The influence of composition, microstructure and firing temperature on the density, porosity, and shrinkage of new zeolite-alumina composite material

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Abstract

In this research work, new zeolite-alumina composite materials were prepared by mechanical activation and reactive sintering method. A detailed investigation of structure and microstructure of the raw materials and the produced ceramic samples have been precisely examined using X-ray diffraction (XRD) and scanning electron microscopy (SEM). Based on the raw material compositions, microstructure, and firing temperature, different characteristics were examined including, density, shrinkage, porosity and water absorption. The authors have found that the compositions and the firing temperature play a major role in the microstructure and the properties of the final product.

Keywords: zeolite-alumina ceramics, composite materials, mechanical activation, reactive sintering.

Kulcsszavak: zeolit-alumínium-oxid kerámia, kompozit anyagok, mechanikai aktiválás, reaktív szinterezés.

1. Introduction

The research work in the field of ceramics technologies has been highly grown in the recent years [1-34]. Among ceramics materials, zeolites which represent a class of crystalline minerals have been highly utilized in a wide range of applications including chemical and petrochemical industries for catalysis, adsorption and the elimination of heavy metal ions from industrial wastewaters. Zeolites offer fascinating properties like high surface area, ion-exchange capacity, high adsorption efficiency and the ability to act as a host in preparation of nanocomposite materials [35]. These outstanding characteristics assigned to the unique structure of zeolites which contain a large number of uniformly distributed micropores. The research work in zeolite is motivated not only by the need to synthesise new materials with expected potential applications [36]. But also the necessity to understand the formation of these interesting materials and to control their microstructure [37]. Zeolite-based composite materials have attracted a great deal of interest as technical materials due to

the interesting complex composition and good mechanical behaviour [38]. Despite the huge research works conducted on zeolite-based composite materials [39-43], view previous works have been conducted concerning the influence of ceramic reinforcement in zeolite-based ceramic composite [44-46].

In this research study, zeolite-based Alumina ceramic composite materials were produced through mechanical activation and reactive sintering technique. The structure and microstructure of the raw materials and the final product have been investigated via X-ray diffraction (XRD) and scanning electron microscopy (SEM), moreover, the effect of composition, firing temperature on the density, porosity, and shrinkage was studied. These new zeolite-alumina mixtures

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which relatively inexpensive materials could be a potential candidate for many technical applications showing better properties.

2. Experimental method

2.1. Synthesis of Zeolite - alumina composite materials

Zeolite-based alumina composite ceramic materials were prepared through the mechanical activation and reactive sintering method. Zeolite powders from Tokaj region (Hungary) and Al_2O_3 powder (98 %; MOTIM) were used as raw materials. An appropriate amount of these ceramic powders was carefully weighted using different compositions as shown in Table 1, then the prepared mixtures were milled and mixed in Retsch PM 400 planetary ball mill for 15 minutes in 150 rpm using silica balls. The milled powders then used to make cylindrical discs with a diameter about 25 mm and thickness of 10 mm using uniaxial pressing machines under a pressure of 500 MPa. The prepared specimens were sintered at different temperatures (1100°C, 1150°C, 1200°C and 1250°C) in a programmable furnace for 3 h, with a heating rate of 60 °C/h.

Zeolite %	Alumina %
100	0
90	10
80	20
70	30
60	40
50	50

Table 1 The composition of the raw materials used to prepare the mixtures
1. táblázat Az alapanyagokból készített keverékek összetétele

2.2. Characterization

The raw materials were characterized by Rigaku Miniflex II X-ray diffractometer (XRD) operated in the Bragg-Brentano geometry and the samples were scanned between 2θ of 0-70° with a scanning rate of 1°/min and a step size of 0.01016° using $\text{CuK}\alpha$ radiation ($\lambda = 1.54184 \text{ \AA}$). For the computer-based investigation, DIFFRACT measurement software was used.

The topographical feature and the surface morphology of both raw materials and the prepared ceramic samples were examined using scanning electron microscopy (SEM) (Model TM-1000, HITACHI). The prepared samples were examined under several magnification values using secondary electrons.

3. Results and discussion

3.1. Structures

The relationship between the sintered temperatures and composition of the produced ceramics is shown in Fig. 1. It is well seen that using different sintering temperatures lead to varieties on the colour of the ceramic samples as well as differences on the shrinkage of the prepared discs. These could be attributed to the fact that above 1100°C zeolite starts to recrystallize and leads to physicochemical reactions which could highly affect the microstructure of the complex ceramics structure, as well as the properties.

Fig. 1 Samples with different composition sintered at different temperature (1100 °C, 1150 °C, 1200 °C and 1250 °C)

1. ábra Az összetétel és az égetési hőmérséklet (1100 °C, 1150 °C, 1200 °C és 1250 °C) hatása a szinterelt zeolit mintákra

Fig. 2 exhibits the XRD pattern of natural zeolite. The analysis of the sample confirms the existence of different minerals, Table 2 reveals the chemical composition and the percentages of the different compounds.

