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Ecologically Friendly Building Materials: A Case Study of Clay–Ash Composites for the Efficient Management of Fly Ash from the Thermal Conversion of Sewage Sludge

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Abstract: The European Union's initiative to reduce carbon dioxide emissions has paved the way for the exploration of innovative building materials that are environmentally friendly and meet all requirements of durability and strength. These criteria can be met by combining natural resources used in the production of building materials with waste materials that would otherwise be landfilled, having a negative impact on the environment. This study focuses on such materials and presents the results of recent research conducted at the Warsaw University of Life Sciences. The aim was to develop a new generation of materials fully compliant with the principles of the circular economy and sustainable development. Simultaneously, these materials should have no adverse effects on human health and be strong enough to carry the required loads. This study proposes the combination of a natural raw material—in the form of clay—with fly ash from the incineration of sewage sludge to produce a new generation of materials. Several samples were prepared using fly ash from two sources and then were fired at 950 °C. The resulting composites underwent physico-chemical and strength tests. These tests not only confirmed the high strength and durability of the obtained product but also the neutralization of the heavy metals originally present in the fly ash.

Keywords: fly ash; clay–ash composite; compression strength; ecological building material

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1. Introduction

One of the most important challenges facing society today is the negative effect of human activity on the environment. A measure of this impact is the so-called carbon trace, which can be defined as the sum of the greenhouse gases directly or indirectly produced by a product, event or subject [1]. The carbon trace is used in order to describe and understand the impacts of the individual actions of a human being and then compare them with each other in order to be able to choose those that least affect the environment.

Building construction is a branch of industry that affects the natural environment in a significant way. It is estimated that this branch is responsible for the production of as much as 40% of the global production of greenhouse gases [2]. Therefore, the European Union has undertaken a number of activities aimed at limiting the greenhouse gases produced during building construction. The main documents intended to help in achieving this goal are the European Green Deal, the Fit for 55 package and the European Performance of Buildings Directive [3].

Greenhouse gas emissions occur throughout the whole life cycle of a building, starting from the acquisition of raw materials needed for the manufacturing of building materials, through the erection and exploitation of the building, to its demolition [4]. Hence, it is crucial to identify the stages of investment that generate the most carbon dioxide. According

to the investigations of Li et al. [5], ca. 90% of the CO_2 emissions occur at the stage of the manufacturing of building materials and during the exploitation of a building. With reference to these investigations, one can state that our main goal should be the development of building materials and technologies whose impact on the environment is as limited as possible, at both the manufacturing and the exploitation stage. The pursuit of this goal should encompass at least two aspects: the development of new, ecological materials and technologies and the application of recycled building materials.

The building material industry also has another important field for exploration—the application of waste originating from other branches of industry for its 'incorporation' into building objects. The application of this technique adheres to the principles of the so-called circular economy (CE), which is a response to the problems of the changing world, including the limited natural resources and the huge quantities of waste generated in the traditional economic model. The assumptions and application of the circular economy in building construction was broadly discussed by Oluleye et al. [6]. One of the waste materials that has been tested for use in the building construction industry for several years is fly ash, generated during the thermal conversion of sewage sludge.

Due to the increasing global population and improved quality of life, both the sewage network and wastewater treatment plants are expanding rapidly. This growth has led to an increase in the dry mass of sewage sludge, which already amounted to approximately 45 million tons by the early 21st century and continues to rise. This presents a significant challenge and is discussed in numerous studies [7–10].

In previous years, sewage sludge has mainly been disposed of in agriculture as an organic fertilizer. This method, however, generates many problems and limitations resulting from flaws in the rational application of such waste and side effects in the form of heavy metal residues in agricultural raw materials and products. It should also be noted that, currently, the quantity of waste being generated exceeds the capacity for its agricultural application. In this context, other methods of waste disposal have been developed—these include the thermal method, consisting of incineration in fluidal, stoker-fired and rotational boilers [11–13]. An important advantage of the thermal method is that it provides a decrease in the waste volume and a reduction in some chemical compounds (e.g., sulfur and nitrogen). A residue from these processes is fly ash, which is still a waste product and is the subject of the rules presented in Directive 2010/75/EU of the European Parliament and of the Council of 24 November 2010 on industry emissions. The basic objective of this document is to determine possible means of limiting the harmful effects on the natural environment resulting from the pollution of the soil and air, as well as surface and underground water.

Based on the information presented above, it can be seen that the two main directions for the possible use of sludge are agriculture (through its appropriate introduction into the soil as an organic fertilizer) and building construction (through the rational use of fly ash as a raw material in the production of basic building materials). Work in this respect is underway, as has been confirmed in numerous publications [14–23].

The use of fly ash in building construction has a relatively long history, but this mainly concerns ash acquired from other sources. In Poland, several decades ago, a technology for the manufacturing of micro-dimensional wall elements (solid and chequer brick) with a very high share of fly ash, reaching 60% [24], was successfully developed and implemented. Such a large proportion was possible only with the rigorous supervision of the whole process, as well as the fly ash itself. Moreover, it must be noted that this case concerns a ceramic fired at appropriately high temperatures. Regarding non-fired materials, the literature also notes attempts to use ash [24]. In most cases, the amount of ash added is significantly lower, reaching only 10%, and an increase in its share to 20–30% negatively affects the strength parameters of the obtained composite.

The ash produced by the incineration of sewage sludge is usually a fine dust; it generates the same possibilities as for ash obtained from the combustion of mineral fuels. However, significant differences may arise concerning the qualitative composition, which

result from the different characteristics of the obtained sediment. There are various methods of water treatment; therefore, the raw material produced in this process is not uniform in terms of composition and physical form (different dry matter content). Of particular note is the assessment of the amounts of iron oxides and other metals that may be significant in the ash. The literature data show that a large share of such oxides adversely affects the overall properties of the ceramic material.

Summarizing the knowledge regarding the use of fly ash, with a particular emphasis on ash resulting from the combustion of sewage sludge, several facts can be presented regarding the method and scope of their use. They mainly involve determining the optimal share of ash in the mixture and selecting the appropriate firing temperature, which largely determines the parameters of the material [25,26]. Of course, the basic assumption should be to obtain materials with characteristics not inferior to those of ceramics produced using traditional methods, taking into account technical and environmental standards [27]. Another problem is to ensure the safety of the users of such materials and the structures based on them, as well as the safety of the environment [28]. Due to the above, in the research discussed below, the authors' team also addressed this issue, conducting an investigation on the leaching of trace elements from a clay–ash composite obtained by firing.

