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## **Sintering of Fly Ash Based Composites with Zeolite and Bentonite Addition for Application in Construction Materials**

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### **Abstract:**

*Due to pozzolanic characteristics, fly ash is commonly used as a cement replacement in construction composites. Addition of natural clays with sorption ability (i.e. zeolite and bentonite) in to the fly ash based construction materials is of both scientific and industrial interest. Namely, due to the application of sorptive clay minerals, it is possible to immobilize toxic heavy metals from the composite structure. The thermal compatibility of fly ash and zeolite, as well as fly ash and bentonite, within the composite was observed during sintering procedure. The starting components were used in 1:1 ratio and they were applied without additional mechanical treatment. The used compaction pressure for the tablets was 2 t·cm<sup>-2</sup>. The sintering process was conducted at 1000 °C and 1200 °C for two hours in the air atmosphere. The mineralogical phase composition of the non-treated and sintered samples was analyzed using X-ray diffraction method. Scanning electron microscopy was applied in the analysis of the microstructure of starting and sintered samples. The thermal behavior was observed via DTA method. The influence of temperature on the properties of fly ash-zeolite and fly ash-bentonite composites was investigated.*

**Keywords:** *Sintering; Thermal behavior; Density; DTA; XRD; SEM; Construction composite.*

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### **1. Introduction**

Construction composites such as mortar and concrete are fundamental constituents of structural units within various civil-engineering objects from the residential and business facilities [1-4] to complex structures like bridges [5,6] or

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dams [7, 8], and even high-temperature operating plants [9]. The improvement of the construction composites inclines towards the increasing of the structural materials' defiance to deterioration. The other goal is lightening of the structures through reduction in raw materials quantity all the while the structure retains the same or even achieves improved performances [10]. In light of such actions, it is common to use the secondary raw materials with specific properties to replace the main components such as cement or fillers. In addition to that, there is a growing concern regarding topics related to the environmental protection [11,12]. The fly ash, as one of the most commonly utilized cement and/or filler replacements was extensively investigated in our previous studies [13-15].

Today, the application of coal combustion products is in accordance with ecological and environmental requirements provided in actual international standards and the fly ash is no longer regarded as hazardous waste, instead it is recognized as a secondary raw material. However, the presence of toxic heavy metals in fly ash composition cannot be ignored even though their abundance is minor [16,17]. Pollution induced by heavy metals is a global problem since these pollutants are non-degradable and accumulative. These minor compounds represent latent environmental threat, especially in ashes that were landfilled without additional treatment. It is their leaching tendency that might induce secondary pollution of the groundwater following the fly ash application in monolithic structures. Recently, much attention has been paid to the investigation of cost-efficient sorbents such as zeolite and bentonite [18, 19]. Owing to their sorption properties, natural clay materials can develop the ability to immobilize heavy metals and other toxic or hazardous substances within the microstructure of a composite material.

Zeolites are minerals with crystalline structure characterized by a framework of linked tetrahedra. Each tetrahedron consists of four oxygen atoms that surround a cation. This framework contains open cavities shaped as channels and cages. These voids are usually occupied by H<sub>2</sub>O molecules and extra-framework cations that are commonly exchangeable. The channels are large enough to allow the passage of guest species [20, 21]. Due to variations in structure-directing agents and Si/Al ratio there is a wide multitude of zeolites. The two natural zeolites that found the most extensive application in ion-exchange uses and as sorbents are clinoptilolite and mordenite[20].

Bentonites are naturally occurring clay mineral mixtures that are mainly composed of smectite, i.e. montmorillonite clay (at least 70 wt.%). Three types of bentonite are distinguished: (1) sodium/high-swelling, (2) calcium/low-swelling and (3) intermediate (containing both sodium and calcium ions)/moderate-swelling [22]. Bentonites are expanding type sheet silicates composed of one octahedral layer situated between two tetrahedral layers. They have a high cationic exchange capacity (Na, K, Ca, and Mg) and high specific surface area. Also, they are able to absorb interlayer water molecules by increasing the basal length [18]. The quality of bentonitic raw materials depends on a number of parameters such as chemical composition and stability, rheological and exchange characteristics, swelling behavior and adsorption abilities [23]. Due to high cation exchange capacity and high specific surface area, bentonite has been predominantly studied in terms of environmental protection as a sealing material in various landfill liners and adsorbent of toxic heavy metals and/or radionuclides [18,22-24]. Regarding its application in composite materials, bentonite can be used as a natural pozzolan and it also plays an important role in sorption of toxic elements and possibly radionuclides. The application in construction materials is justified by high content of swelling clay - montmorillonite, low permeability, good stability in comparison to other sorbents, and high sorption ability for many toxic substances and radionuclides [24].

In this paper, the compatibility of fly ash and zeolite and/or bentonite in construction composites was investigated regarding their sintering behavior and the high-temperature characteristics.

## 2. Experimental procedure

The composites were prepared as 1:1 ratio mixtures of fly ash and zeolite (composite labeled as ZLK), i.e. fly ash and bentonite (composite labeled as BLK). The chemical compositions of starting materials are given in Tab. I. The atomic emission spectroscopy technique performed by a PinAAcle 900 atomic absorption spectrometer (Perkin Elmer, USA) and X-ray fluorescence method (XRF spectrophotometer ED 2000 - Oxford) were applied in the chemical analyses. The loss of ignition (LoI) content was determined by the weight difference between room temperature and 1000 °C.

