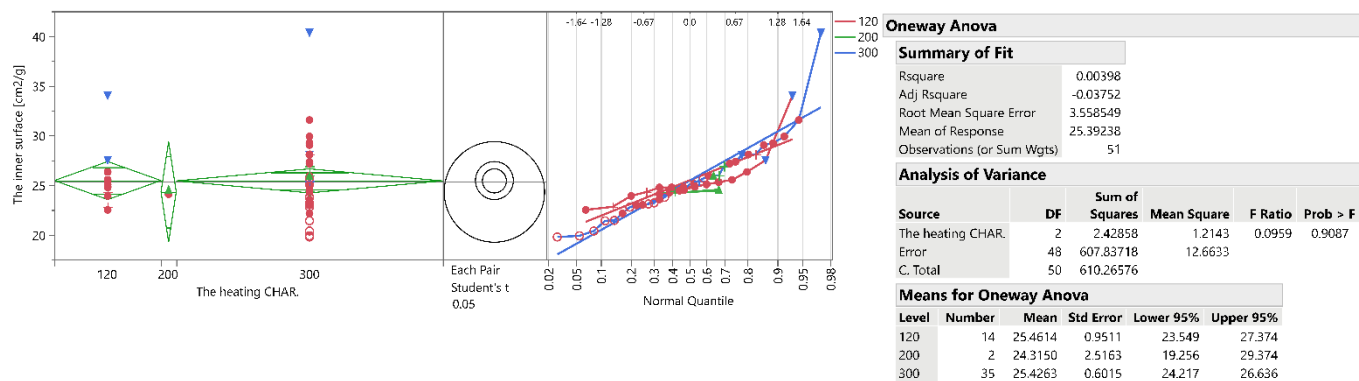


Figure 9: Analysis ANOVA - specific surface versus heating temperature

C. TYPE OF ADDITIONAL ADJUSTMENTS (WITH additional modifications / He / Vac + He)

Individual samples subjected to heat treatment were subsequently exposed to He and vacuum in various combinations. In the analyzes, these variants are marked as follows:

The effect of the individual variants of the additional treatments was assessed by the ANOVA analysis concerning the outlet values of the inner surface and the weight of the evaporated water of the individual samples.

The weight of evaporated water is strongly dependent on the type of treatment – the values are significantly higher due to the effects of helium and vacuum. The ANOVA analysis confirmed a statistically significant difference in the resulting evaporated water values depending on the type of sample treatment (See the picture Figure 10).

