Creation of Slag-Containing Composite Material Prototypes Using Powder Metallurgy Methods

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ABSTRACT

This study explores the powder metallurgy methods for obtaining slag-containing composite materials that can be utilized in the ceramic industry, and especially in catalysis, as raw materials for the production of building materials, and also as refractories. The main components of the synthesized samples of composite materials are natural aluminosilicates from the east of Kazakhstan and metallurgical slag of lead production. Varying the content of components in the range: slag 10-30 wt.%, bentonite clay 30-40 wt.%, and natural zeolite 40-60 wt.%, a pilot batch of composite materials was obtained. The results show that the samples had high mechanical strength, ranging from 20.7 to 50.53 MPa, after sintering at a temperature of 1000 °C.

Keywords- metallurgical slag; powder metallurgy; composite materials; mechanical strength; sintering

I. INTRODUCTION

Powder Metallurgy is a modern industrial method based on the use of powders of various materials to form products of specified dimensions and subsequent heat treatment at temperatures below the melting point. This approach not only allows for the fabrication of products of various shapes and purposes, but also facilitates the creation of new materials with unique properties [1]. Mixtures of metals, alloys, ceramics, and polymers can be used as raw materials for powders [2-4], promoting the development of composites with characteristics that cannot be achieved when using only one component [5]. Composite materials have a significant role in various industries due to their possibility of creating individual properties. The combination of two or more physically and chemically bonded phases allows the creation of materials with improved mechanical, thermal, electrical, and other characteristics [6-7].

Metallurgical slags generated during ore processing and metal production represent significant volumes of raw materials that require efficient management [8]. In the context of growing environmental concerns and depletion of natural resources, there is an increased interest worldwide in the use of metallurgical slags as sustainable alternatives to traditional raw materials in various industries, which not only reduces the waste volumes, but also creates new materials with improved properties [9-10].

One of the most common areas of recycling metallurgical slag is its use in building materials. For example, slag is used to produce concrete and asphalt mixtures. Authors in [11] demonstrate that adding slag to concrete can improve its strength characteristics and durability. Authors in [12] also show that using slag in the production of lightweight concrete blocks can reduce their weight and improve the thermal insulation properties. It has been shown that adding slag to the concrete mixtures improves the strength characteristics and durability of the final product. In particular, the use of blast furnace slag can increase the resistance of concrete to aggressive environments [13]. In some cases, metallurgical slags are used as fertilizers or soil additives. For example, certain types of slag can improve the soil structure and increase its fertility due to their mineral content [14, 15]. This area is being actively researched in countries with developed agricultural sectors. Metallurgical slags are also utilized in the production of ceramic materials [16]. Research is being conducted on the use of slags to create ceramic tiles [17]. Authors in [18] demonstrated that slags can serve not only as additives, but also as active components that promote the formation of a glassy phase during firing. It has also been proposed to obtain CaO–MgO–Al_2O_3–SiO_2 (CMAS) glass ceramics using silicate-containing metallurgical slags. It has been found that a large number of metallurgical slags, including blast furnace slag, steel slag, stainless steel slag, electric furnace ferronickel slag, copper slag, ferromanganese slag, and lead fuming slag, have similarities in their chemical composition with the natural raw materials.

There has been a steady interest in the development of new composite materials that combine environmental friendliness, load resistance, and high physical and mechanical properties. The choice of reinforcing and matrix components has a great influence on the strength and performance properties of the composites [19-22]. Modern approaches to the development of such materials involve the use of both synthetic and natural components, including recycling waste and secondary resources [23]. In this regard, natural aluminosilicates, such as bentonites, zeolites and others, characterized by low cost and large reserves, deserve special attention.

Composite materials are used in a wide range of industries and engineering for various purposes due to their operational characteristics [24]. It is known about the use of blast furnace slag as reinforcing particles in the production of aluminum composites [25]. The use of steelmaking slag for the production of a tin-based composite is also reported in [26]. The use of copper slag (waste from copper extraction) for reinforcement, a metal matrix composite of 95% aluminum and 5% copper slag led to an increase in the mechanical properties compared to the base metal aluminum, and a decrease in the weight of the composite [27].

However, the utilization of lead production slags remains insufficiently studied, generating the need for systematic research to assess their chemical composition, physical properties, and possible methods of processing them. The efficient use of this raw material not only helps to solve environmental problems associated with filling landfills and potential environmental pollution, but also opens enables the creation of cost-effective and sustainable alternatives to traditional raw materials.