**Tab. I** Chemical composition of componential materials.

Oxide (%)	Fly ash	Zeolite	Bentonite
SiO <sub>2</sub>	58.32	66.4	60.21
Al <sub>2</sub> O <sub>3</sub>	18.88	12.8	15.25
Fe <sub>2</sub> O <sub>3</sub>	6.75	1.62	3.21
TiO <sub>2</sub>	0.57	0.11	0.15
CaO	8.71	3.33	4.04
MgO	2.30	1.11	2.00
P <sub>2</sub> O <sub>5</sub>	0.025	-	-
SO <sub>3</sub>	1.29	-	-
Na <sub>2</sub> O	0.50	0.85	1.88
K <sub>2</sub> O	1.16	-	-
MnO	0.026	-	-
CO <sub>2</sub>	0.11	-	-
LoI	1.84	13.55	12.46

The binder-free powdery mixtures (0.5 g of powder per mixture) were pressed into cylindrical “Tablets” using the uniaxial double action compression process in an 8 mm diameter tool. The tablets were produced via laboratory hydraulic press (hydraulic press RING P-14, VEB THURINGER). The pressure used in the experiment was 2.0 t·cm<sup>-2</sup> (196 MPa).

The prepared tablets were placed in an alumina boat and heated in a tube furnace (Lenton Thermal Design, Type 1600), and afterwards isothermally sintered at two temperatures: 1000 °C and 1200 °C. The sintering was conducted in the air atmosphere for 120 minutes with a 10 °C/min heating rate. The density of specimens was calculated from the measurements of the tablets’ diameter, height, and mass.

The X-ray powder diffraction measurements were performed on the composites before and after thermal treatment on a Philips PW 1050 X-ray powder diffractometer using Ni-filtered CuK $\alpha_{1,2}$  ( $\lambda=1.54178$  Å) radiation and the Bragg–Brentano focusing geometry. Measurements were conducted at room temperature (25 °C) over the 2 $\theta$  range of 10–90° with a scanning step width of 0.05° and a counting time of 1 s per step.

The thermal behavior was determined by simultaneous TG–DTA (Setsys, SETARAM Instrumentation, Caluire, France) in the temperature range between 25 °C

and 1000 °C in argon or air flow, in an alumina pan at a constant heating rate of 10 °C/min.

The morphology of the obtained composites before and after heating was characterized by scanning electron microscopy (JEOL JSM-6390 LV). The tablets were crushed and covered with gold in order to perform these analyses.

### 3. Results and discussion

The results obtained on the fly ash (LK), fly ash-zeolite (ZLK), and fly ash-bentonite (BLK) tablets before thermal treatment and after sintering are presented in Tab. II. The density values of the observed specimens showed increasing with the applied temperature. The highest densities were obtained after sintering procedures conducted at 1200 °C. Also, densities of the BLK and ZLK samples were higher than those of the samples made of fly ash (LK).

**Tab. II** Density (apparent density  $\rho_a$ ) and dimensional change ( $h$ ) of composite specimens obtained before and after thermal treatment.

T (°C)	$\rho_{LK}$ (g·cm <sup>-3</sup> )	$\rho_{ZLK}$ (g·cm <sup>-3</sup> )	$\rho_{BLK}$ (g·cm <sup>-3</sup> )	$h_{LK}$ (mm)	$h_{ZLK}$ (mm)	$h_{BLK}$ (mm)
20	1.248	1.308	1.603	7.73	7.63	6.6
1000	1.265	1.416	1.698	7.60	7.12	6.05
1200	1.622	1.667	1.938	7.07	6.11	4.95

The apparent porosity ( $P_a$ ) was determined as:

$$P_a = \frac{m_1 - m}{m_2 - m_1} \cdot 100, \% \quad (1)$$

Where:  $m$  is the mass of a dry sample (g);  $m_1$  is the mass of the water saturated sample (g), and  $m_2$  is the mass of the saturated sample measured in water (g).

The apparent porosities and total porosities (calculated as the ratio of pores volume and the volume of specimen) of the ZLK and BLK samples obtained at ambient temperature and after thermal treatments at 1000 °C and 1200 °C are presented in Tab. III. Total porosity ( $P$ ) is calculated as given in Eq. (2):

$$P = \left( 1 - \frac{\rho_a}{\rho} \right) \cdot 100, \% \quad (2)$$

The density ( $\rho$ ) is determined by pycnometer method and calculated as follows:

$$\rho = \frac{(m_1) - m}{(m_1 - m) - (m_2 - m_3)} \cdot \rho_{liq}, \text{g} \cdot \text{cm}^{-3} \quad (3)$$

Where:  $m$  is the mass of the dry pycnometer (g);  $m_1$  is the mass of the sample and the pycnometer, (g);  $m_2$  is the sum of the masses of pycnometer with water, (g); and  $m_3$  is the mass of the pycnometer with sample and water, (g).

The degree of sintering is determined as:

$$SD = \frac{P_a}{\rho}, \% \quad (4)$$

The calculated sintering degree for ZLK sample treated at 1000 °C was 0.470 %. After treatment at 1200 °C the SD of the ZLK sample reached 0.533 %. The calculated SD values for the BLK composite were: 0.539 % and 0.737 % at 1000 °C and 1200 °C, respectively. The higher degree of sintering was obtained at higher temperatures, as it was expected. The BLK composite showed higher sintering degree than ZLK.

