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Micronization of zeolite in vibration mill

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ABSTRACT

Dry micronizing grinding of mineral raw materials is a very complex process and depends on a whole range of parameters such as: shape, particle size, size distribution of the starting and micronized material, hardness, moisture, density, surface properties, structural characteristics of mills, etc. In order to obtain a clear picture of dry micronizing milling process, a good knowledge of the theoretical principles of high-energy mills operation is necessary.

Classical mills are not suitable for micronization to a fine and particularly very fine particles size (upper particle size limit below 5 µm), which is necessary for modern industrial application domains. The intensive fundamental and developmental research of the dry micronizing milling process for the new materials development has also enabled the development of new construction solutions for ultra-fine high-energy mills - mechanics. These mills have found application into the state-of-the-art production technologies of new very profitable materials. For the production of powders - particles of micron dimensions, the mechano-actuators are used and all micronizers - disintegrators, colloidal (perl mills), vibratory, planetary, ultra-centrifugal mill, Jet-current mills and others operate on the principle of impact, shock and friction.

This paper presents the experimentally achieved results of dry micronizing grinding of zeolites in a vibrating mill with rings, as well as the improvement of its reactivity. All experimental micronization investigations were carried out on a previously prepared coarse particle size class (- 3.35 + 0) mm with different starting mass (50 g and 200 g), and carried out in different grinding time intervals (45, 120, 900 seconds), in order to examine the impact of these parameters on the micronization process. Grinding success was evaluated by the particle size measurements and content of class (- 5 + 0) µm, and particle specific surface. It was shown that for a sample of four times larger initial mass it is need longer grinding time to achieve the maximum content of the class (- 5 + 0) µm, but for a sample of the bulk class (- 3.35 + 0) mm with a lower starting mass the effects of amorphization due to prolonged micronizing grinding are very pronounced, and the zeolite minerals in crystalline form are practically in the trace and the largest part is amorphous. Generally, it can be concluded that a vibrating mill with a rings proved to be a good device for efficient ultra-fine micronizing grinding.

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1. Introduction

In the history of humanity non-metallic mineral raw materials were the first natural materials that a man began to use, and most of them until the beginning of the 20th century did not attract attention. Between the two World Wars, and especially during and after the Second World War, there is a significant change in relation to these raw materials, they become increasingly important and necessary to the modern industry. One of such non-metallic mineral raw materials are certainly zeolites that represent precious inorganic material that has a wide range of applications (Andrić, 2014).

Natural zeolites are the most porous crystalline hydrated alumosilicates (tectosilicate) whose structures contain infinite three-dimensional frames. The basis of the three-dimensional network consists of tetrahedral groups SiO_4 and AlO_4 (Johan et al., 2015; Stocker et al., 2017). The adjacent tetrahedrons are bound, at their corners, through a common oxygen atom, making the zeolites structure rich in cavities and channels. The channel opening consists of 6, 8, 10 or 12 oxygen atoms together with the same number of silicon and / or aluminum atoms. Tetrahedral groups are connected in space through common oxygen atoms by constructing secondary polyhedron units whose connection creates a crystalline (alumosilicate) grid (Baerlocher et al., 2007).

The alumosilicate grid is formed by connecting Si(IV)-O and Al(III)-O of the building units, and it is negatively charged. Electroneutrality is provided by the alkali and / or alkaline earth metal cations located inside the channel and the cavity of the grid. These hydrated cations are mainly Na^+, K^+, Mg^{2+} and Ca^{2+} and rarely Ba^{2+} and Sr^{2+}.

Due to poor electrostatic interactions with the alumosilicate gratings, these cations are mobile, and in contact with the solution, they can be easily exchanged with ions from the solution, which gives the zeolites the ion exchanger properties (Jovanović, 2016). Zeolites are actually forms of "molecular sieves" associated with micropores and cavities. Structural empty spaces in the molecular-scale range (3-10 A°) can provide space for receiving cations (Na^+, K^+, Mg^+, Ba^+, Ca^+), and various metal cations, transition metal ions (Co, Fe, Mn, Zn), as well as molecules and ions from the group H_2O, NH_3, CO_3^{2-}, NO_3^-. In the channels and cavities there are also water molecules that can be reversibly desorbed, which gives to zeolites the properties of the drying agents (Alver et al., 2010; Terzić et al., 2017).