These data highlight the relevance of studying the structural characteristics of composites based on metallurgical slags and natural raw materials. An important aspect is the support of scientific research aimed at developing new production methods and technologies capable of meeting the modern environmental and climate challenges [28].

Previous research [29] has already considered the possibility of using metallurgical slags to create ceramic materials. The purpose of this work is to study the potential of metallurgical slags from lead production for creating new composite materials prototypes that can be used in the ceramic industry, in catalysis, as a raw material for the production of building materials, and also as refractories.

II. MATERIALS AND METHODS

In the current investigation, lead production slag was used to manufacture composite materials. Aluminosilicates represented by zeolite and bentonite (from deposits in the East Kazakhstan region) were used as natural raw materials. To conduct a set of studies on the creation of new composite materials, X-ray Diffraction (XRD) analysis was performed using an X'Pert PRO diffractometer, along with simultaneous thermal analysis methods (thermogravimetry/differential thermal analysis, TGA/DTA) on a METTLER TOLEDO device. Microscopic analysis was carried out on an Olympus BX 51 optical microscope deploying the light field method in reflected light. Based on powder metallurgy methods, including grinding, mixing, pressing, sintering, and firing by varying the components of the mixture, a pilot batch of new composite materials was manufactured. The initial components of the charge (lead slag, zeolite, and bentonite) were dried, crushed, and ground to a fraction of 0.01 mm. Then, to obtain a mass of a given composition, the slag, zeolite, and bentonite powders were mixed until a homogeneous mass was obtained in a mixer. An important characteristic is the moisture capacity of the initial components and the resulting mixture. The moisture content was varied in the range of 15–25% to ensure the required molding moisture. After mixing, the prepared batch was sent for molding either by pressing or extrusion, depending on the required shape of the product. The molding of the samples in the form of tablets was carried out by pressing on a universal Shimadzu Autograph AG-Xplus machine. The molded products were dried and sintered. The optimization of the heat treatment mode is an important aspect in the production of composite materials, since it is at this stage that the mechanical properties of the final product are formed. The original samples were pre-dried at a temperature of 100 °C in a drying cabinet after which they were sintered in a muffle electric furnace at a temperature range of 500-1000 °C. The process of moisture evaporation from composite systems during heating should proceed smoothly and continuously at a controlled rate to avoid product deformation or the appearance of swellings and cracks.

The resulting samples were tested for strength. The strength of the obtained samples was determined on a universal Shimadzu Autograph AG-Xplus machine, using TRAPEZIUM X software. The machine has a capacity of 10 kN. The ultimate strength of the samples was measured under compression.

III. RESULTS AND DISCUSSION

The properties of composite materials and microstructural changes depend on the raw materials from which they are made, the method of their processing, the composition of the batch, and the established heat treatment [24, 30, 31]. Studies have been conducted on the compositions and properties of the initial raw materials and composite systems synthesized on their basis. X-ray phase analysis was deployed to study the chemical and mineralogical compositions of the studied samples of the starting materials. The diffraction patterns were interpreted using the ICDD card index. Figures 1 (a) and (b) show that the highest intensity absorption peaks in the X-ray spectra of the aluminosilicates correspond to quartz, albite, clinoptilolite, and others.