**Tab. III** Apparent porosity ( $P_a$ ), total porosity ( $P$ ), and sintering degree (SD) of composite specimens obtained before and after thermal treatment.

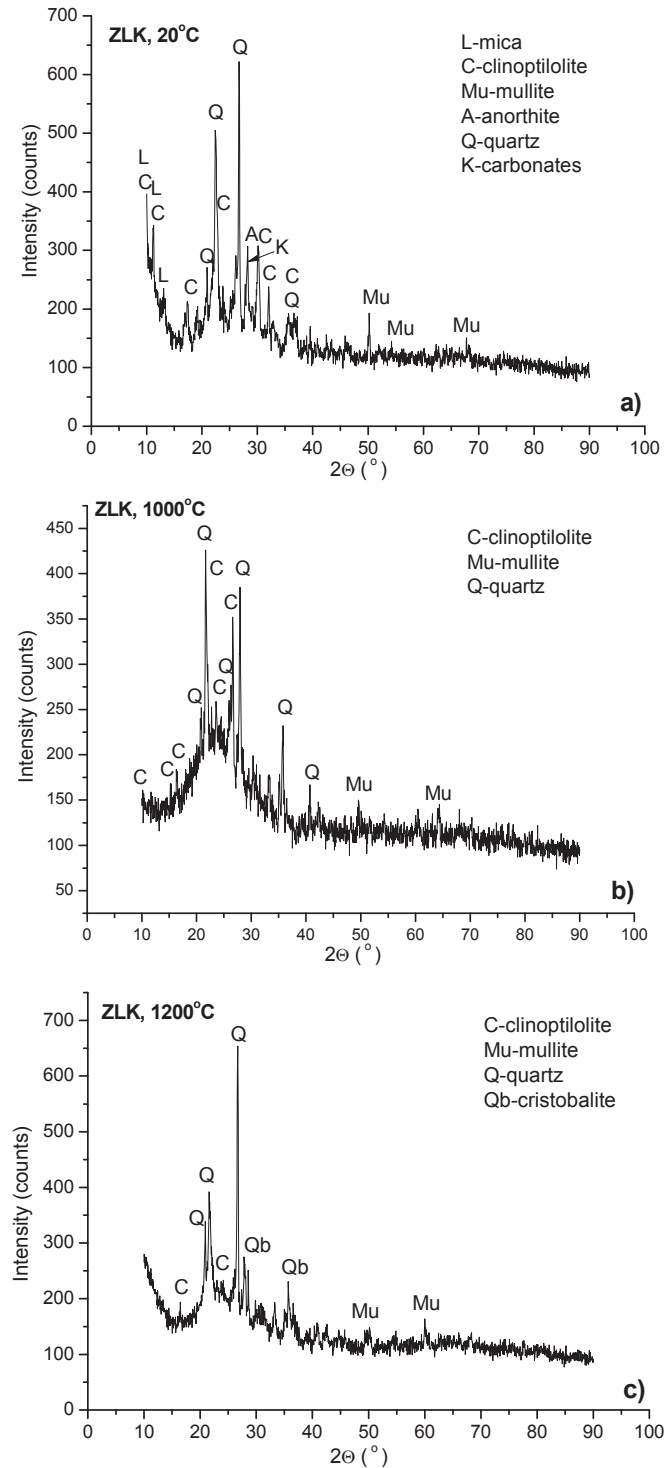
T (°C)	$P_{a\text{ZLK}}$ (%)	$P_{a\text{BLK}}$ (%)	$P_{\text{ZLK}}$ (%)	$P_{\text{BLK}}$ (%)	$SD_{\text{ZLK}}$ (%)	$SD_{\text{BLK}}$ (%)
20	18.21	15.25	26.39	22.32	-	-
1000	16.51	11.36	20.15	18.42	0.470	0.539
1200	10.21	9.05	15.24	11.57	0.533	0.737

The XRD patterns obtained on the fly ash-zeolite (ZLK) and fly ash-bentonite (BLK) samples at ambient temperature (a), and after sintering procedures that lasted two hours at 1000 °C and 1200 °C (b and c) are given in Figs. 1 and 2.