Due to their unique structure, zeolites have wide application (Charkhi et al., 2010). Zeolites are inactive in nature, and their adsorption capability and cation exchange capacity are activated by mechanical treatment. Activated zeolites are used as organic products in construction, agriculture and livestock, veterinary medicine, antioxidants and as adsorbents for heavy and radioactive metals, in food, textile and other industries. Zeolites are also used for the separation of gases due to its adsorption potential (Ackley et al., 2003; Wang et al., 2015; Chen et al., 2017), for the collection of unpleasant odors and removing moisture traces from organic solutions, from the rooms and therefore also been used as aquarium filters, purification of water and wastewater containing heavy metals and ammonia (Cooney et al., 1999; Margeta et al., 2013; Kahler, 2014; Ghasemi et al., 2016), gas sorption as catalysts (Tomić, 2016).

2. Micronization milling of non-metallic mineral raw materials

The process of micronizing grinding is intended to grind the mineral raw material to particles of micron dimensions and thus prepare for direct application or for further technological processing. The micronizing grinding process is mainly used for grinding mineral raw materials that have been already crushed and grinded. This process is carried out under the influence of external forces, as is the case with standard grinding (Andrić and Trumić, 2013), and the intensity of the micronizing grinding process depends on the characteristics of the micronized mineral raw material, the purposes and the market requirements regarding the quality of the product. During micronizing grinding there is a decrease in the regularity of particle shape, which reflects in the increase in the content of fine particles at the expense of the large ones. One of the basic factors affecting the shape and interaction of the individual particles content are the type of process and the type of micronizing grinding device which is used (Prziwara et al., 2018). Today, special attention is paid to the effect of micronizing grinding mills, in other words, to the micronized product costs, especially if the mineral resource is low. However, recently, all micronized mineral raw materials, especially non-metallic mineral raw materials, have market value. In the micronizing grinding of non-metallic mineral raw materials, special attention is paid to the following basic parameters of product quality: the content of individual size classes, the specific surface, the average particle diameter, the chemical and mineralogical characteristics. In addition to the bulk, as a basic parameter of the quality of micronizing grinding products, it is necessary to mention other very important parameters: volume mass, surface activity, particle shape, optical properties, etc. The mentioned parameters of the quality of micronizing grinding products depend on the physical, mechanical, chemical and mineralogical properties of the starting materials, such as: structure, texture, resistance, plasticity, durability, hardness, abrasiveness, sagnosticity, elasticity, stickiness (Andrić et al., 2010; Andrić and Trumić, 2013).

3. Material and methods

The micronizing grinding of zeolite was carried out in a laboratory vibrating disc mill „SIEBTECHNIK TS-250“, with rings at 1,000 rpm over a time interval of (45, 120 and 900) seconds. For all laboratory experiments of the zeolite micronization, a grossness class (- 3.35 + 0) mm was used with different starting mass (50 and 200) g and as a characteristic grossness class, the share of the class (- 5 + 0) µm was also monitored. On each micronizing grinding product, the particle size distribution and the specific surface area were monitored all in order to obtain the finest micronized product of improved reactivity.

After analyzing the particle size distribution of micronizing grinding products, X-ray diffraction analysis was carried out to determine and monitor the phase composition of the samples. The samples were analyzed on the "PHILIPS", X-ray diffractometer, the PW-1710 model with a curved graphite monohromator and a scintillation counter. The diffracted CuKα X-ray diffraction intensities (λ=1.54178Å) were measured at room temperature at intervals of 0.02 °2θ and at a time of 1 a in the range of 4 to 65 °2θ. The X-ray tube was loaded with a voltage of 40 kV and a current of 30 mA, while the slots for direct and diffracted beam direction were 1 ° and 0.1 mm.

3.1. Physico-chemical and mineralogical characteristics of the initial zeolite sample

The measured density of the zeolite sample was determined by glass pycnometers, and the average value after three measurements was: ρ=2,186 kg/m^3, which fits the theoretical values for zeolite ρ=2,000 – 3,000 kg/m^3 (Majstorović-Necković, 2015; Mihajlović-Kostić, 2016).