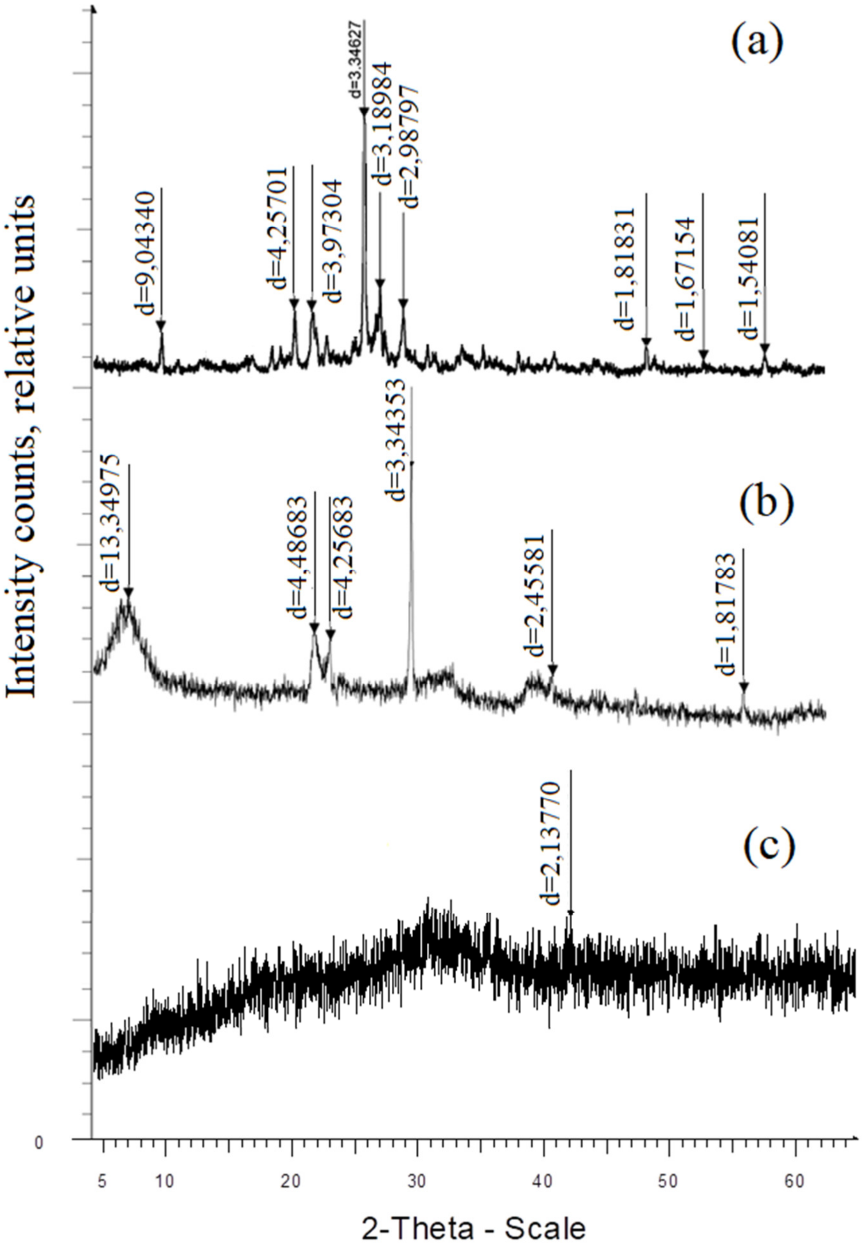
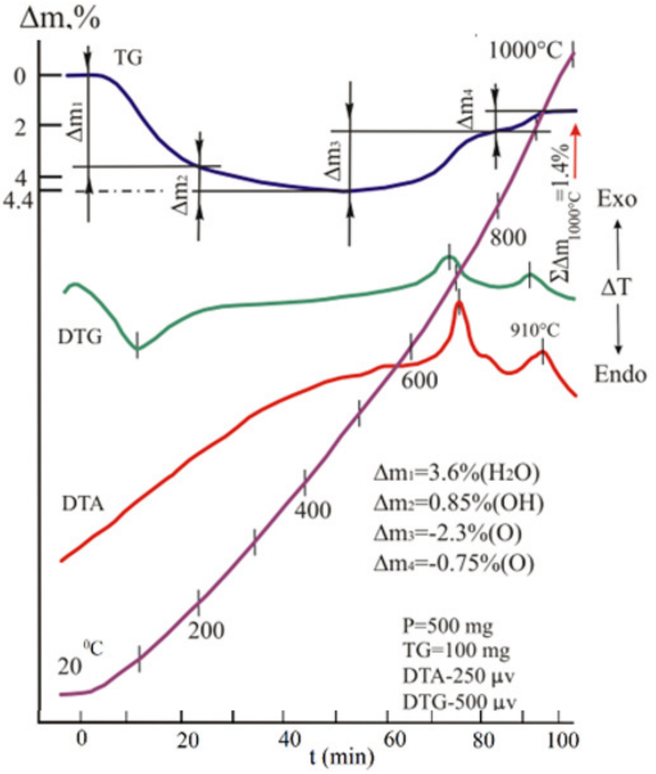


Fig. 1. XRF spectra: (a) zeolite, (b) bentonite, (c) slag.