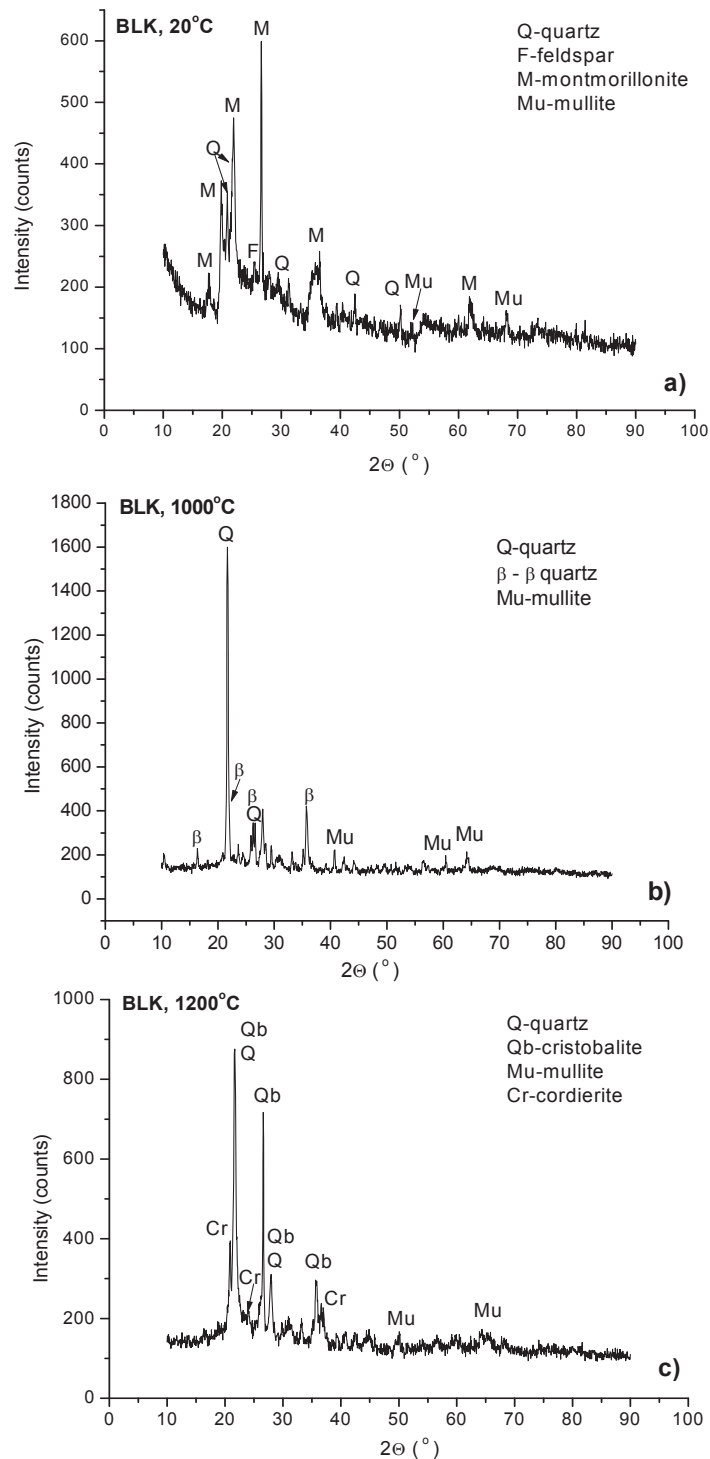
The crystalline phases that appeared in the mineralogical compositions of both composites (ZLK and BLK) were quartz ( $\text{SiO}_2$ ) and mullite ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ ), and they are characteristic for the fly ash. Both of these phases are able to sustain high temperatures. Calcite, magnetite, hematite, fluorite, and anhydrite were present in negligible, almost untraceable amounts, and they also originate from the ash [17]. The most of the peaks that belong to these phases were either too low for identification or they were mutually overlapped and/or covered by peaks that belong to other phases. The mineralogical structure of the fly ash, regarded as the base of the investigated composite, consists of both crystalline phases and certain amount of amorphous phase (aluminosilicate glass) [13,17]. The glassy phase is characterized by the baseline arch situated between 10° and 40° in all X-ray spectrums that illustrated in Fig. 1 and 2. Furthermore, the baselines of ZLK and BLK samples' diffractograms obtained both at ambient temperature and after sintering are elevated. The elevated baseline indicates the presence of certain amount of amorphous material. In ZLK and BLK samples, the position of baseline is not changing with temperature, namely it remains positioned at approximately 100-150 counts on the diagrams a), b) and c). This means that the increase of the temperature does not induce melting and creation of additional amount of glassy phase.

As it can be seen in Fig. 1a, at the ambient temperature ZLK sample contains the fly ash mineral crystalline phases (quartz and mullite), and the most prominent zeolite phases: clinoptilolite, mica, anorthite and carbonates. Clinoptilolite, which represents the basic zeolite mineral, is the most abundant mineral phase in the investigated sample. Clinoptilolite represents a microporous arrangement of silica and alumina tetrahedra with a complex formula:  $(\text{Na,K,Ca})_{2-3}\text{Al}_3(\text{Al,Si})_2\text{Si}_{13}\text{O}_{36} \cdot 12\text{H}_2\text{O}$  [25]. It is shaped as a group of monoclinic tectosilicate crystals [26]. Melting point of clinoptilolite is at approximately 1300 °C; therefore it is identified on the diffractograms at both sintering temperatures. The content of zeolitic mineral phases is at least 40 % of all crystalline phases (the starting zeolite sample had zeolitic mineral content over 75 %). Small amounts of mica that were found in the ZLK composite (Fig. 1a) originate from the zeolite. Melting point of mica is usually situated in the interval from 700 °C to 1000 °C, thus mica is not present in the sintered samples (Fig. 1b and 1c). Carbonates (i.e. various calcites) were present in low amounts at ambient temperature. Anorthite, as a form of feldspar, is also present only at ambient temperature. At 725 °C anorthite transforms into zoisite, kyanite and liquid + vapor or quartz [27]. The quartz

was identified in the ZLK sample treated at 1000 °C (Fig. 1b). At approximately 1200 °C quartz transforms into cristobalite (Fig. 1c).



**Fig. 1.** XRD patterns of the fly ash-zeolite samples: a) at ambient temperature, b) after sintering at 1000 °C, and c) after sintering at 1200 °C.