In Table 1, the results of the particle size distribution and the theoretical specific surface area of the initial zeolite sample of the bulk class (- 3.35 + 0) mm are shown.

Table 1
Particle size distribution of the bulk zeolite sample (- 3.35 + 0) mm

	Dry screening		Wet screening	
Particle size (mm)	W (%)	D (%)	W (%)	D (%)
- 3.35 + 2.36	17.96	100	14.71	100
- 2.36 + 1.70	14.93	82.04	10.04	85.29
- 1.70 + 1.18	13.21	67.11	9.75	75.25
- 1.18 + 0.85	9.03	53.90	5.18	65.50
- 0.85 + 0.600	7.97	44.87	6.47	60.32
- 0.600 + 0.425	5.93	36.90	4.37	53.85
- 0.425 + 0.300	5.31	30.97	3.82	49.48
- 0.300 + 0.212	3.70	25.66	3.19	45.66
- 0.212 + 0.150	4.18	21.96	3.29	42.47
- 0.150 + 0.106	3.68	17.78	3.36	39.18
- 0.106 + 0.075	3.44	14.10	3.07	35.82
- 0.075 + 0.040	4.56	10.66	5.87	32.75
- 0.040 + 0.030	0.05	6.10	0.29	26.88
- 0.030 + 0.020	0.05	6.05	0.23	26.59
- 0.020 + 0.010	0.15	6.00	0.08	26.36
- 0.010 + 0.005	0.06	5.85	0.17	26.28
- 0.005 + 0	5.79	5.79	26.11	26.11
∑	100.00	-	100.00	-
Calculated theoretical specific surface area, S (m^2/g)	**70.79**		**294.07**	

The particle size distribution of the initial sample of the bulk class (- 3.35 + 0) mm sieving with dry and wet method are significantly different. It can be noticed that when dry procedure is followed, the finest particles formed aggregates (larger particles), which does not occur with wet sieving process. Such a phenomenon leads to a decrease in the content of the finest class (- 5 + 0) µm from 26.11 % to 5.79 %, and therefore, up to four times lower value of the specific surface is obtained.

The determination of the chemical composition of the initial sample of zeolite, was carried out using a standard analytical method. The results of the chemical analysis are given in Table 2.

Table 2
Chemical composition of the initial zeolite sample

Component	Content, %
SiO_2	64.05
Al_2O_3	15.29
Fe_2O_3	2.52
CaO	4.82
MgO	1.33
K_2O	0.77
Na_2O	1.27
LoI *	9.86

* LoI - The loss on ignition (LoI) was determined as a weight difference between 20° and 1000° C.

The diffractogram of the initial zeolite sample a polycrystalline powder is shown in Figure 1 and it can be seen that the most prevalent minerals in the analyzed sample are zeolite minerals, while quartz, feldspars and micas are significantly less represented. Of feldspar, plagioclases are more dominant than K-feldspar. From the carbonate minerals the presence of calcite appears in the trace.

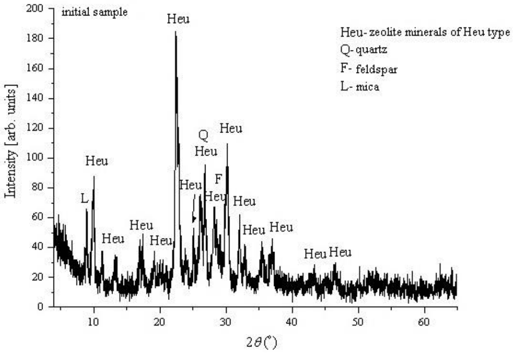


Figure 1. XRD analysis of the initial sample of zeolite

4. Results and discussion

4.1. Micronization grinding of zeolites

The results of the micronizing grinding of zeolite particle size class (- 3.35 + 0) mm are shown in Table 3 and Table 4.