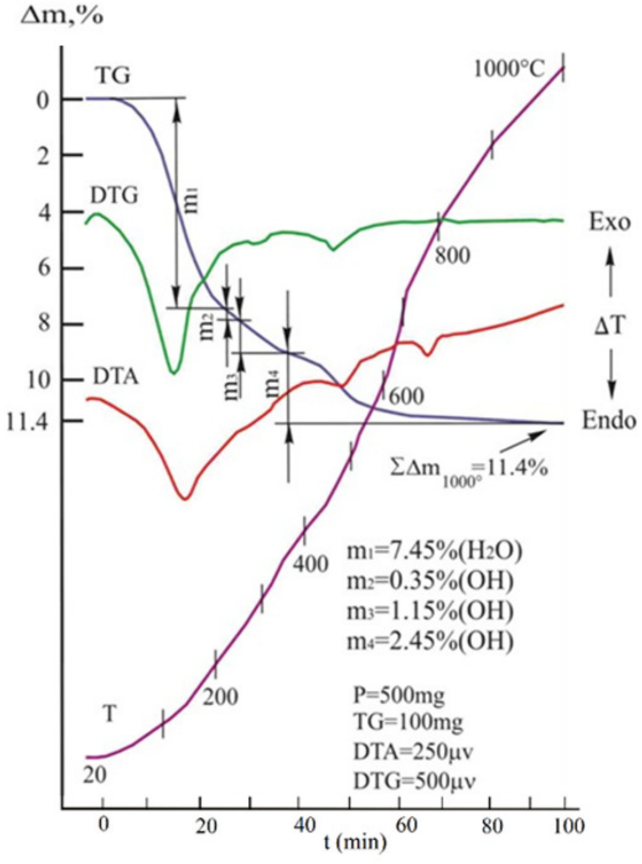
The phase composition of aluminosilicates in wt.% was: oxides SiO_2 (55.48 – 69.45 %), Al_2O_3 (13.14 – 19.38 %), Fe_2O_3 (0.96 – 4.42 %), CaO (1.96 – 1.98 %), Na_2O (0.14 – 1.99 %), etc.

The spectrum in Figure 1 (c) characterizes the mineral composition of the lead slag, showing that the sample is represented by an amorphous phase close to crystalline phases of natural origin, such as fayalite, mullite, and wollastonite. It is evident that the slag contains a significant amount of iron, silicon, aluminum, magnesium oxides wt.%: FeO-43.30 %, SiO_2-29.8 %, Al_2O_3-5.85 %, MgO-3.25 %, and others, which indicates a complex slag structure.

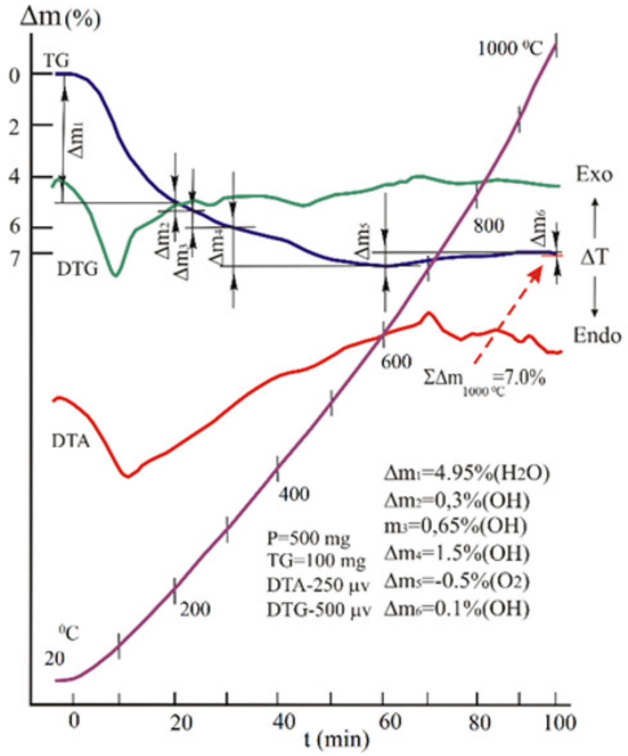
To determine the thermally active part and identify the thermal behavior of the slag samples under dynamic temperature increase, a thermal analysis, as shown in Figure 2, was carried out in the air, at a temperature range of 20-1000 °C, according to the DTA, TGA, and TG measurements. In a dynamic heating mode, the slag produced a series of effects in different temperature ranges on the DTA, DTG, and TG curves, due to endothermic and exothermic reactions, as depicted in Figure 2 (a). During heating up to 1000 °C, the curves first demonstrated the release of hydrates (H_2O, OH) and fine inclusions (20–500 °C) into the atmosphere, followed by the introduction of oxygen into the system (500–1000 °C), caused by the oxidation of metal-containing components in the sample.



(a)



(b)



(c)

Fig. 2. Spectra: (a) slag, (b) zeolite-bentonite, (c) slag-zeolite-bentonite.