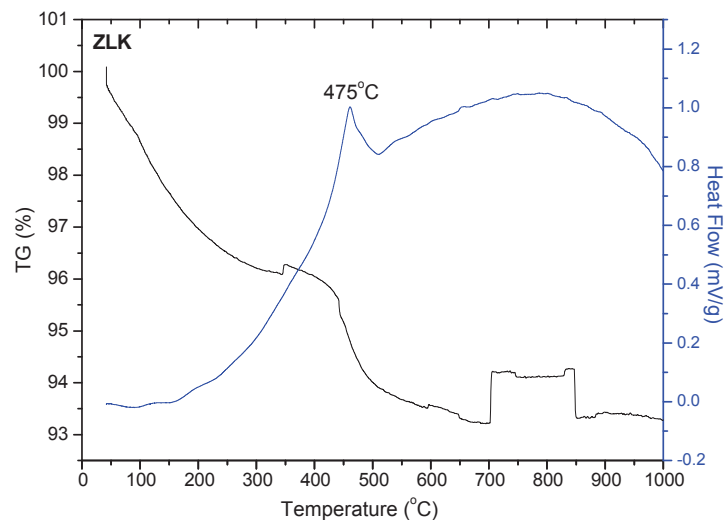
**Fig. 2.** XRD patterns of the fly ash-bentonite samples: a) at ambient temperature, b) after sintering at 1000  $^{\circ}$ C, and c) after sintering at 1200  $^{\circ}$ C.

Quartz and mullite that originate from fly ash are also present in BLK samples at all temperatures (Fig. 2). The original bentonite sample contained smectite minerals (mostly montmorillonite), quartz and feldspars.

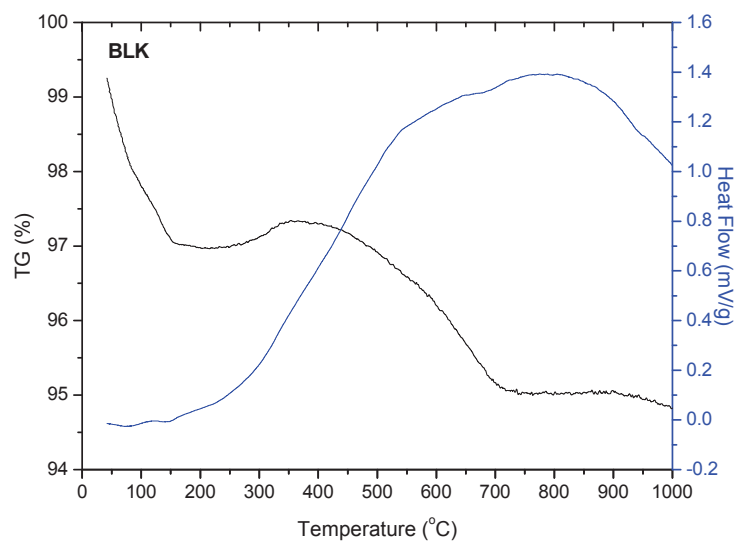
The montmorillonite structure in the samples is preserved up to 850-900  $^{\circ}$ C, where it is swiftly lost during a short temperature interval of approximately 50  $^{\circ}$ C [28]. The first high



temperature phase that succeeds montmorillonite is  $\beta$ -quartz. Beta quartz appears between approximately 900 °C and 1000 °C. It develops during temperature interval that is 50-125 °C higher than that during which the loss of the montmorillonite structure occurred. During the mentioned transition interval the samples show no X-ray diffraction effects [28]. The beta quartz develops rapidly. The increase in the quartz diffraction intensity is noticeable. Namely, the diffraction data for quartz indicate that the peaks promptly increase (Fig. 2b). Beta cristobalite usually appears abruptly at 1100 °C and from that point this mineral phase develops rapidly [28]. The beta quartz phase disappears as the cristobalite develops, indicating a phase inversion. In the BLK sample (Fig. 2c), the development of cristobalite began during 1000-1200 °C interval, namely it was initiated before quartz completely disappeared. Cordierite normally appears at 1200-1300 °C. At the very same temperature cristobalite begins to disappear. The cordierite diffraction effects increase in intensity as those of cristobalite decrease. Small cordierite peaks are registered in the Fig. 2c, i.e. during investigation of the BLK sample sintered at 1200 °C. Based on the crystalline phases that were identified after sintering at 1200 °C, the investigated ZLK and BLK composites are expected to fuse above 1300 °C.



**Fig. 3.** DTA/TGA curve of the ZLK sample.



**Fig. 4.** DTA/TGA curve of the BLK sample.



The processes that took place in the ZLK and BLK composites during thermal treatment up to 1000 °C were identified by means of DT and TG methods. The DTA and TGA curves obtained for the ZLK and BLK samples are presented in the Figs. 3 and 4.