Table 3
Particle size distribution and specific surface of micronized zeolite, bulk class (-3.35 + 0) mm, starting mass 50 g

SAMPLE: **(- 3.35 + 0) mm**	**45 seconds**		**120 seconds**		**900 seconds**	
Particle size, (μm)	W (%)	D (%)	W (%)	D (%)	W (%)	D (%)
- 212 + 150	0.29	100.00	0.80	100.00	3.12	100.00
- 150 + 106	0.29	99.71	0.68	99.20	5.20	96.88
- 106 + 75	0.87	99.42	1.82	98.52	7.69	91.68
- 75 + 30	0.66	98.55	1.55	96.70	2.29	83.99
- 30 + 20	0.45	97.89	0.82	95.15	1.02	81.70
- 20 + 10	0.02	97.44	0.02	94.33	0.12	80.68
- 10 + 5	0.02	97.42	0.23	94.31	0.38	80.56
- 5 + 0	97.40	97.40	94.08	94.08	80.18	80.18
∑	100.00	-	100.00	-	100.00	-
Calculated-theoretical specific surface area, S (m^2/g)	**1.07**		**1.04**		**0.89**	

Table 4
Particle size distribution and specific surface of micronized zeolite, bulk class (-3.35 + 0) mm, starting mass 200 g

SAMPLE: **(- 3.35 + 0) mm**	**45 seconds**		**120 seconds**		**900 seconds**	
Particle size (μm)	W (%)	D (%)	W (%)	D (%)	W (%)	D (%)
- 212 + 150	0.25	100.00	0.54	100.00	7.25	100.00
- 150 + 106	0.97	99.75	0.25	99.46	7.76	92.75
- 106 + 75	2.74	98.78	1.28	99.21	8.85	84.99
- 75 + 30	1.40	96.04	2.16	97.93	1.89	76.14
- 30 + 20	1.83	94.64	1.24	95.77	1.06	74.25
- 20 + 10	0.20	92.81	0.53	94.53	0.69	73.19
- 10 + 5	0.12	92.61	0.61	94.00	1.01	72.50
- 5 + 0	92.49	92.49	93.39	93.39	71.49	71.49
∑	100.00	-	100.00	-	100.00	-
Calculated-theoretical specific surface area, S (m^2/g)	**1.02**		**1.03**		**0.80**	

Based on Table 3, it can be concluded that grinding takes place up to 45 seconds of micronizing grinding. Subsequently, the content of the class (- 5 + 0) μm begins to slowly decrease. Only after 45 seconds of micronizing grinding, the content of the class (- 5 + 0) μm was 97.40 %, but after 900 seconds of grinding the content decreased to almost 80 %, indicating that 17 % of the particles had a larger mean diameter.

Based on the results in Table 4, for a sample of the bulk class (- 3.35 + 0) mm, and the starting mass of 200 g, it can be observed that grinding takes place up to 120 seconds of micronizing grinding. Then, the content of the class (- 5 + 0) μm begins to decrease. After 120 seconds of micronizing grinding, the content of the class (- 5 + 0) was 93.39 %, and after 900 seconds of grinding the content decreased to almost 72 %, indicating that about 21 % of the raw material was reduced.When the particle size distribution of the starting mass 50 g and 200 g (Figure 2) is comparing, it can be noted that for a sample of 50 g a shorter grinding time is required to achieve the maximum value of the class (- 5 + 0) μm. At the starting mass sample of 50 g grinding takes place up to 45 seconds, and the content of the class (- 5 + 0) μm is 97.40 %. For a sample of four times larger initial mass 200 g it is need about 2.5 times higher grinding time to achieve the maximum content 93.39 % of the class (- 5 + 0) μm.