The effect of adding slag to the zeolite-bentonite mixture on the properties and structure of the composite materials was evaluated next. At the first stage, the zeolite-bentonite mixture was analyzed, and the results are presented in Figure (2b). Under conditions of dynamic heating from 20 to 1000 °C, a series of thermal effects associated with the destruction of water-containing mineral inclusions are observed. Heating the zeolite-bentonite powder mixture leads to a step-by-step decomposition of its components. Within 60-220 °C, the analyzed sample loses 7.45% of its weight, then, at higher temperatures, 3.95% weight is lost.

Both bentonite and zeolite undergo low-temperature dehydration during which the zeolite loses 1.74% of its weight, and the mass of bentonite decreases by 5.71%. Both weight losses occur in the same temperature range, although the nature of their bonding in the crystalline structures is different. The thermal analyses showed a good correspondence between the content of differently bound water in the mixture and the actual quantitative composition of zeolite and bentonite. Figure 2 (c) illustrates the spectrum of the synthesized material based on the slag-zeolite-bentonite mixture. When this composite mixture was heated, lines that carried information corresponding to their material composition were formed. The slag introduced during firing played the role of chemical reagents, regulating the chronology of destruction of the clay substance and the development of new crystalline formation. It was observed that during calcination in the temperature range from room temperature to 180-200 °C, hygroscopic moisture evaporates, and then structural water is removed up to 600 °C. Due to the sintering processes, a crystalline structure is formed, providing the required mechanical strength of the composite samples from a slag-zeolite-bentonite mixture.

Along with studying the properties and behavior during heat treatment, it is also important to examine the morphology and structure of the samples. The microscopic analysis of the zeolite, as shown in Figure 3 (a), revealed that the sample has a non-uniform structure due to the presence of dense and porous inclusions (grains). The grains are brown, white, grey, and transparent. The white and grey inclusions are mainly represented by quartz, the brown (red) grains are iron-containing semi-decomposed (weathered) rock of a variable/varying composition, and transparent grains are feldspars. When the calcination temperature is increased to 1000 °C, a crystalline phase with pronounced pores is formed in the sample, indicating changes in the structure of the material.

Bentonites, as shown in Figure 3 (b), also contain quartz, feldspars, and, less frequently, mica among the rock-forming minerals. When calcined to 1000 °C, a similar crystalline phase formation with pronounced pores occurs. Figure 3 (c) displays a two-phase structural component of a zeolite-bentonite mixture. At a calcination temperature of 500 °C, the grains are uniformly distributed over the entire surface of the sample, with highly dispersed particles of about 0.01-0.04 μm predominating. When the temperature increases to 1000 °C, the structure becomes more uniform, and the color of the mixture changes to a brick shade. The samples synthesized from the slag-zeolite-bentonite mixture, as presented in Figure 3 (d), also demonstrate a heterogeneous structure with dense and porous inclusions in a fine-grained sintered base mass. The white inclusions are quartz, and the brown (red) grains are iron-containing semi-decomposed rock of a variable/varying composition. Black and grey grains have a fundamentally different composition; they consist of iron-containing metallurgical granulated slags with a silicate glass phase and inclusions of magnetite, spinel, and metallic iron.