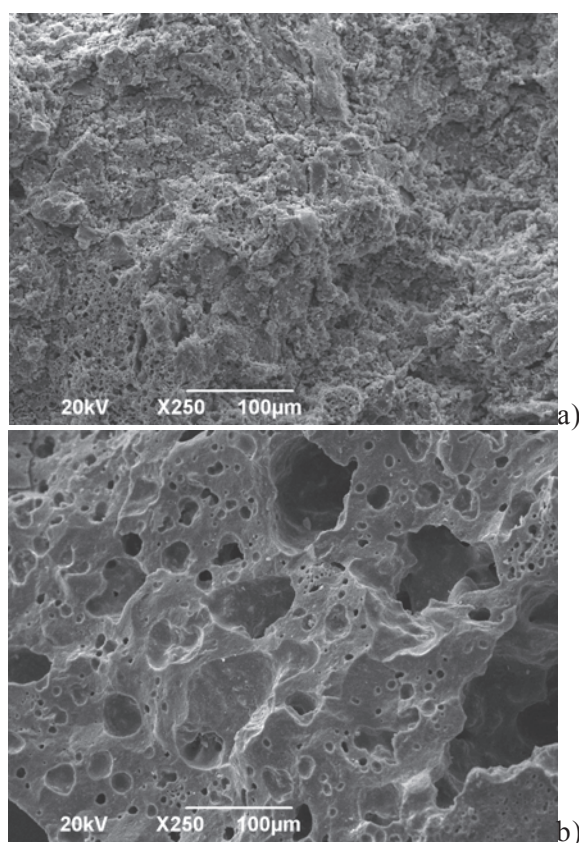
The DTA curves of the investigated ZLK (Fig. 3) and BLK (Fig. 4) samples show a constant increase during heating up to approximately 500 °C, which is characteristic for zeolite, i.e. bentonite based materials [26,28]. When fly ash was solely submitted to the differential thermal analysis [17], it showed a small peak at approximately 200 °C. Such peak corresponds to the volatilization of the water that was mechanically bonded in form of H<sub>2</sub>O molecule in the fly ash sample. However, this effect located at around 200 °C is only scarcely visible in ZLK and BLK samples. The sequences of thermal reactions are causing a complete elimination of the hydration water within the investigated composites during thermal interval from 100 °C to 450 °C. The adsorbed water within zeolite is being removed by heating up to temperature 500 °C, without significant decomposition of the structure taking place [30]. The ash structure is also preserved at this temperature. The percentage of water that is lost from the structure of zeolitic material during heating is a primary measure of its adsorptive capacity. The void volume that is left by the departed water becomes available for the adsorption of other molecules [30]. Since the bentonite is also natural clay sorber, the bentonite based composite BLK shows similar thermal behavior as ZLK sample, i.e. the similarities of their DTA curves up to 450 °C are evident in Fig. 3 and 4. The peak on the DTA curve of ZLK sample (Fig. 3) appearing at 475 °C is exothermic. The exothermic effect occurring at about 500 °C can be explained by initiation of the polymorphic transformation of  $\beta$ -quartz into  $\alpha$ -quartz [31]. The quartz polymorphs detected by XRD on the ambient temperature and after sintering at 1000 °C (Fig. 1) in ZLK sample confirm this assumption. The BLK sample which exhibited approximately the same thermal behavior as ZLK up to 450 °C (Fig. 4.), shows no peak in 450-500 °C interval. The quartz polymorphic transformation is not registered in BLK sample due to the changes in montmorillonite structure which is predominant mineral in bentonite based material [28]. Furthermore, the ZLK exhibits a broad endothermic effect between 500 °C and 600 °C which corresponds to the loss of hydroxyl water. This effect is absent in BLK sample (Fig. 4). Above 600 °C, both curves are increasing into a broad exothermic formation that probably corresponds to the mutual effect of transformation of organic matter incorporated in all starting materials, decomposition of CaCO<sub>3</sub> and the burning of residual coal present in the fly ash [32-34]. The wide exothermic hump that is located at approximately 800 °C is additionally intensified by further transformation of beta quartz. In the BLK sample, the curve's slope goes into an endothermic effect starting from 850 °C, which is the temperature when the structure of the montmorillonite starts to decompose. The results of DT analysis led to the conclusion that composites were thermally stable at temperatures up to 1000 °C and that applied componential materials are compatible. The changes that occurred above 900 °C, point out to the structural rearrangements and the initiation of sintering process.

The most of the effects detected on the TGA diagrams are related to the loss of humidity. The first dimensional changes registered by TG analyses are located between room temperature and approximately 300 °C. For the ZLK sample, the zone of the first dimensional changes comprises wider interval (Fig. 3) than for the BLK sample (Fig. 4). Namely, the fly ash contains certain additional amount of moisture that evolves during the heating procedure. This is also typical for zeolite minerals [35].

The TG curves showed more or less a continuous weight loss during heating up to ~700 °C due to the loss of water. As it can be deduced from Figs. 3 and 4, several smaller mass loss steps have been obtained in the temperature range from 25 °C to 700 °C: 1) 25 to 100 °C - the weight loss due to the hygroscopic water desorption; 2) 100 to 200 °C - the rapid weight loss of the ZLK sample documented by the steep slope of the TG curve which is attributed to the loss of loosely bonded water. During this interval the line of the BLK

diagram is almost horizontal, i.e. the mass of the sample remains unchanged; 3) 200 to 300 °C – the weight loss of the ZLK sample is smaller which is indicated by the slighter slope of the TG curve. BLK sample shows no mass change; 4) 300 to 400 °C – a gradual weight increase is present in both samples; 5) 400 to 500 °C - the slope of the TG curve regains light decrease, i.e. ZLK and BLK samples show weight loss; 6) 500 to 700 °C – period when remaining water is gradually removed [26, 35]. The TG curve obtained on the BLK sample shows an endothermic effect near 100 °C which may be due to water that is adsorbed onto the external surface and/or coordinated to exchangeable interlayer cations and adsorbed between the clay mineral layers [36]. The exothermic hump around 400 °C (Fig. 4) can be correlated with structural rearrangements that are typical for smectite minerals (i.e. montmorillonite) [37]. The effect on the TG curve of ZLK sample (Fig. 3) located between 700-850 °C is strong indication of structural rearrangement, and it can be correlated for example with mica and/or carbonates decomposition.