The research presented below is a continuation of research conducted at the Institute of Civil Engineering of the Warsaw University of Life Sciences on the influence of the addition of fly ash from sewage sludge combustion on the strength parameters of a clay–ash composite. The above goal is analyzed mainly in the context of selecting the appropriate amount of ash in relation to the amount of clay and selecting the optimal firing temperature. In the first stage of the investigation, described in the publication [29], the proportions of clay and fly ash used were 60/40 and the firing temperatures were 300 and 700 °C. The results obtained at the preliminary stage suggested that a product modified with fly ash can be produced; however, the processing (firing) temperature was found to be a significant parameter and was appropriately modified in the series of tests presented in this paper. The paper discusses effects of increasing the temperature to 950 °C, with unchanged ingredient proportions (60% clay, 40% ash).

The presented research could play a key role in promoting sustainable development by addressing key challenges related to the utilization of fly ash, especially that resulting from the thermal conversion of sewage sludge. It focuses on determining the optimal proportion of ash in composite materials and selecting firing temperatures that ensure the desired material properties while meeting technical and environmental standards. Furthermore, the study highlights the importance of ensuring user and environmental safety, particularly by examining the leaching of trace elements from the clay–ash composite. By refining the ash-to-clay ratio and optimizing the firing temperature, the research aimed to develop high-quality materials that not only meet traditional ceramic standards but also contribute to sustainable construction practices. By exploiting the potential of these ecological building materials, we can not only minimize the impact of waste disposal on the environment but also significantly contribute to the implementation of the overarching EU goal of achieving a circular economy. With innovative approaches like the one presented in this study, one can pave the way for a more sustainable future where resource efficiency and environmental protection are at the forefront of construction practices.

2. Materials and Methods

2.1. Characteristics of Fly Ash Used in Research

Within 48 h of delivery to the laboratory, each batch of fly ash from sewage sludge incineration was assessed for its water content in accordance with the standard EN 1097-5:2008 [30]. The densities of the fly ash were identified using the pycnometer method according to EN 1936:2010 [31]. Using the known value of the water density, the mass of the pycnometer filled with water at a constant temperature was measured in an analytical balance. Then, a weighed portion of the material was placed in the pycnometer and it was

filled with water. The prepared sample was placed in a bath for 24 h. After this time, the measurement was performed on an analytical balance. The purpose of the above steps was to determine the "absolute" volume. The bulk density was determined in accordance with the standard EN 1097-3:2000 [32]. This density includes the pores and voids inside the entire material and the spaces between the grains. Knowing the values of the volumetric and bulk densities, the pore coefficient B was determined.

The chemical composition of the ash was determined using an Epsilon-3 spectrometer from Panalytical (Panalytical, Eindhoven, The Netherlands). The tests were carried out in the measurement range for the elements Na–Am using a device equipped with a Rh X-ray tube (9 W, 50 kV, 1 mA), a 4096-channel spectrum analyzer, 6 measurement filters (Cu-500, Cu-300, Ti, Al-50, Al-200, Ag) and a high-resolution semiconductor SDD detector from Panalytical (50-µm-thick beryllium window), cooled with a Peltier cell. The analysis of the chemical composition was based on the energy dispersive X-ray spectroscopy (EDS) method. The morphology was identified using a Quanta 250 FEG scanning microscope from FEI, also equipped with an EDS system [30].

The mineral composition of the ash was determined using X-ray phase analysis (XRD). Measurements were made using the powder diffraction method, using an X'pertPRO MPD X-ray diffractometer from Panalytical, with a PW 3020 goniometer.

The grain size distribution was determined using the Mastersizer 3000 analyzer (Malvern Instruments, Malvern, UK), using laser diffraction. The measurement environment comprised deionized water as a dispersing liquid, exposed to an ultrasonic probe.

The activity index of the used fly ash was determined in accordance with the standard EN 450-1:2012 [33] and the chemical method in accordance with the USA standard ASTM C379-65T [34]. According to the EN 450-1:2012 standard [33], this index is expressed as a percentage of the compressive strength of beams composed of a standard mortar containing 25% fly ash and 75% Portland cement (CEM I 42,5R) in relation to the compressive strength of standard beams composed only of Portland cement. The activity index after 28 days of maturation should be $\geq$75% and that after 90 days should be $\geq$85%. The index was determined after 28, 90 and 180 days.

The determination of the pozzolanic activity of fly ash by chemical methods is based on the total content of Al_2O_3 and SiO_2 (according to ASTM C379-65T [34]). After heating at a temperature of 80 °C in 0.5-mole NaOH solution for 1.5 h, these components are leached from the tested sample. Those parts of Al_2O_3 and SiO_2 that have been dissolved in NaOH are considered reactive for $Ca(OH)_2$. The total content of Al_2O_3 and SiO_2 exceeding 20% suggests the pozzolanic nature of the sample.

2.2. Characteristics of Clay Used in Research

The chemical and mineral characteristics of the clay used for the investigations have been presented in [27].

2.3. Preparation of Samples and Research Methods

Clay and an additive—fly ash from the incineration of sewage sludge—were used to prepare the samples. Fly ash from the incineration of sewage sludge was obtained from two sources: sewage treatment plants in Warsaw and in Łódź. Batches of fly ash were collected in 2019 in Warsaw and in 2020 in Łódź and then stored in closed containers.

When preparing the mixed samples (clay–ash), a proportion of 60% clay and 40% ash was added to all samples. These proportions were identical to those for the first stage of the research [29]. For comparison, a batch of samples without the addition of ash was also prepared. The samples were marked as follows:

- 0–i—without addition;
- 1–i—with the addition of fly ash from Łódź;
- 2–i—with the addition of fly ash from Warsaw.

Samples were prepared in two sizes: in cubic molds with dimensions of 4.0 × 4.0 × 4.0 cm^3 and in cuboidal molds with dimensions of 4.0 × 4.0 × 16.0 cm^3. A total of 23 pieces were

prepared: 11 square samples, with 5 samples containing each type of ash and one sample without ash, and 12 rectangular samples, with 4 samples containing each type of ash and 4 samples without ash.

Preparing the clay for sample production involved several steps. First, the clay was dried, ground and sifted through a 2 mm sieve to remove any impurities. The clay was then divided into three parts, forming the basis for the three types of samples. Two parts of clay were then mixed with fly ash in the specified proportions and mixed thoroughly. Water was added to the samples to obtain the desired consistency. The consistency was checked by forming a 200 g ball from the mixture and dropping it on a flat surface from a height of 2 m, measuring the diameter of the resulting deformation. Samples prepared with various ash amounts, as well as samples without any ash, showed a comparable level of plasticity after this procedure. The samples were then molded, ensuring that the molds were thoroughly cleaned and lubricated before use.