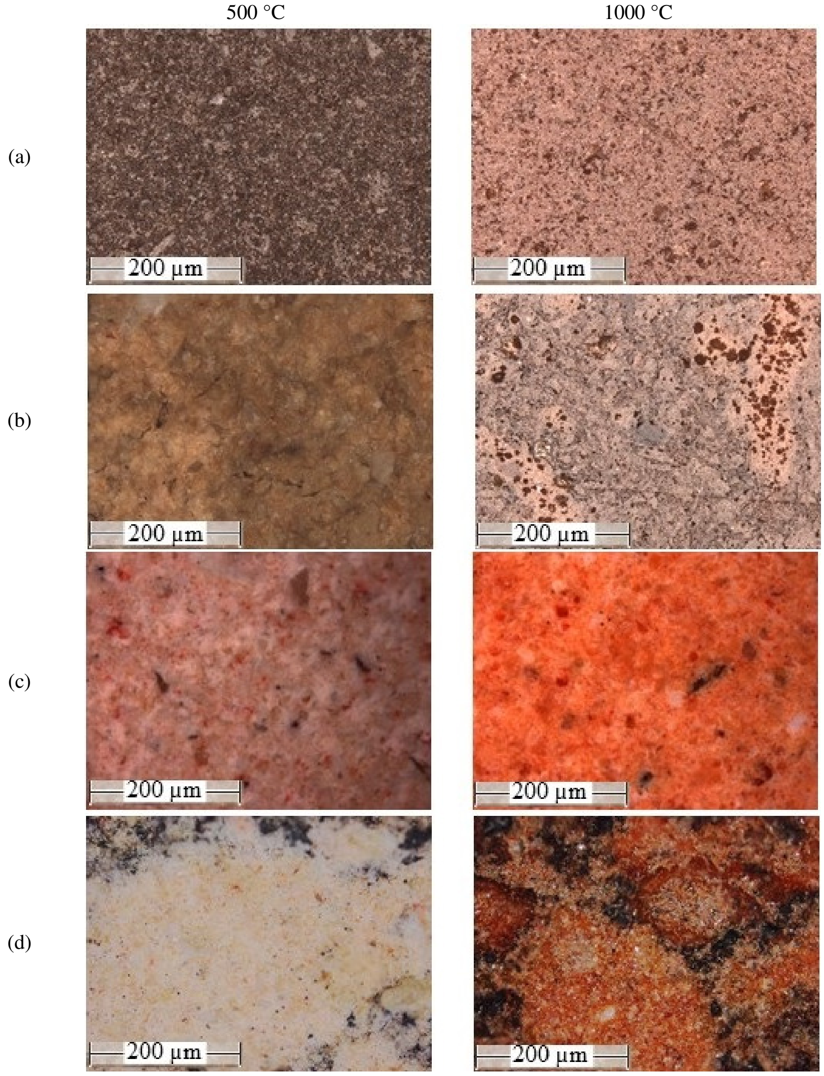


Fig. 3. Optical images obtained at 100x magnification: (a) zeolite, (b) bentonite, (c) zeolite bentonite, (d) slag-zeolite-bentonite.

When the calcination temperature is increased to 1000 °C, the basic mixture also changes color to a brick shade. After firing, the clay minerals are transformed into mullite and cristobalite, which gives the material the hardness of stone. This indicates that when melted, the clay components are transformed into quartz, the sodium into albite, and the remaining elements are transferred to the composition of mullite. Thus, the results of the microscopic analysis confirm the complex morphology of the studied materials and their ability to structural changes under the influence of temperature. These data are important for a further study of the properties of composite materials based on slags of metallurgical enterprises and natural raw materials.

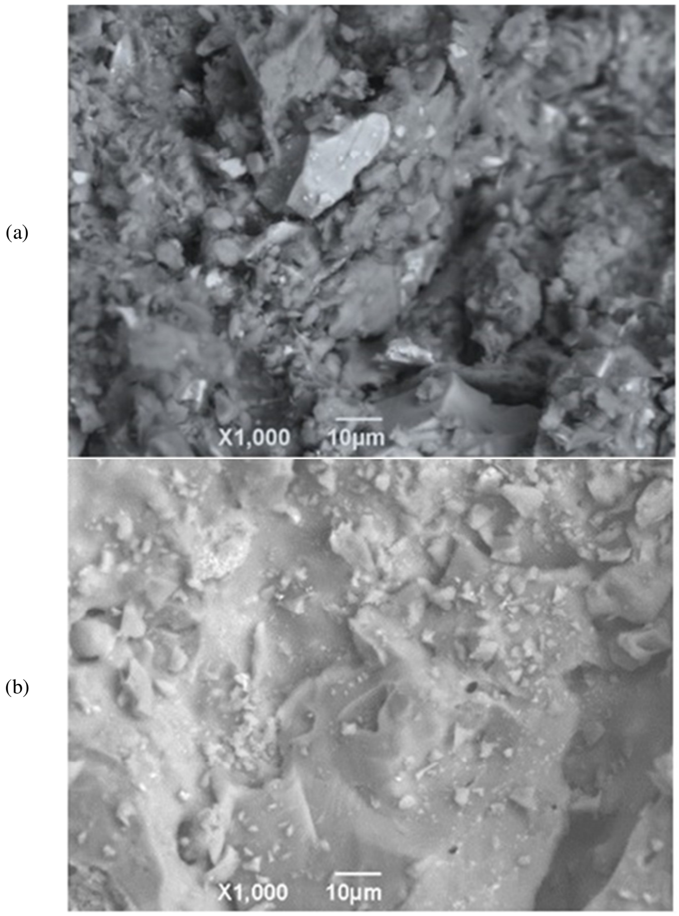


Fig. 4. SEM images of slag-containing composite material at 1000x magnification, calcined at: (a) 500 °C, (b) 1000 °C.