Fig. 2 XRD diffractogram of the natural Zeolite

2. ábra A természetes zeolit XRD diffraktogramja

	Quartz	Cristo- balite	Montmo- rillonite	Calcite	Clinopti- lilit	Total
wt %	8.00	50.00	30.00	2.00	10.00	100.00
CaO				1.12		1.12
SiO ₂	8.00	50.00	19.13		5.79	82.92
Al ₂ O ₃			4.06		1.89	5.95
MgO			3.21			3.21
Na ₂ O			0.74		0.57	1.31
CO ₂				0.88		0.88
H ₂ O			2.87		1.60	4.47
Loss on ignition	0.00	0.00	2.87	0.88	1.75	5.50

Table 2 Mineralogical constituents, chemical composition and loss on ignition (LOI) of the natural zeolite in wt%, resulted from XRD analysis

2. táblázat A természetes zeolit ásványi XRD vizsgálata során megállapított összetevői, kémiai összetétele és izzítási vesztesége (LOI) tömeg%-ban

3.2. SEM investigation of the raw materials

Fig. 3 shows secondary electron images of the natural zeolite and alumina powders. The typical grains size of zeolite and alumina were found to be in the range of 0.5 - 25 μm and 0.5-

50 µm, respectively. While Fig. 4 shows the fractured surface of the produced ceramics samples (80% zeolite-20% alumina) sintered at 1100 °C and 1200 °C, it is clear that increasing the firing temperature leads to reduce the pores of the produced ceramics hence increase the density.

Fig. 3 SEM images of a) natural zeolite and b) alumina powders
3. ábra A természetes zeolit a) és az alumínium-oxid por b) mikroszerkezete (SEM)

Fig. 4 SEM images of the fractured surface of 80% zeolite-20% alumina sintered a) at 1100 °C, b) at 1200 °C
4. ábra Az 1100 °C-on a) és 1200 °C-on b) szinterelt 80 m% zeolit és 20 m% alumínium-oxid felhasználásával készült minták töretfelülete (SEM)

The composition and sintering temperature show a substantial impact on the final density of sintered ceramic samples. The pure zeolite exhibits low density, this could be assigned to the highly porous structure of the zeolite. As the amount of alumina % increasing, the density of the produced samples is gradually increased, likewise, increasing the sintering temperature also leads to increase in the density of the prepared discs, which also confirmed by SEM (Fig. 4). Pure zeolite sintered at 1100 °C offers the lowest density about 1.6 g/cm³, on the other hand, 50% zeolite- 50% alumina samples sintered at 1250 °C displays the highest density of approximately 2 g/cm³. The effect of composition and sintering temperature is shown graphically in Fig. 5.

Fig. 5 The density of the zeolite-alumina samples sintered at different temperatures
5. ábra A különböző hőmérsékleteken szinterelt zeolit-alumínium-oxid minták sűrűsége

The firing shrinkages of the produced specimens as a function of zeolite composition prepared at different sintering temperatures (1100 °C, 1150 °C, 1200 °C and 1250 °C) are shown in Fig. 6. 50% zeolite- 50% alumina samples fired at 1100 °C exhibits the lowest shrinkage of approximately 5%, with decreasing the % of alumina in the sample, the firing shrinkage is progressively increased, furthermore, increasing the sintering temperature is highly increase the firing shrinkage. Pure zeolite sintered at 1250 °C shows the highest firing shrinkage about 23%.

Fig. 6 The bulk shrinkage of the zeolite-alumina samples sintered at different temperatures
6. ábra A különböző hőmérsékleteken szinterelt zeolit-alumínium-oxid minták térfogati zsugorodása

Fig. 7 demonstrates the weight loss of the different composition of the zeolite-alumina samples sintered at variable temperatures, with increasing the sintering temperature from 1150 °C to 1250 °C the weight is highly increased from about 6% to more than 11%. Pure Zeolite shows the highest weight loss.

Fig. 7 The weight loss of the zeolite-alumina samples sintered at different temperatures
7. ábra A különböző hőmérsékleteken szinterelt zeolit-alumínium-oxid minták tömegvesztése

Fig. 8 and 9 show the water absorption and apparent porosity of the prepared ceramic samples with different composition sintered in a range of temperature (1100 °C, 1150 °C, 1200 °C and 1250 °C) the two graphs are quite similar to each other, the two graphs exhibit low values of water absorption and apparent porosity when a large amount of zeolite and high sintering temperature are used.

Fig. 8 The water absorption of the zeolite-alumina samples sintered at different temperatures
8. ábra A különböző hőmérsékleteken szinterelt zeolit-alumínium-oxid minták vízfelvevő képessége

Fig. 9 The apparent porosity of the zeolite-alumina samples sintered at different temperatures
9. ábra A különböző hőmérsékleteken szinterelt zeolit-alumínium-oxid minták látszólagos porozitása

4. Conclusion

In conclusion, the important notice to be enlightened is that the composition and the firing temperature can highly influence the characteristics of the final ceramics products such as density, sintering weight loss, porosity and shrinkage. The data examined in

this research work could be used to control the microstructure and, hence, the properties of sintered ceramic materials such as thermal conductivity, dilatation, thermal expansion coefficient, mechanical strength and other physical, chemical and mechanical properties.

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