In both versions, the molds were prepared and filled in the same way. The samples were marked according to Table 1. Before drying, each sample was weighed on a certified laboratory scale. The masses of the samples (before drying) are shown in Table 1 and the marked cubic samples are shown in Figure 1.

Table 1. Markings and masses of samples before drying.

Clay (100% Clay)		Composite—Łódź (60% Clay + 40% Fly Ash)		Composite—Warsaw (60% Clay + 40% Fly Ash)		
No.	**Mass [g]**	**No.**	**Mass [g]**	**No.**	**Mass [g]**	
0-5	122.1	1-1	111.2	2-1	113.2	Cubic samples
		1-2	119.2	2-2	112.6	
		1-3	119.0	2-3	117.4	
		1-4	116.0	2-4	113.8	
		1-5	119.8	2-5	112.3	
0-1	465.8	1-6	471.2	2-6	460.4	
0-2	509.7	1-7	476.0	2-7	460.3	Cuboid samples
0-3	482.5	1-8	479.3	2-8	450.7	
0-4	479.1	1-9	457.7	2-9	458.3	

Figure 1. Mixed cubic samples.

The drying of the prepared specimens took a total of 5 days. Initially, the specimens were left to dry for three days at ambient temperature. Then, they were transferred to a dryer for another 2 days, where they were exposed to a temperature of about 40 °C. This gradual drying process was implemented to prevent shrinkage cracks from occurring. After completing the drying process, the samples were left at ambient temperature for approximately 7 h before further processing. Then, all samples were dried to a constant mass at a temperature of 105 °C ± 5 °C. In order to determine the firing time at particular temperatures, a preliminary test was carried out on specially prepared samples with

holes for thermocouples. The temperature distribution was monitored using four NiCr-Ni thermocouples that met the requirements of the standard [35]. Then, the samples were heated at a temperature of 950 °C in the PK 1100/5 furnace from Termolab S.C. (Warsaw, Poland), with electric heating sections. The station was equipped with a dedicated ThermoPro program, enabling the programming of the heating process. The firing profile of the samples (standard "temperature vs. time" curve) is presented in Figure 2. Samples during the firing process are shown in Figure 3.

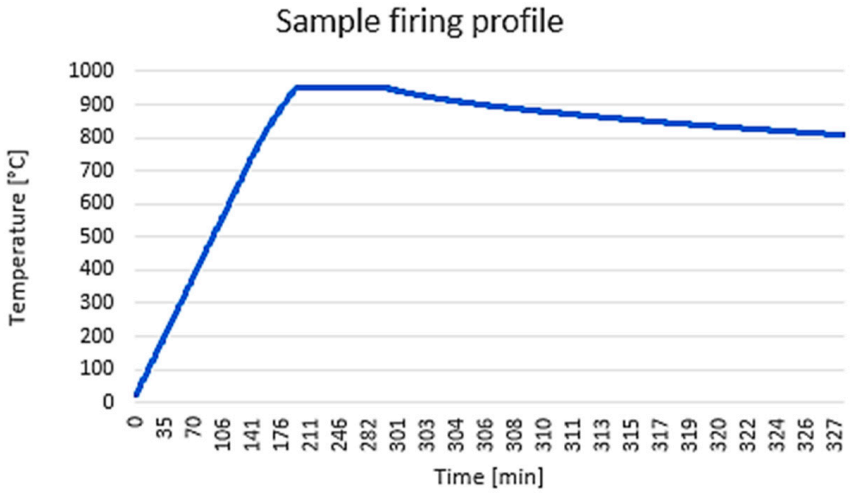


Figure 2. Sample firing profile: temperature vs. time relationship.

Figure 3. Samples during the firing process, with thermocouples visible.

A series of tests were carried out on the prepared samples. In the case of square specimens, water absorption was first tested, and then the compressive strength of the specimens was tested. In the case of rectangular specimens, the shrinkage, water absorption and flexural strength were first tested, and then compressive strength tests were performed on specimens that had cracked in the previous tests. The results of all tests conducted, their summaries and their comparisons with the results of previous tests are presented in the next subsection.

Compressive strength tests were carried out on the Advantest 9 mortar testing machine from Controls, equipped with an arm with a pressure force of up to 250 kN.

3. Results and Discussion

3.1. Physical and Chemical Properties of Fly Ash from Incineration of Sewage Sludge

Before the investigation, the basic characteristics of the fly ash from the thermal conversion of sewage sludge were identified. First, the color of the investigated waste was determined through visual identification. Code 19 01 14 ash is dark red/pink in color. Despite the different shades, the material differed significantly from the ash generated in the energy industry, the color of which oscillates within a wide gray scale [36,37]. It was a dry material, which can be expected from a material produced at the temperature of approximately 900 °C, obtained in the dry state. The moisture content of the ash obtained from the sewage treatment plants in Warsaw and Łódź was 0.1% (EN 1097-5:2008 [30]). The values of the volumetric and bulk density as well as the pore ratio are presented in Table 2.

Table 2. Density and pore ratio of fly ash from incineration of sewage sludge.