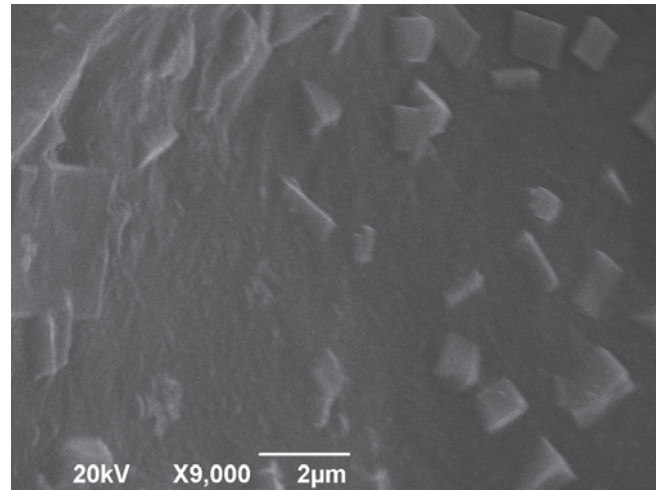
Fig. 5 shows the SEM micrographs of the fly ash-zeolite (ZLK) composites before and after applied thermal treatment.



**Fig. 5.** SEM images of the ZLK composites: a) before sintering, b) after sintering at 1200 °C.

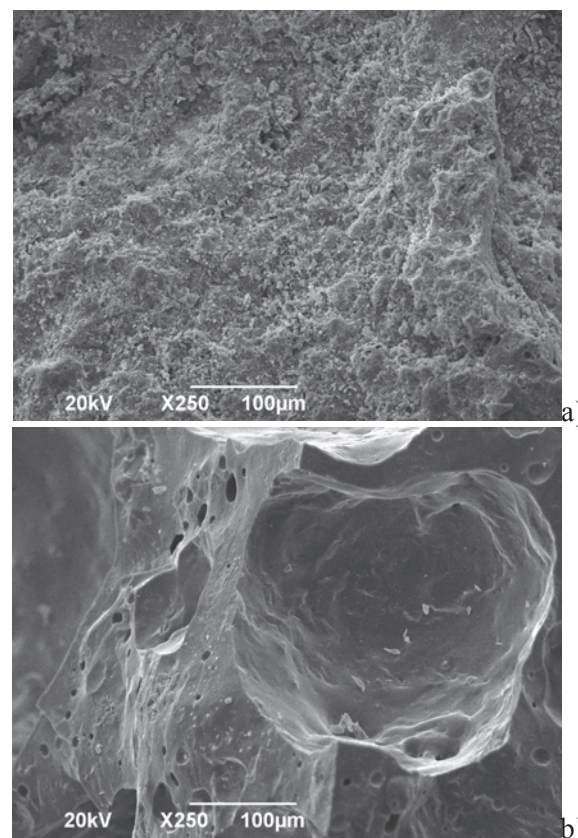
The fly ash-zeolite composite (Fig. 5a) is a mixture of a variety of particles that are characterized by different sizes and different shapes. Diversity in particles' appearance is a consequence of participation of different inorganic phases in the mineralogical structure of the composite. The fly ash grains are mainly spherical and hollow. The superficial porosity of all ash grains is accentuated. The visible pores are commonly round shaped. The fly ash grains consist of particles of different shapes, sizes, and textures. Namely, large part of particles are spherical, however irregularly shaped particles are also present. The fly ash, together with zeolite mineral grains, forming clusters and agglomeration of particles. Mullite needle like particles, and quartz and cristobalite irregularly shaped particles are immersed in the mass of

ash-zeolite mixture. Due to the compression applied to ZLK tablets it is hard to distinguish actual grains, but from the SEM image analysis it can be said that pseudospheres (spherical particles composed of various layers of solid matter and numerous external and internal pore channels and voids) belong to fly ash, while the rectangular particles (Fig. 6) are characteristic for the zeolite mineral – clinoptilolite.



**Fig. 6.** SEM image of ZLK sample detail with visible clinoptilolite particles (after sintering at 1200 °C).

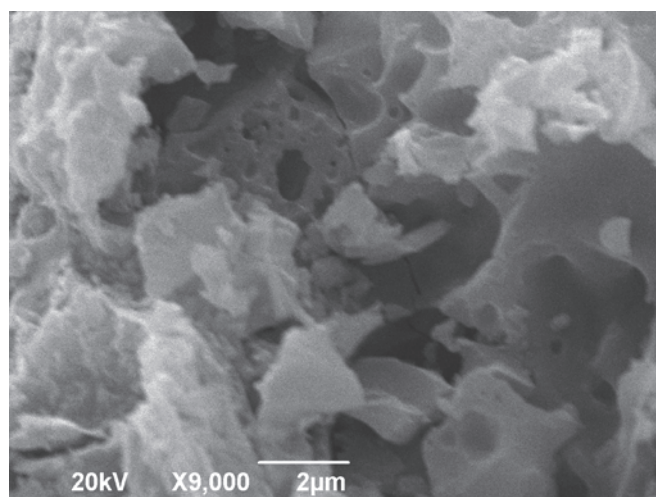
Fig. 7 shows the SEM micrographs of the fly ash-bentonite (BLK) composites before and after applied thermal treatment.