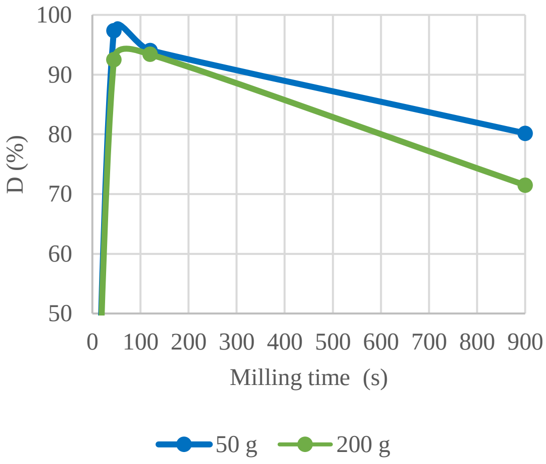
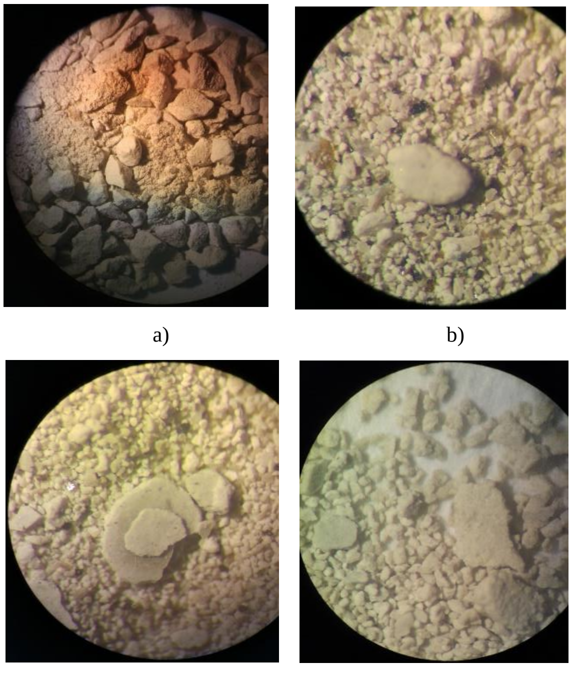


Figure 2. Cumulative mass of the bulk class (- 5 + 0) μm depending on the grinding time

In the ultra-fine micronizing grinding process, the energy consumption is very high. Due to this fact, the ring mill has proven to be an economical device for efficient ultra-fine micronizing grinding. Depending on the starting mass, (50 or 200) g, more than 90 % of the raw material is smaller than 5 μm after only 45 seconds of micronizing grinding, which is better than the results obtained by other researchers. Based on the literature, it was found that various authors in their work came up with the following results: Herceg et al., (2004) carried out tribomechanical micronization of zeolites using equipment designed for tribomechanical micronization and activation (TMA), and only after 60 seconds of grinding, was achieved the 90 % cumulative mass of the class (- 44.12 + 0) µm and of the class (-31.15 + 0) µm at a speed of 16,000 rpm and 22,000 rpm, respectively; Tunç and Demirkıran, (2014) investigated the effects of mechanical activation of the natural zeolite in a planetary mill with balls, and found that after 2 hours of grinding cumulative mass of the class (- 33.16 + 0) µm was 90 %; Burris and Juenger, (2016) performed micronization of zeolites in a gravity mill with balls, and found that after 8 hours of grinding cumulative mass of the class (- 24.3 + 0) µm was 90 %; Terzić et al., (2017) investigated the mechanochemical activation of zeolites in the ultra-centrifugal mill, and found that after 30 minutes of grinding 38.8 % of the raw material, (- 5 + 0) µm, was obtained.

By comparing the obtained results with these from literature it follows that the vibrating mill with rings proved to be a very good solution when ultra-fine micronizing grinding (- 5 +0) µm, is needed.

In Figure 3, a microscopic analysis of the samples before and after grinding were shoved. In Figure 3 c-d, it can be noticed that after micronizing grinding of 120 and 900 seconds the finest particles were formed aggregates.



a) b)