The Scanning Electron Microscopy (SEM) images of the slag-zeolite-bentonite mixture are shown in Figure 4. At 500 °C, the irregularly shaped lead slag grains are visible, which do not have direct contact with each other. Increasing the calcination temperature of the system from 500 °C to 1000 °C leads to the formation of a matrix microstructure with closed, round or oval pores. Also, the boundary layer contains irregularly elongated macropores. With a temperature treatment up to 1000 °C, the structure becomes more uniform and denser and molten crystals are visible, which contributes to an increase in reactivity during the sintering of the composite material. Such a matrix structure has a high mechanical strength.

An important aspect of the study was to investigate the effect of the component ratio on obtaining composite materials with different characteristics, such as porosity and mechanical strength. To assess the possibility of using metallurgical slag in the production of composite materials, a comparative analysis of the mechanical compressive strength of samples with and without the addition of metallurgical slag was performed, as presented in Table I.

The composition of the batch without the addition of slag is 50-60 wt. % zeolite and 40-50 wt. %. When adding slag, the components of the batch varied within the following limits: slag 10-30 wt. %, bentonite 30-40 wt. %, and zeolite 40-60 wt. %. When introducing slag, the bentonite content was reduced by 20 wt. %.

As a result, the composite material samples were obtained in the form of tablets with a diameter of 12.6 mm and a height of 13 mm, as presented in Figure 5. The manufactured pilot batch was tested for mechanical strength. The obtained samples were tested in a hydraulic press by squeezing a vertically applied controlled load, and the compressive strength was calculated.

Fig. 5. Samples of composite materials in the form of tablets.

Table I presents the values of the compressive strength limit during the sintering of samples at temperatures of 500°C, 750°C, and 1000°C, depending on the composition and humidity. The data analysis reveals that the changes in the batch composition affect the mechanical properties of the samples. A significant increase in the strength characteristics of the samples is observed when varying the batch composition by introducing the metallurgical lead slag. This increase reaches 2–3-fold compared to the samples without the slag addition. This effect is due to the fact that lead slag, being a rich source of metal oxides and other compounds, contributes to the formation of a stronger structure of the material due to the improvement of the binding properties. In addition, the introduction of slag promotes the activation of crystallization processes in the matrix, which further increases its mechanical stability. The influence of the bentonite composition plays an important role, a change in its concentration affects water absorption and plasticity, which, along with the addition of slag, allows optimizing the structure of the material to achieve maximum strength. The obtained data confirm the possibility of using metallurgical slags as additives for obtaining stronger ceramic composites.

The results show that the combinations with zeolite content (50%) and moderate slag content (20%) have good potential as a raw material base for the creation of durable composite systems. Based on these data, optimal compositions were determined in terms of processability and mechanical properties: composition No. 5 with a content of wt.%: 20% slag, 60% zeolite, 20% bentonite, and composition No. 6 with a content of wt.%: 20% slag, 50% zeolite, 30% bentonite.

Composition No. 5 is shown in Figure 6. It can be observed that with the addition of moisture from 15 to 25 wt.%, the strength changed in the range of 7.65-24.32 MPa at 500 °C. With an increase in the calcination temperature, the strength value also increased. Hence, for a given ratio of components at 1000 °C, the strength value range is 24.27-47.35 MPa.

TABLE I. STRENGTH OF SAMPLES (MPa) AT DIFFERENT TEMPERATURES

N_2	Slag	Zeolite	Bentonite	T(°C)	25%	20%	17.5%	15%
1	-	50	50	500	5.45 <	8.020 >	7.480 <	8.530
				750	7.560 <	13.07 >	12.78 <	13.57
				1000	12.34 <	18.76 >	18.56 <	23.45
2	-	60	40	500	5.450 <	6.210 <	6.65 <	7.43
				750	7.32 0<	8.98 <	10.12 <	13.26
				1000	11.32 <	13.45 <	18.72 <	22.82
3	10	60	30	500	13.68 <	23.06 <	23.21 <	23.62
				750	25.49 <	34.56 >	25.60 <	39.79
				1000	32.06 <	40.60 <	40.66 <	44.31
4	20	40	40	500	5.490 <	6.120 <	7.040 <	15.15
				750	12.76 <	24.72 <	25.01 <	25.93
				1000	20.79 <	26.87 <	28.29 <	29.13
5	20	60	20	500	7.650 <	8.560 <	23.34 <	24.32
				750	9.670 <	14.30 <	36.12 <	41.67
				1000	24.27 <	45.57 >	44.23 <	47.35
6	20	50	30	500	10.06 <	18.03 >	11.47 <	20.70
				750	25.69 <	32.47 >	20.76 <	38.47
				1000	27.09 <	40.70 <	41.73 <	50.53
7	30	40	30	500	14.50 <	14.96 <	18.86 <	19.48
				750	19.54 <	34.01 >	26.98 <	38.62
				1000	37.54 <	39.13 <	39.45 <	41.89