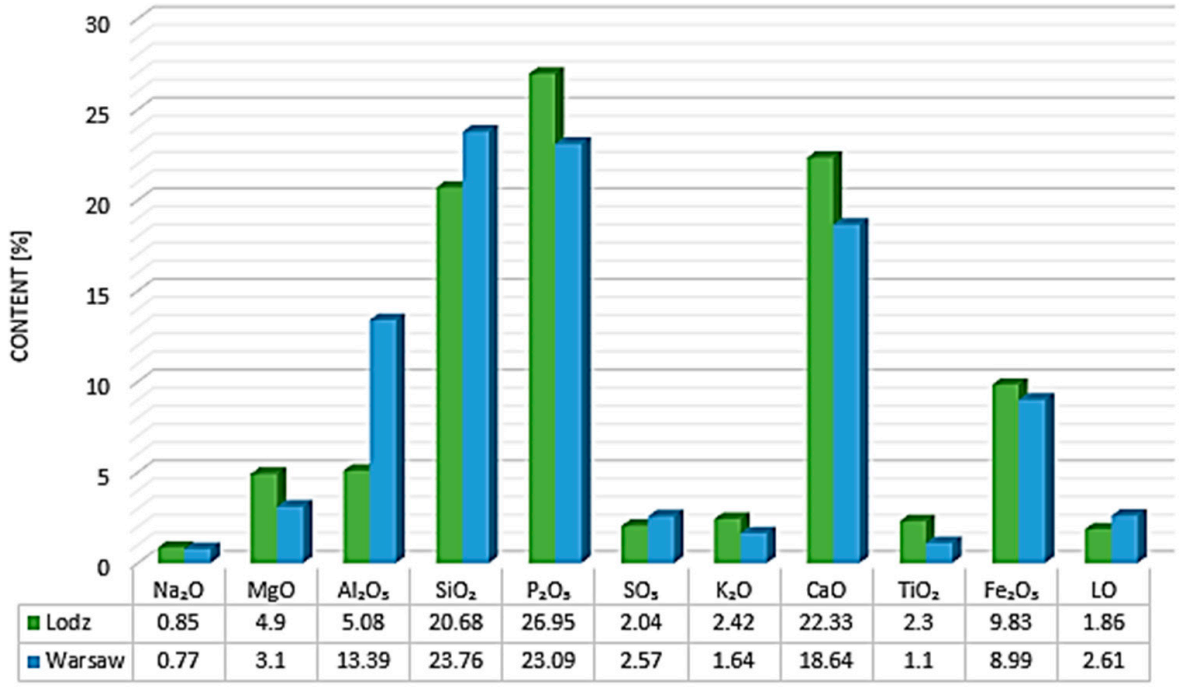
Ash Type	Volumetric Density [kg/m^3]	Bulk Density [kg/m^3]	Pore Ratio B [%]
Lódź—LO	2620	820	68.70
Warsaw—WA	2530	780	66.79

The bulk density of the investigated ash ranged from 780 kg/m^3 for the WA ash to 820 kg/m^3 for the LO ash. For comparison, the bulk density of ash from coal combustion is generally within the range of 400–1200 kg/m^3 [38,39].

The pore ratio, calculated according the density, ranged between approximately 67 and 69% for the 19 01 14 ash. It was observed that the share of pores in the ash from the combustion of sewage sludge was lower than the lowest value for the ash from the combustion of brown coal (74.37–76.39%) and hard coal (73.42–81.34%) [20,40–43] and similar to the values reported by other researchers for the fly ash from sludge combustion [26].

According to the results obtained, it can be concluded that the variability in the free CaO content in the fly ash was small (LO—0.12% by mass, WA—0.09% by mass). All samples met the requirements of the EN 450-1:2012 standard [33]—the limit value for the individual content is 1.6%. The variability in the total CaO content can be considered high (LO—22.02% by mass, WA—18.64% by mass). The requirements for reactive CaO (limit value for an individual result lower than 11% of mass) influencing the formation of the C-S-H phase were met for the Łódź ash (10.92% of mass). The Warsaw ash exceeded the permissible value by 3.74% by mass. Higher content of reactive CaO affects the hydraulic properties of the ash. None of the batches of ash from sewage sludge (LO—16.14% by mass, WA—13.32% by mass) fulfilled the standard requirements regarding the amount of reactive SiO_2 (the lower value is 25% by mass). Considering that the basic oxides forming the cement clinker were CaO, SiO_2, Al_2O_3 and Fe_2O_3, the requirements for the sum of the content of three of them (SiO_2, Al_2O_3, Fe_2O_3) were not fulfilled for any of the batches (Figure 4). The investigated samples of fly ash from sewage sludge were characterized in terms of the dispersion of the results, with 36.37% for LO ash and 46.14% for WA ash. According to the ASTM-C618-03 standard [34], the sewage sludge ash satisfied the requirements for total oxides (SiO_2, $Al_2O_3 \geq 20\%$).

The content of Cl^- chlorides and sulfuric anhydride SO_3 was also determined in each sample. In each batch of sewage sludge ash, the chloride content was similar—0.03 and 0.04% by mass—and was lower than the limit values for individual results. The SO_3 content was 1.98% by mass for the LO ash and 2.57% by mass for the WA ash. According to the literature, the presence of chlorides and sulfates negatively affects the properties of the produced composites. Chloride aggression leads to a decrease in the pH of the material and the formation of expansive compounds.



	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Fe_2O_3	LO
Lodz	0.85	4.9	5.08	20.68	26.95	2.04	2.42	22.33	2.3	9.83	1.86
Warsaw	0.77	3.1	13.39	23.76	23.09	2.57	1.64	18.64	1.1	8.99	2.61

Figure 4. Chemical composition of fly ash from sewage sludge.

The particle size distribution of the investigated fly ash was monomodal, with a maximum value of 125 µm (Łódź) and 85 µm (Warsaw). Grains with a diameter of 2–250 µm occupied over 91% of the volume in all batches of the investigated ash. It can be stated that the ash should be classified as a coarse-grained material. The dominant grain fractions in this range were 20–50 µm (20.18%—LO, 25.01%—WA), 50–100 µm (25.88%—LO, 28.12%—WA) and 100–250 µm (34.79%—LO, 27.75%—WA)—see Figure 5. The presented results were in line with those of other authors dealing with sewage sludge ash [44,45]. Additionally, Zabielska-Adamska pointed out significant differences in the grain size distribution of 19 01 14 ash, related to the thermal conversion technology. For comparison, finer particles (0.25 µm) in the sewage sludge fly ash investigated by Kosior-Kazberuk [26] constituted approximately 7%.

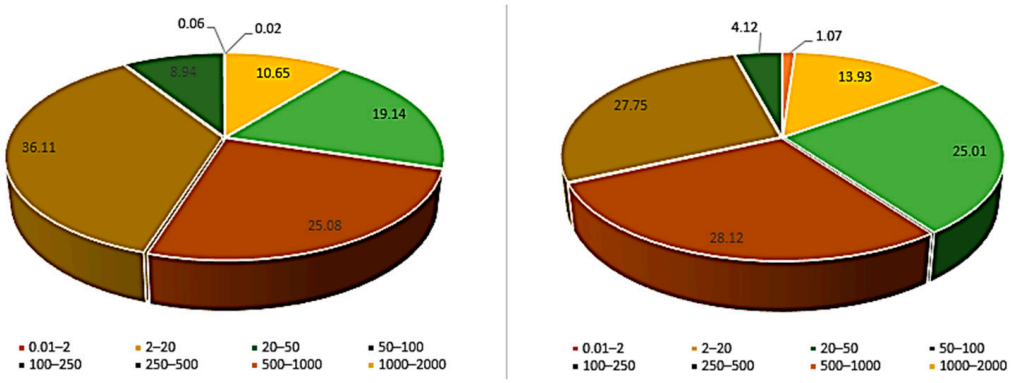


Figure 5. Grain size distribution for fly ash from sewage sludge from Łódź and Warsaw.