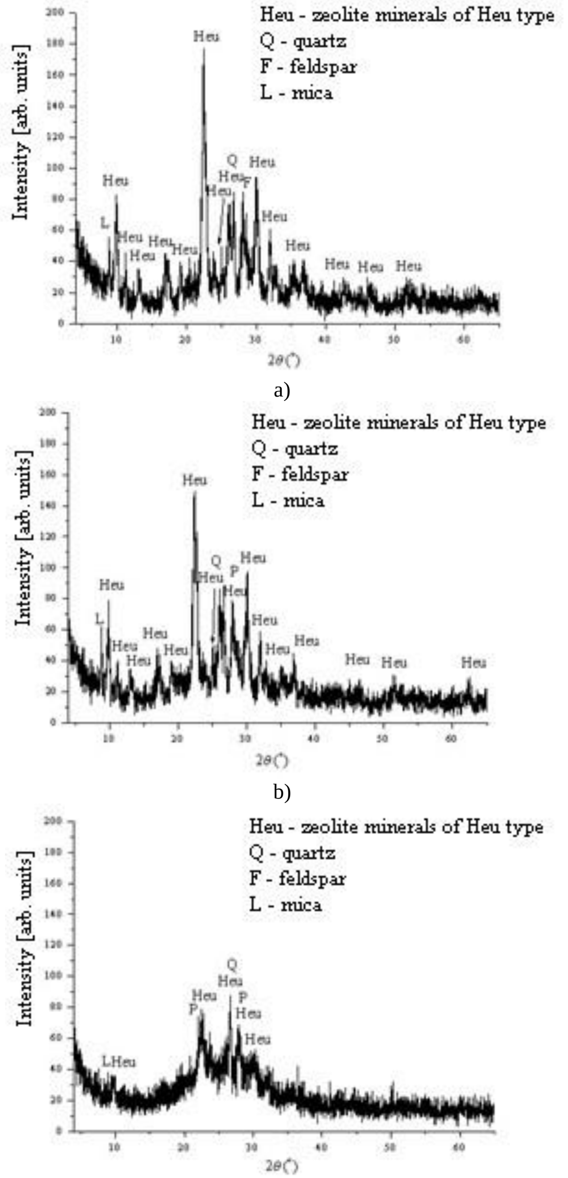
c) d)

Figure 3. Microscopic image of zeolite samples before and after micronizing grinding: a) the initial sample obtained with the x10 magnification; b) the micronizing grinding zeolite sample after 45 seconds obtained at x100 magnification; c) the micronizing grinding zeolite sample after 120 seconds obtained at x100 magnification; d) the micronizing grinding zeolite sample after 900 seconds, obtained at x100 magnification

When comparing the microscopic view of the initial sample of the class (- 3.35 + 0) mm with microscopic images obtained after micronizing grinding of (45, 120 and 900) seconds, it can be noticed that the particles in initial sample has a cubic shape, and after micronization, the majority of the particles changed shape in a spherical, elliptical or irregular shape. The formed agglomerates have a plate shape, which can be seen at Figure 3 c-d.

4.2. XRD analysis

The redgene assay was tested on the initial sample (Figure 1), and on a samples with the starting mass of (50 and 200) g after micronizing grinding (Figure 4 and Figure 5).



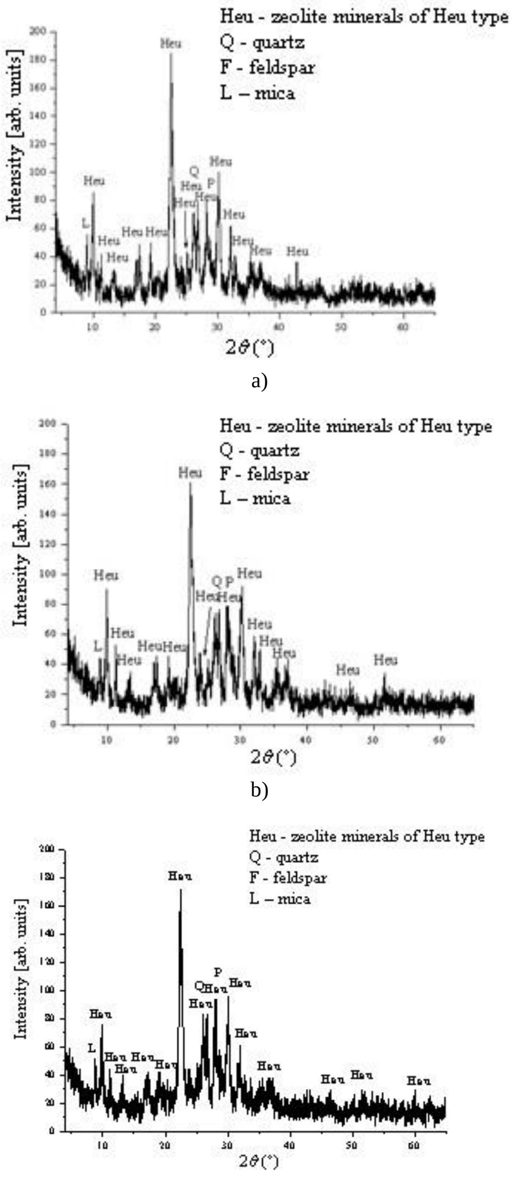
a)

b)

c)