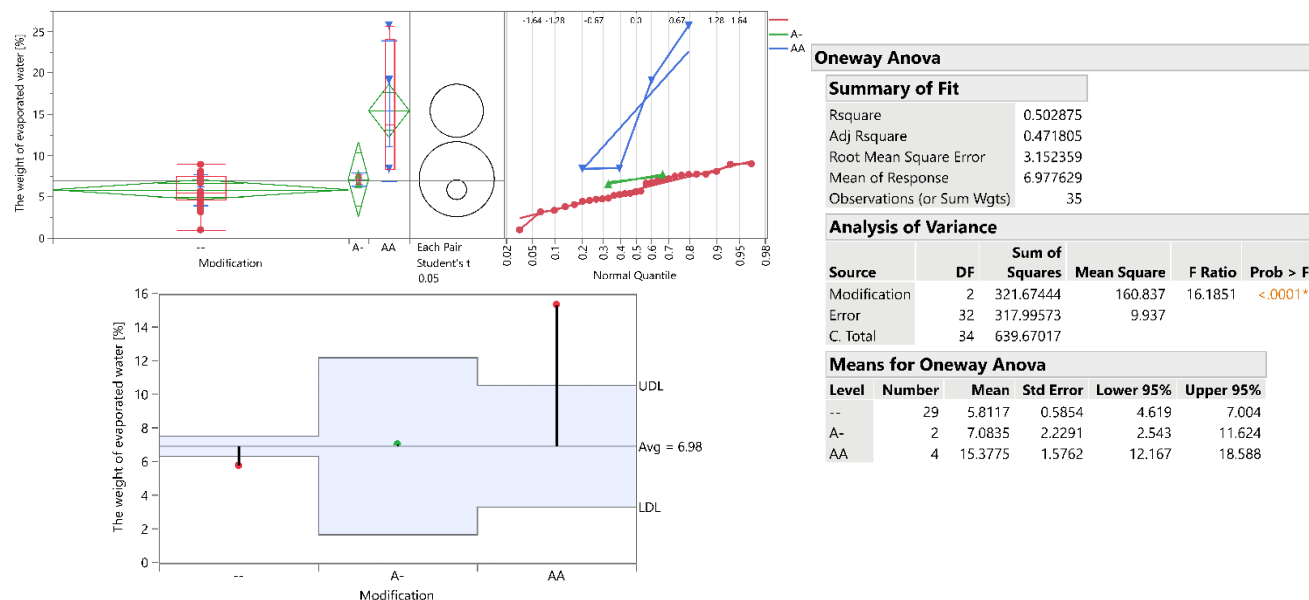


Figure 10: ANOVA analysis - evaporation water weight versus the type of treatment (He, Vac)

Further adjustments strongly influence the results of the internal indicator - the values are significantly higher due to the effects of helium, and Vac. Analysis of ANOVA confirmed a statistically significant difference in the resulting specific surface values, depending on the type of sample treatment (See the picture Figure 11). Variation of the "AA" effect – He and vacuum effects significantly increase the specific surface area of the samples above the average value.

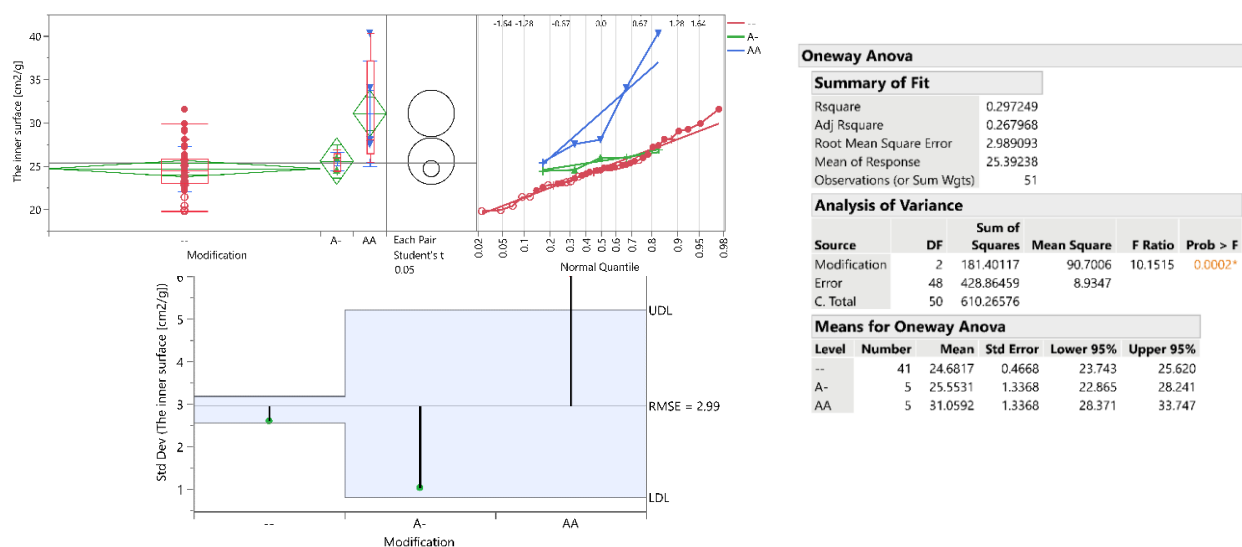


Figure 11: ANOVA analysis - inner surface versus the type of treatment (He, Vac)

5. Conclusion and recommendations

When measuring the specific surface area of the samples of natural zeolites, we used the top apparatus and followed all the recommended procedures.