Figures 6 and 7 present images of the fly ash from the combustion of sewage sludge, generated with the scanning electron microscope. The images clearly show that irregular grains dominate, with variable sizes and highly developed surfaces. There is no glass phase or spherical grains. The fly ash from the combustion of sewage sludge does not comply with the definition of fly ash given in the EN 450-1:2012 standard [33]. By analogy to the fluidized bed combustion of pulverized coal, it can be noted that the grains of such

fly ash are compact (create conglomerates) and have many open pores, which causes a high demand for water. This may lead to higher water absorption due to the higher water demand of concrete with a share of fly ash from sewage sludge [46]. There were very few spherical and cuboidal forms.

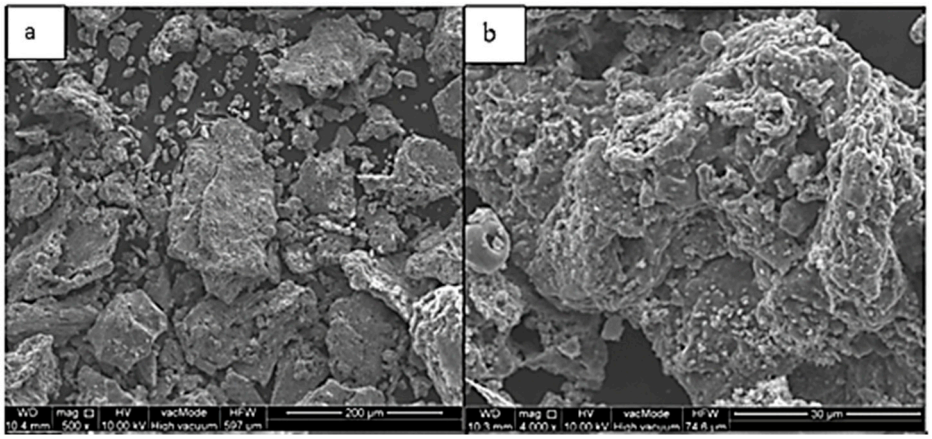


Figure 6. SEM images of Łódź fly ash from sewage sludge: (**a**) magnified 500 times, (**b**) magnified 4000 times.

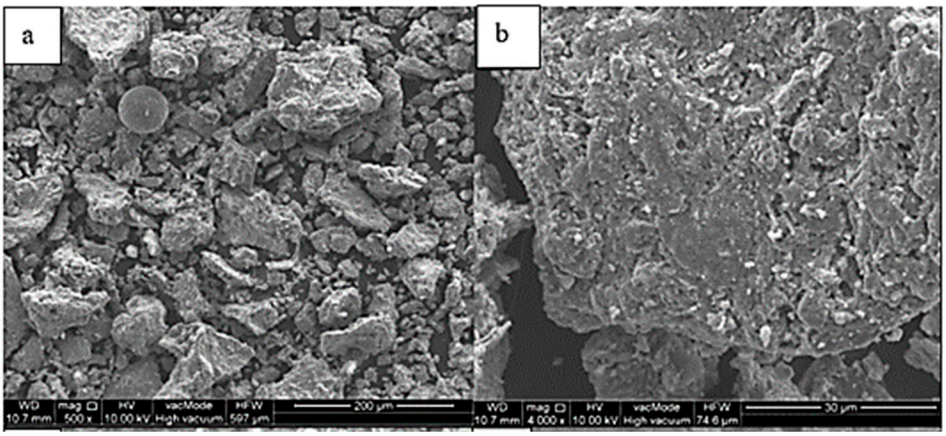


Figure 7. SEM images of Warsaw fly ash from sewage sludge: (**a**) magnified 500 times, (**b**) magnified 4000 times.

The chemical analysis in the micro-range (SEM-EDS) presented a diverse element composition. Grains containing silicon, aluminum, phosphorus and iron predominated, and grains containing calcium were also observed. However, the typical components of the fly ash differed significantly in morphology. The remaining components, natrium, magnesium and potassium, were present in negligible amounts. The fly ash samples were dominated by irregular grains of various sizes, with a strongly developed surface, illustrating the high porosity of the material, with a loose and rough structure. This could lead to higher water absorption related to the increased water demand of a clay–ash composite containing fly ash from the thermal conversion of sewage sludge. Spherical and cuboidal forms were very rare.

Figures 8 and 9 present the XRD diffraction spectra of the fly ash from sewage sludge combustion. The mineral composition of the ash is dominated by quartz and anhydrite, which were identified on the basis of the characteristic interphase distances. The mineral composition of the fly ash from sewage sludge combustion is supplemented by phosphates in the form of apatite and fluorapatite. These mineral phases are primarily carriers of P_2O_5, existing here in higher quantities if compared to the content of conventional ash. The phase composition of this group of ash can be considered homogeneous, which is confirmed in

the studies of other scholars [47–50]. Moreover, the content of mullite ($Al_6Si_2O_{13}$) in the investigated ash was marginal. For comparison, fly ash from the combustion of hard and brown coal, both in fluidal and pulverized-fuel boilers, are characterized primarily by high content of SiO_2 quartz [51]. In the ash from coal combusted in pulverized-fuel furnaces, mullite occurs in an amount of approximately 20%, while, in fluidal boilers, it does not occur at all [51]. Other scholars [47–50] do not find phases other than those identified in the fly ash from Polish water treatment plants.

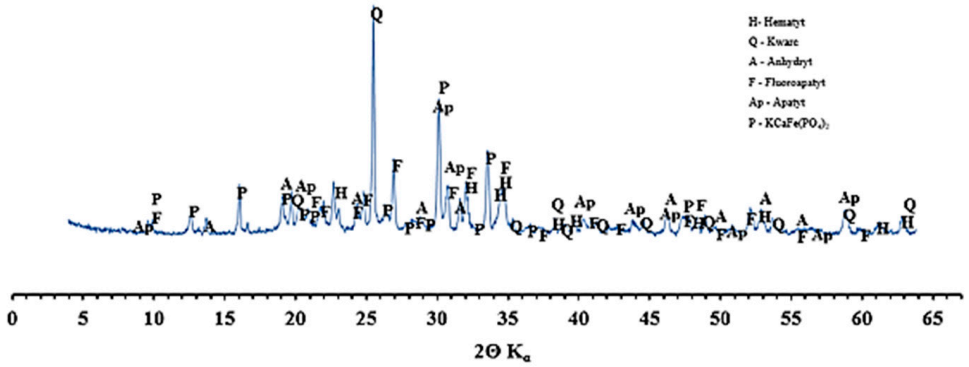


Figure 8. XRD diffraction patterns for Łódź fly ash from sewage sludge combustion.