Figure 4. XRD analysis of the micronized sample: a) 45 seconds; b) 120 seconds; c) 900 seconds

Figure 4 shows XRD analysis of the micronized sample with a starting mass of 50 g after micronizing grinding process (45, 120 and 900) seconds. It can be noticed that after 45 seconds of grinding the zeolite mineral content has not decreased in relation to the initial sample, and the effects of the amorphization due to grinding are not visible. For micronized sample which was obtained after 120 seconds of grinding, the zeolite mineral content begins to decrease, and the effects of grinding amorphization are poorly expressed. With an increase in grinding time 900 seconds, on the obtained sample, the effects of amorphization due to grinding are very pronounced, and the zeolite minerals in crystalline form are practically in the trace and the largest part is amorphous.



a)

b)

c)

Figure 5. XRD analysis of the sample micronized: a) 45 seconds; b) 120 seconds; c) 900 seconds

Figure 5 shows XRD analysis of the micronized sample with a starting mass of 200 g after micronizing grinding process (45, 120 and 900) seconds.

Figure 5 shows that a micronized samples with 4 times large mass, which were obtained after 45 seconds and after 120 seconds of grinding, have the same zeolite minerals content as well as a sample with smaller mass. The effects of amorphization due to grinding are not visible too. This shows that the sample mass has no effect on the mineralogical composition of the zeolite if grinding takes place in a short time interval. With an increase in grinding time 900 seconds, on the obtained sample, the effects of amorphization due to grinding are not visible unlike a sample of 4 times less mass.

4.3. Reactivity of zeolite during micronizing grinding

By correlation of the operating parameter of the vibratory disc mill and the zeolite micronizing grinding product parameter, the functional dependence of the specific surface area on the micronizing grinding time was formed. This functional dependence shows that as the micronizing grinding time increases, the specific surfaces in all experiments increase to the limit value upon which over time passes into the stationary level, which corresponds to the balance in the ultra-fine micronizing milling.

The results of the functional dependence of the specific surface from the time of micronizing grinding are shown in Figure 6. This analysis also confirms the visual conclusion that after a very short micronization grinding time, particle agglomeration occurs, i.e. the specific surface area decreases.

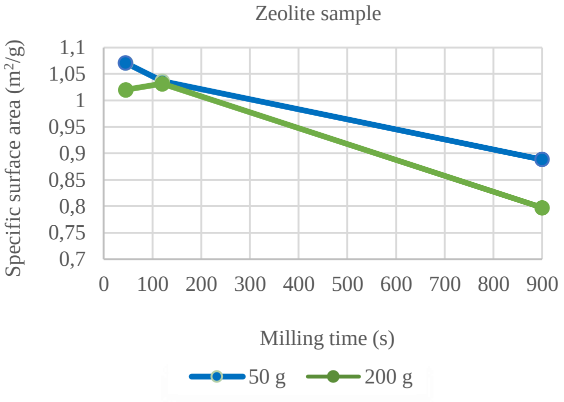


Figure 6. Change of the zeolite specific surface during micronizing grinding

Based on the above, it can be said that the specific surface area parameter is a very important indicator of the micronizing grinding efficiency.

5. Conclusion

Based on experimental research and experimentally achieved results, it can be concluded that the study of micronizing grinding zeolite in the domain of modern technologies is not a simple task, it is complex and very important.

Zeolites after micronizing grinding have wide application, and are especially used as adsorbents, due to the significant power of sorption of metal ions, radionuclides, micotossine. Thanks to their physico-mechanical and physico-chemical properties, as well as their composition and structure, zeolites are also used as ion exchangers, and have been shown to be very powerful detoxicans and antioxidants because of significant capacity to deactivate free radicals.