**Fig. 7.** SEM images of the BLK composites: a) before sintering, b) after sintering at 1200 °C.



Same as the ZLK composite, the fly ash-bentonite sample (Fig. 7a) is a mixture of grains and particles that are characteristic for ash and for clay as the two main constituents of the mixture. Aggregates and clusters that are formed within the mixture during preparation of the testing tablets contain various regularly and irregularly shaped particles of different inorganic origin. The structure of BLK sample observed at the ambient temperature show high resemblance to ZLK sample due to the presence of similar mineral phases (quartz, mullite, and feldspar/anorthite). The stacks of plate-like platelets in montmorillonite incorporated with larger irregularly shaped quartz particles and pore spaces can be seen in SEM image of the BLK sample (Fig. 8.).



**Fig. 8.** SEM image of a detail of BLK sample (at ambient temperature).

With increasing temperature the powdery particles of both composites (Fig. 5b and 7b) are getting closer, forming thicker and more solid agglomerations. It is clearly visible that samples sintered at 1200 °C have more solid base structure and an increased number of mutual contacts. The pores widened and the shapes of single particles are not so distinctly visible any longer. The BLK sample sintered at 1200 °C has lower porosity than ZLK sample which is in consistency with calculated results of porosity and sintering degree. The BLK sample has more solid structure and it appears that it has sintered more rapidly. Also, at 1200 °C there are neither glassy inclusions nor particle cluster delaminations which highlights that the used materials (i.e. fly ash, zeolite and bentonite) are thermally compatible and can be applied in a construction composite exposed to the influence of high temperature.

#### 4. Conclusions

The results of this study showed that fly ash and zeolite and/or bentonite applied in the construction composites were compatible regarding their sintering behavior and the high-temperature characteristics. The obtained results are presented below:

The composites density values increased with the elevation of the assigned thermal treatment temperature. The highest densities were obtained after sintering at 1200 °C. The BLK had higher density than ZLK composite. The apparent porosities of the samples decreased with increasing temperature. The higher degree of sintering was obtained at higher temperatures. The BLK showed higher sintering degree than ZLK.

The XRD analysis has shown a different mineral phase composition of the samples at ambient temperature and in the sintered samples. The quartz phase (including its temperature-dependent polymorphs) is the dominant phase in both composites. Clinoptilolite originating

from zeolite was characteristic for the ZLK sample. The montmorillonite appeared in BLK sample at room temperature. At 1200 °C montmorillonite already started its transformation into cordierite. Mullite which gives micro-reinforcement and adds the strength to the material structure with its needle-like particles was present in both composites.

The most of the effects detected on the DTA/TGA diagrams are related and explained by loss of humidity. Only the effects located at higher temperatures, i.e. above 700-850 °C are giving indication of structural rearrangement that is correlated to transition and/or decomposition of mineral phases.

The SEM micrographs indicated that with increasing temperature the powdery particles of both composites are getting closer, forming thicker and more solid agglomerations. The samples sintered at 1200 °C have more solid base structure and an increased number of mutual contacts, than samples observed at room temperature. The BLK sample has more solid structure than ZLK and it appears that it has sintered more rapidly.

Regarding the fact that at 1200 °C there are no glassy inclusions or particle cluster delaminations, it can be assumed that fly ash, zeolite and/or bentonite are thermally compatible and they can be used as a base material for construction composites exposed to influence of the temperature.

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**Садржај:** *Захваљујући својим порцеланским својствима, летећи пепео је данас најчешћа замена за цемент у конструкционим композитима. Додавање природних глина које показују сорпциона својства (нпр. зеолит и бентонит) у грађевинске материјале на бази летећег пепела је од великог научног и индустријског значаја. Наиме, захваљујући примени сорптивних глинених минерала, могуће је имобилизовати токсичне тешке метале из структуре композита. Термичка компатибилност летећег пепела и зеолита, као и летећег пепела и бентонита, у самој структури композита изучавана је у току процеса синтеровања. Полазне компоненте су примењене у односу 1:1 и то без додатног механичког*

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третмана. Примењен је притисак од  $2 \text{ t}\cdot\text{cm}^{-2}$  за припрему таблета за испитивање композита. Синтеровање је обављено на  $1000 \text{ }^\circ\text{C}$  и  $1200 \text{ }^\circ\text{C}$  у трајању од 2 сата, у атмосфери ваздуха. Минерални фазни састав нетретираних и синтерованих узорака је анализиран помоћу X-ray дифракционе методе. Скенирајућа електронска микроскопија примењена је у анализи микроструктуре полазних и синтерованих узорака. Термичко понашање праћено је помоћу DTA методе. Испитиван је утицај температуре на својства композита на бази летећег пепела и зеолита, односно летећег пепела и бентонита.

**Кључне речи:** Синтеровање; термичка својства; густина; DTA; XRD; SEM; конструкциони композити.

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