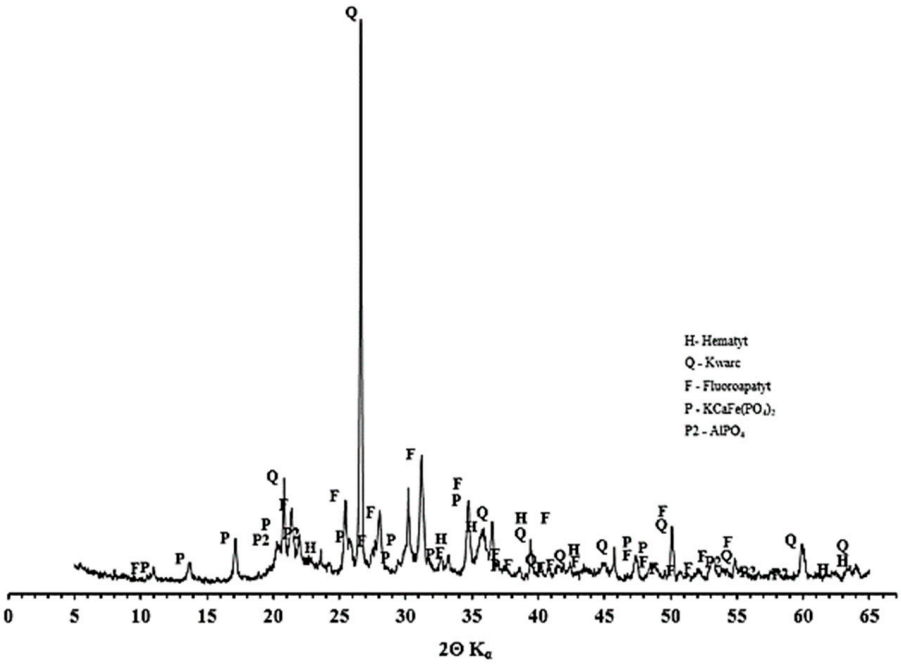


Figure 9. XRD diffraction patterns for fly ash from Warsaw from combustion of sewage sludge.

3.2. Properties of Clay Samples

The composite samples were dried and heated and then weighed and visually inspected to assess their quality.

For the cubic samples, the parameters evaluated were the dimensions of the samples (length and width) and their mass after firing. Based on the collected values, the percentage of water absorbed in the samples was also determined. The obtained results are presented in Tables 3 and 4.

Table 3. Dimensions of samples after firing process.

Type of Sample	Marking of Sample	Dimensions of Sample after Firing [mm]	
		a (Width)	b (Length)
Clay	0-5	37.69	37.68
Composite—Łódź	1-1	38.28	38.02
	1-2	38.33	38.13
	1-3	38.42	37.63
	1-4	37.71	37.9
	1-5	37.97	37.92
Composite—Warsaw	2-1	37.51	37.54
	2-2	37.37	37.45
	2-3	37.37	37.26
	2-4	37.39	37.21
	2-5	37.77	37.62

Table 4. Mass of cubic samples before and after firing and share of absorbed water.

Type of Sample	Marking of Sample	Mass of Sample before Firing [g]	Mass of Sample after Firing [g]	Absorption of Water [%]
Clay	0-5	115.3	105.9	15.3
Composite—Łódź	1-1	91.5	85.4	30.2
	1-2	96.5	90.6	31.6
	1-3	93.5	90.5	31.5
	1-4	95.6	89.7	29.3
	1-5	95.7	90	33.1
Composite—Warsaw	2-1	93.1	87.1	30.0
	2-2	92.2	86.1	30.8
	2-3	95.1	88.9	32.1
	2-4	92.2	86.3	31.9
	2-5	92.2	86.4	30.0

After firing, the samples decreased in size by approximately 2 mm, regardless of the addition of fly ash. Water absorption was found to be approximately 50% higher for composite samples compared to pure clay samples. The difference in water absorption between samples with Łódź fly ash and Warsaw ash was negligible.

The next step was a static compression test, performed on a dedicated testing machine. The results of this test, along with the compressive strength of each sample, are presented in Table 5.

After comparing the average values of the composite samples with those of pure clay, it can be seen that the compressive strength for the samples with Warsaw ash was similar to that of pure clay samples; however, composites with Łódź fly ash were characterized by a slightly lower average strength—it was 4.36 MPa lower than that of pure clay. The maximum difference in compressive strength was 16.46 MPa, which is a significant value, while, within each group, the maximum differences were 11.88 MPa (Łódź) and 14.95 MPa (Warsaw). It is important to note here that the samples were created manually, which may partly explain these significant differences. It can also be assumed that the mechanization of the sample preparation process would improve their homogeneity, which should lead to the lower dispersion of the results obtained in the strength tests.

The compressive strengths for the three types of samples (pure clay, composite with Warsaw ash, composite with Łódź ash) are presented in Figure 10.

Table 5. Strength characteristics of cubic samples.

Type of Sample	Marking of Sample	Compressive Force [kN]	Compressive Strength [MPa]	Compressive Strength—Arithmetic Mean [MPa]
Clay	0-5	41.0	28.87	28.87
Composite—Łódź	1-1	27.0	18.55	24.51
	1-2	27.5	18.82	
	1-3	44.0	30.43	
	1-4	41.5	29.04	
	1-5	37.0	25.70	
Composite—Warsaw	2-1	40.0	28.41	28.19
	2-2	49.0	35.01	
	2-3	33.0	23.70	
	2-4	47.0	33.78	
	2-5	28.5	20.06	