Based on experimentally achieved results and their analysis, the following conclusions can be made:

- It is important to select a sieving procedure to avoid particle agglomeration. The sieving of micronized products of zeolite should be carried out wet.
- For a sample of four times larger initial mass 200 g it is need about 2.5 times longer grinding time to achieve the maximum content of the class (- 5 + 0) μm.
- For a sample of the bulk class (- 3.35 + 0) mm with a starting mass of 50 g the effects of amorphization due to micronizing grinding (after 900 seconds) are very pronounced, and the zeolite minerals in crystalline form are practically in the trace and the largest part is amorphous.

In this work, complex experimental investigations of the micronizing grinding of zeolite have enabled:

- To determine the kinetic parameters of the vibratory mill with rings in the micronizing grinding process, which influence the appearance of structural defects and the reactivity of zeolites, which can be efficiently used to rationalize process of grinding and the obtaining of high quality zeolites.
- To determine the optimum values of all the most important technological and technical parameters (particle size distribution, mean particle diameter and particle shape, specific surface areas, etc.).
- To establish the correlation of the change in the specific surface and the reaction capacity, determine the changes in the specific surface energy that have a major influence on the flow of the micronizing grinding of the zeolite.

Generally, it can be concluded that a vibrating mill with a rings proved to be a good device for efficient ultra-fine micronizing grinding.

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Mikronizirajuće mlevenje zeolita u vibracionom mlinu

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I Z V O D

Suvo mikronizirajće mlevenje mineralnih sirovina je vrlo složen proces i zavisi od čitavog niza parametara kao što su: oblik, krupnoća čestica, granulometrijski sastav polaznog i mikroniziranog materijala, tvrdoća, vlažnost, gustina, svojstva površina, konstruktivne karakteristike mlinova itd. Da bi se dobila što jasnija slika procesa suvog mikronzirajućeg mlevenja, neophodno je dobro poznavanje teorijskih principa rada visokoenergetskih mlinova.

Klasični mlinovi nisu pogodni za mikronizaciju finih i posebno veoma finih čestica (gornja granična krupnoća čestice manja od 5 μm), koja je neophodna za savremene domene industrijske primene. Intenzivna fundamentalna i razvojna istraživanja procesa suvog mikronizirajućeg mlevenja za razvoj novih materijala omogućila su i razvoj novih konstrukcionih rešenja za ultra fine visokoenergetske mlinove - mehano-aktivatore. Ovi mlinovi su našli primenu u najsavremenijim tehnologijama koje se bave proizvodnjom novih profitabilnih materijala. Za dobijanje mikronskih čestica - prahova, koriste se mehano-aktivatori i ostali mikronizeri-dezintegratori, koloidni (perl-atritori), vibracioni, planetarni, ultra-centrifugalni, Jet-strujni mlinovi i dr., koji rade na principu udarnog dejstva, udara i trenja.

U radu su prikazani eksperimentalno postignuti rezultati suvog mikronizirajućeg mlevenja zeolita u vibracionom mlinu sa prstenovima, kao i poboljšanja njegove reaktivnosti. Svi eksperimenti mikronizacije su se izvodili na prethodno pripremljenoj široj klasi krupnoće (- 3,35 + 0) mm sa različitim polaznim masama (50 g i 200 g), i u različitim vremenskim intervalima (45, 120, 900 sekundi), kako bi se ispitao uticaj ovih parametara na proces mikronizacije. Proizvodi mlevenja su praćeni preko granulometrijskog sastava, sadržaja klase (- 5 + 0) μm i preko specifične površine. Pokazalo se da je za uzorak zeolita koji ima četiri puta veću masu u poređenju sa inicijalnim uzorkom, potrebno duže mlevenje za postizanje maksimalnog sadržaja klase (- 5 + 0) μm, ali i da kod uzorka zeolita klase (- 3,35 + 0) mm sa manjom početnom masom su efekti amorfizacije usled mikronizirajućeg mlevenja vrlo jako izraženi, a minerali zeolita u kristalnom obliku praktično su u tragu, odnosno najveći deo je amorfizovan. Generalno, može se zaključiti da se vibracioni mlin sa prstenovima pokazao kao dobar uređaj za efikasno ultra fino mikronizirajuće mlevenje.