The results can be summarized as follows:

Small or insignificant influence of changes in other monitored parameters:

1. The amount of the water evaporated from the zeolite with respect to the time of heating of the samples prior to the evaluation of the zeolite specific surface. As a result, it is known that the different heating time of the sample (0.5, 1.5 or 2 hours) does not have a significant effect on the change of the monitored parameter. (See the picture Figure 6).
2. Similar conclusions can come from the analysis of results where the different heating temperatures of zeolite samples (120, 200 and 300 ° C) were compared before determining their specific surfaces and the amount of evaporated water from the samples. (See the picture Figure 8).
3. Even the study of the influence of temperature change in the preparation of zeolite samples before measuring the specific surfaces did not show the effect of the change of this parameter on the resulting measured specific surfaces of the zeolite samples.

Significant effects of parameters on the resulting parameters:

1. In contrast, the different sampling times (0.5, 1.5 and 2 hours respectively) in their preparation for measuring the specific surfaces of the investigated zeolite samples significantly affect the measured specific surfaces (See the picture Figure 7)
2. The weight of evaporated water is strongly dependent on the type of treatment - the values are significantly higher due to the effects of helium and vacuum. Variation of the "AA" effect - helium and vacuum effects significantly increase the specific surface area of the samples above the average value. (See the Picture Figure 11).

Recommendations resulting from the measurement and statistical analysis performed:

In case it is necessary to know the values of the specific surfaces of the minerals, it will be appropriate to respect the established facts:

1. Increasing the heating temperatures of zeolite samples before measuring their specific surfaces above 120 ° C is inefficient
2. Extend heating time up to 2 hours increases the measured values of the specific surfaces of the zeolite samples

3. The most important factor in the increase of the measured values of the specific surfaces is the treatment of the samples before measuring the helium purification and vacuuming them.

In the study, we showed that, depending on the sample preparation, the measured specific surfaces of the zeolite are significantly affected. Achieving above-average values in the monitored property is conditioned by the type of selected treatment.

The facts found are valid for zeolite samples. In order to be generalized, measurements will also need to be repeated on other rock samples.

6. References

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Štúdium zmien povrchových vlastností zeolitu v dôsledku jeho úprav

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Souhrn

Zeolity sú minerálnym zdrojom, ktorý sa používa hlavne kvôli svojim špecifickým vlastnostiam. V článku sme skúmali, ako sa menia vlastnosti – vnútorná kapacita zeolitov – za určitých špecifických podmienok, ako je zmena teploty, zmena času, aplikácia hélia. Všetky merania špecifických povrchov pre zeolit sa uskutočňovali na prístroji Micromeritics Gemini VII – analyzátory povrchovej plochy a pórovitosti.

Vzorky sa merali (prach, rôzne frakcie, pelety) za rôznych podmienok: zmena teploty, aplikácia hélia (He), časové zmeny.

Vstupné a výstupné parametre jednotlivých experimentálnych vzoriek boli vyhodnotené pomocou deskriptívnej štatistiky. Všetky namerané údaje boli potom analyzované multidimenzionálnou analýzou a analýzou ANOVA s našou ambíciou identifikovať a kvantifikovať závislosť jednotlivých úprav vzoriek od celkového výkonu – namerané hodnoty hmotnosti odparenej vody, špecifický povrch.

Výsledky sú na konci rozdelené na malý alebo nevýznamný vplyv zmien v ďalších sledovaných parametroch a na významný vplyv parametrov na výsledné parametre.

Malý alebo nevýznamný vplyv zmien bol stanovený množstvom vody odparenej zo zeolitu vzhľadom na dobu ohrevu vzoriek pred vyhodnotením špecifického povrchu zeolitu. Podobné závery môžeme odvodiť z analýzy výsledkov, kde boli porovnané rôzne teploty zahrievania vzoriek zeolitu pred stanovením ich špecifických povrchov a množstva odparenej vody zo vzoriek.

Záverečné zistenia: významné účinky parametrov na výsledné parametre: rozdielny čas vystavenia vzoriek účinkom, ako je teplo, použitie vákua a hélia pri ich príprave na meranie špecifických povrchov skúmaných zeolitových vzoriek významne ovplyvňujú namerané špecifické povrchy. Hmotnosť odparenej vody silne závisí od typu úpravy - hodnoty sú vďaka účinkom hélia a vákua výrazne vyššie.

Kľúčové slová: zeolit, vzorky zeolitov, špecifický povrch, ANOVA, numerické analýzy, priemyselné horniny, teplota, hélium, čas