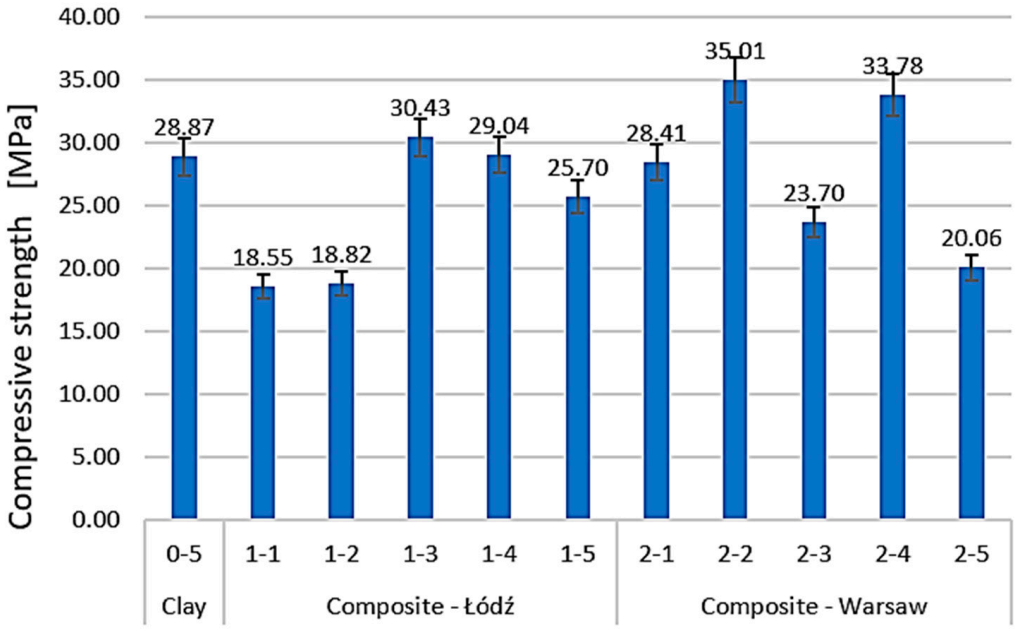


Figure 10. Compressive strength for three types of samples (pure clay, composite with Warsaw ash, composite with Łódź ash)—with 5% error bar.

Based on the results obtained in the first stage of the research and presented in [27], where the firing temperatures were assumed to be 300 and 700 °C, it was possible to prepare a graph of the dependence of the temperature and obtained compressive strength, which was the arithmetic mean of the values obtained for individual samples. Such a graph is presented in Figure 11.

Lower firing temperatures resulted in significantly lower compressive strength values for the fly ash samples. An increase in the temperature to 950 °C reduced the influence of the ash content on the decrease in compressive strength—the differences in the compressive strength values were small.

In the case of cuboidal samples, the mass, shrinkage, mass absorption and strength parameters of the samples were determined after firing. The obtained results are presented in Tables 6 and 7.

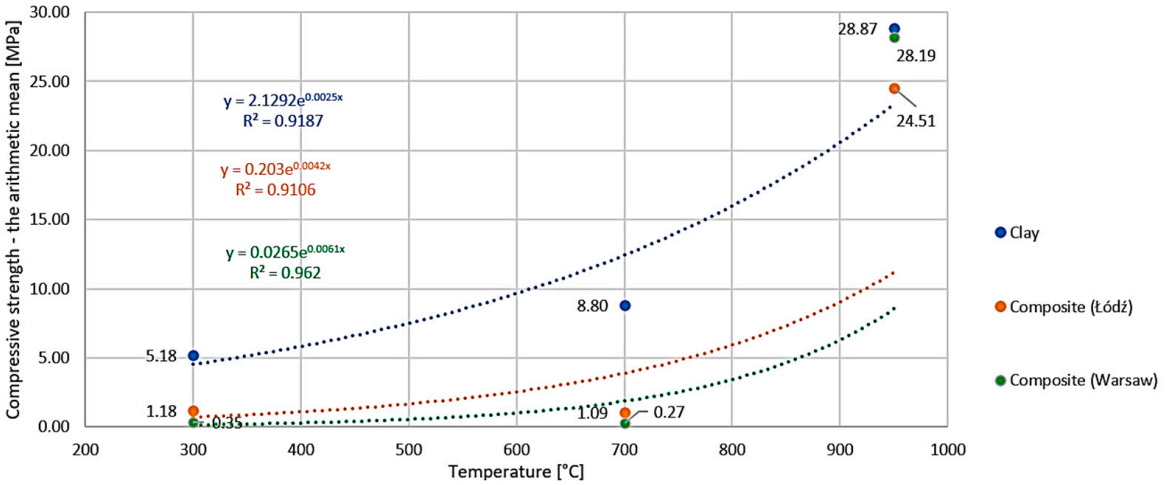


Figure 11. Relation between firing temperature and obtained average compressive strength with consideration of exponential trend line.

Table 6. Mass of cuboid samples before and after firing and absorption of water.

Type of Sample	Marking of Sample	Total Shrinkage [mm]	Mass of Sample before Firing [g]	Mass of Sample after Firing [g]	Absorption of Water [%]	Absorbability [%]
Clay	0-1	2.9	465.8	378.5	23.1	11.2
	0-2	2.9	509.7	411.8	23.8	11.2
	0-3	3.2	482.5	390.1	23.7	11.3
	0-4	3.0	479.1	387.9	23.5	11.4
Composite—Łódź	1-6	3.4	471.2	355.3	32.6	16.9
	1-7	3.6	476	361.2	31.8	16.9
	1-8	3.2	479.3	340.9	40.6	17.5
	1-9	3.7	457.7	349.0	31.1	17.5
Composite—Warsaw	2-6	3.6	460.4	346.0	33.1	19.6
	2-7	3.7	460.3	347.4	32.5	19.5
	2-8	3.5	450.7	341.4	32.0	19.4
	2-9	3.3	458.2	340.1	34.8	20.3

Table 7. Strength characteristics of cuboid samples.

Type of Sample	Marking of Sample	Compressive Strength—Part 1 [MPa]	Compressive Strength—Part 2 [MPa]	Compressive Strength—Arithmetic Mean [MPa]
Clay	0-1	7.03	9.93	10.19
	0-2	13.70	13.11	
	0-3	10.29	9.67	
	0-4	9.71	8.05	
Composite—Łódź	1-6	12.45	10.90	13.32
	1-7	14.18	15.80	
	1-8	14.37	12.56	
	1-9	11.70	14.59	
Composite—Warsaw	2-6	8.77	8.69	10.89
	2-7	13.35	11.34	
	2-8	11.96	11.69	
	2-9	9.56	11.74	

The average shrinkage of pure clay samples was 3.0 mm, which corresponds to approximately 6%. However, the average shrinkage of composite samples (clay–ash) was 3.5 mm, which corresponds to approximately 7%. It can therefore be concluded that the composition and origin of the ash have no influence on the shrinkage.

However, if the composite samples are compared to the pure clay samples, the average difference is approximately 1%. It should therefore be noted that the addition of 40% ash does not significantly affect the final shrinkage, even after firing at a temperature close to 950 °C. As expected, water absorption is higher for the composite samples than for the

pure clay samples. The difference in water absorption between the samples with ash from Warsaw and Łódź is negligible. After assessing the percentage absorbability (in relation to the mass after firing), a similar relationship can be observed as in the case of the water absorption. The average percentage absorbability for clay samples is 11.3%, and that for clay–ash samples is 17.2% for Łódź ash and 19.7% for Warsaw ash. Hence, a slightly greater difference in water absorption in relation to the ash's origin is visible.