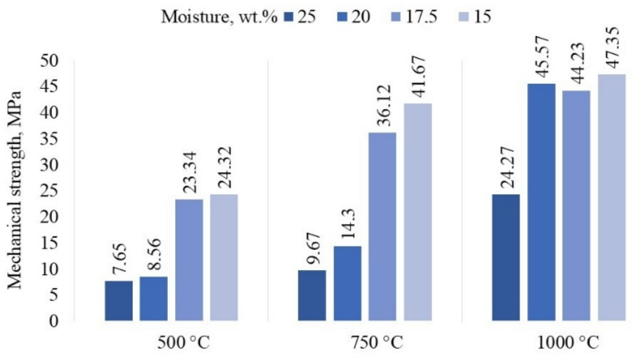


Fig. 6. Mechanical strength of composition 5.

Composition No. 6 is displayed in Figure 7. This composition is generally identical to composition No. 3 regarding the strength characteristics. However, with this ratio, the maximum value of mechanical strength (50.53 MPa) is observed at a humidity of 15% and a calcination temperature of 1000 °C. It was found that heat treatment at 1000 °C is necessary to achieve high mechanical strength. The high strength of the samples is due to the sintering processes, which result in the formation of a crystalline structure that provides the necessary mechanical characteristics of composite materials based on a slag-zeolite-bentonite mixture. It was also found that an increase in humidity from 15 to 25 wt.% leads to a decrease in the mechanical strength, with the optimal humidity level being 15 wt.%.

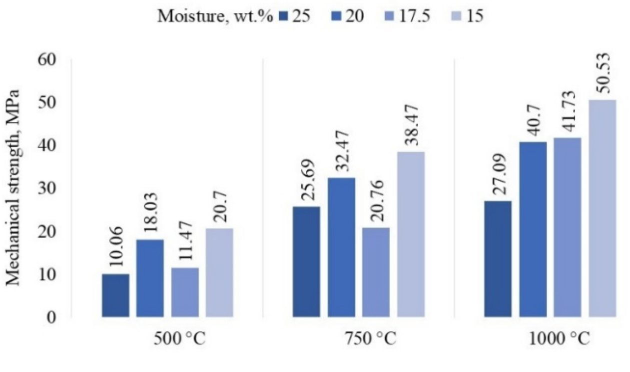


Fig. 7. Mechanical strength of composition 6.

The combination of powder metallurgy methods allows obtaining high-strength composite materials based on aluminosilicates with the addition of lead slag in an amount of 10-30%. The conducted studies to determine the optimal concentration of slag additives in the composition of composite materials showed that the introduction of slag in the range of 10-30% of the total mass of the charge contributes to an increase in the mechanical strength. The highest strength value is observed with the addition of 20 wt.% slag to the charge.

IV. CONCLUSIONS

The use of metallurgical slags in combination with natural aluminosilicates enables creating effective composite materials prototypes with improved performance characteristics. Metallurgical slags formed during metal processing can serve not only as secondary raw materials, but also as active components that help improve the mechanical properties of the final products. To obtain a composite materials consisting of metallurgical slag from lead smelting mixed with natural aluminosilicates with a mechanical strength of 50.53 MPa, the proposed component ratio is 20:50:30 - slag: zeolite: bentonite, with a moisture content of 15 wt.%, and a calcination temperature of 1000 °C.

It was demonstrated that the key factors affecting the compressive strength of the resulting samples are the moisture content of the molding mixture, the ratio of the components of the initial mixture, and the sintering temperature. For example, an increase in the moisture content can lead to an improvement in the binding properties of the mixture, but excessive moisture can negatively affect the strength due to the formation of cracks during drying. Similarly, a change in the ratio of the components can significantly affect the structure and properties of the final material. The sintering temperature also plays a critical role: if the temperature is not high enough, the components do not sinter completely, which can lead to a decrease in strength. At the same time, a too high temperature can cause the destruction of the structure of the material or its overheating. The obtained experimental results indicate that the potential of using powder metallurgy methods to create prototypes of composite materials containing metallurgical slags for the production of non-trivial materials and products has not been fully realized, especially in terms of creating materials with high mechanical strength.

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