In the next stage, the cuboid samples were subjected to strength tests. First, the bending strength was tested by loading the sample with a concentrated force at the center of the length. Then, after breaking the sample, both halves were tested for its compressive strength. The results of the bending strength tests are presented in Figure 12, and the compressive strength test results are presented in Table 7.

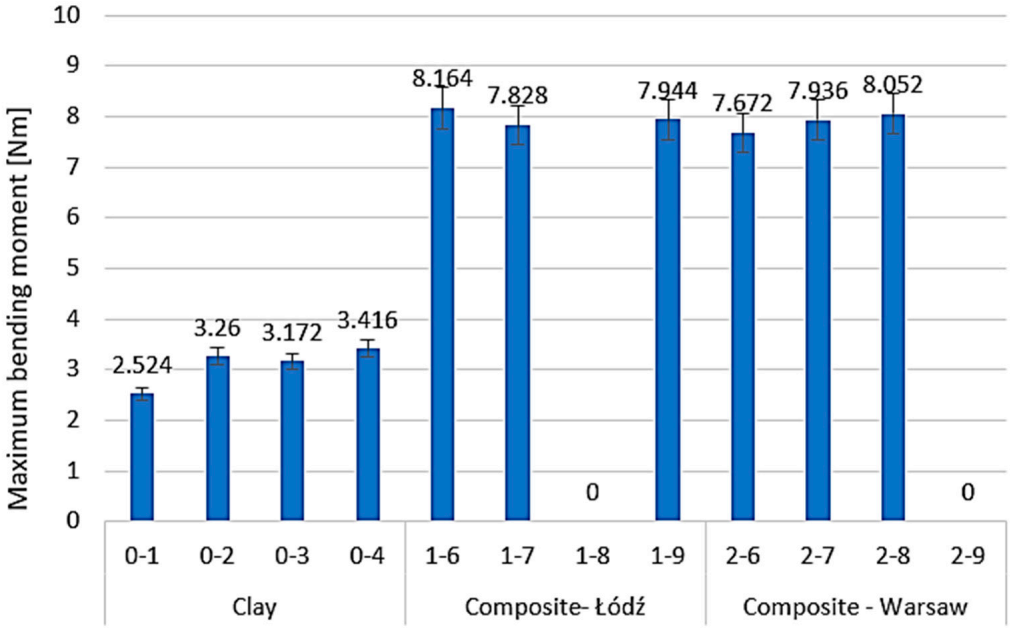


Figure 12. Bending strength for three types of samples: pure clay, clay–ash composite with Łódź ash, clay–ash composite with Warsaw ash—with an error of 5%.

The flexural strength was significantly higher for composite samples than for pure clay samples. This difference reached 50%. The average bending strength of clay samples was 3.09 Nm, while that of composite samples with Łódź ash was 7.97 Nm and for those with Warsaw ash was 7.88 Nm. In each group, there was one sample in which a crack formed during firing, making it impossible to check the bending strength —these were the samples marked 1-8 and 2-9. These samples were not taken into account when calculating the average values.

The last stage of the strength research was the testing of the compressive strength of the samples previously subjected to the bending tests, i.e., the samples with dimensions of $4 \times 4 \times 8$ cm^3. The results of the tests are presented in Table 7. The mean value was calculated from all tested samples within a given type.

3.3. Leachability of Heavy Metals

The samples were tested for the leaching of heavy metals, including Cu, Zn, Cd, Pb and Ni. The research was carried out at the Institute of Civil Engineering of the Warsaw University of Life Sciences, using atomic absorption spectrometry (ASA), on 25 water samples, in which the clay–ash samples were immersed for 14 days. Distilled water at room temperature was used for the test. The heavy metal content was determined according to the standard procedure described in the literature [52]. The permissible limit values of metals depending on the application after processing in Poland are regulated by the

Ministry of Economy and the Ministry of Maritime Economy and Inland Navigation [53,54]. The results of these tests were identical for all samples and were as follows:

- Cu: <0.041 mg/L;
- Zn: <0.013 mg/L;
- Cd: <0.005 mg/L;
- Pb: <0.1 mg/L;
- Ni: <0.063 mg/L.

3.4. Practical Implementation and Future Recommendations

This work focused on the development of innovative building materials using fly ash from the thermal conversion of sewage sludge as an addition to ceramic elements made from clay. Research in this area has been conducted for a long time [55,56], but there are still no clear guidelines regarding the permissible ash content, its safety of use and the mechanical properties of such materials.

In this study, the tests carried out confirmed the possibility of obtaining composites containing a significant amount of fly ash and demonstrated their high strength properties. In addition, the safety of the product was confirmed via leachability testing. Nevertheless, such studies are at a preliminary stage, and further research is necessary to improve the clay–fly ash composite and investigate its durability and potential applications in construction. In the next step, we will focus on fine-tuning the firing temperature and holding time at the highest temperature, which should result in an increase in compressive strength and a decrease in water absorption. Previous studies have shown that increasing the firing temperature increases the amount of the glassy phase, which, in turn, increases the compressive strength and reduces the water absorption. Finding the optimal firing temperature will allow us to obtain a product with the best properties in the final phase of testing.

Further research should also focus on a thorough analysis of the types of construction products that can be produced using aluminous foam composites and on the benefits that can be obtained by the manufacturers and users of such materials. It will also be necessary to identify the possible technical and environmental challenges related to the practical implementation of these materials and to develop appropriate guidelines and standards regulating their production and use.

4. Conclusions

Based on the presented research, the following conclusions and plans for further work can be formulated.

1. The tests confirmed the possibility of producing clay–ash composites with high content of fly ash from the combustion of sewage sludge.
2. Fly ash from two water treatment plants in Warsaw and Łódź was used for the tests. In the case of most of the analyzed parameters, the origin of the ash did not cause significant differences.
3. The obtained results made it possible to compare the characteristics of the samples composed of pure clay and the clay–ash composite.
4. High compressive strength was obtained for the composite samples with high fly ash content. The values of this strength were comparable to the strength of a pure clay sample.
5. Leachability tests showed that the content of heavy metals after firing was negligible. It can be concluded that they bind in the resulting product.
6. With large amounts of fly ash (over 20%), the achievement of adequate compressive strength depends significantly on the firing temperature.

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