Repair and filling mortars based on metallurgical waste

Jana DAŇKOVÁ[a], Jana SEIDLEROVÁ[b, d], Adéla VALENTOVÁ[a], Petr BĚČÁK[b,d], Petr UNUCKA[c]

[a] VSB – Technical University of Ostrava, Faculty of Civil Engineering, Department of Building Materials and Diagnostics of Structures, Ludvíka Podéště 1875/17, 708 00 Ostrava – Poruba, Czech Republic
[b] VSB – Technical University of Ostrava, Nanotechnology Centre CEET, 17. listopadu 2172/15, 708 00 Ostrava – Poruba, Czech Republic
[c] ECOCOAL s. r. o, Mrštíkova 4, 709 00 Ostrava 9, Czech Republic
[d] Department of Chemistry and Physico-Chemical Processes, Faculty of Material Science and Technology, VSB-Technical University of Ostrava, 17. listopadu 15/2172, 708 00, Ostrava, Poruba, Czech Republic
e-mail: jana.dankova@vsb.cz jana.seidlerovava@vsb.cz adela.valentova@vsb.cz: petr.becak@vsb.cz unuckap@plazkat.cz

Abstract

The topic of this paper is an experimental analysis of the possibility of using selected metallurgical wastes as the main raw materials for the production of repair and filling mortars for masonry. Technology for reducing solid particles in industrial emissions leads to the production of fine-grained wastes, which cannot be recycled without treatment. These include metallurgical dust from dry cleaning of waste gases from agglomeration, iron and steel production, or fly ash from combustion processes, cement plants, etc. The main components of metallurgical dust are Fe_2O_3, FeO, Al_2O_3, CaO, MgO, MnO, SiO_2 in various weight ratios. Fly ash from combustion processes contains mainly Al2O3, SiO2, CaO in the form of free CaO and sulphates. On the basis of the chemical composition of these wastes, their use in construction can be considered. In the construction industry, the implementation of remediation measures is a fundamental approach to eliminating waste generation and extending the life of buildings. Remediation filling mortars are, among other things, intended to fill cavities and joints in masonry. Repair plaster mortars for masonry are materials intended for secondary measures to reduce moisture and salinity in masonry structures. The current range of repair and filling mortars is mainly based on cement-based materials, high-quality sorted natural non-renewable raw materials, or mixed polymer-cement materials. This experimental study verified the properties of mortars in which the binder and filler were largely replaced by selected metallurgical wastes. The results of the initial study are promising and will be used for further research and development in this area.

Keywords: *mortars, natural cement, metallurgical waste, building materials, masonry repair mortars*

Introduction

The sustainable development of the construction industry within the European Community is enshrined in the basic requirements for construction works[1]. According to strategic approaches to this basic requirement, the remediation of damaged structures is prioritised over demolition, thus fulfilling the principle of waste prevention[2].

Developments in the field of repair building materials and technologies are an important and relatively complex construction discipline, preceded by a structural and technical survey of the building. The aim of repair work on fired brick masonry is to restore its functions in order to ensure mechanical resistance and stability, reduce moisture in the masonry, and ensure that the indoor environment of buildings meets hygiene requirements. There are many structural and material solutions to this problem. However, each specific remediation measure must take into account all parameters of the existing building structure and must be implemented to achieve meaningful objectives. The most common problem with masonry is increased moisture. Repair projects aimed at removing moisture from masonry are technically and financially demanding. It is usually a long-term process that involves several gradual steps. These steps may be direct or indirect measures. Indirect measures include the application of remediation mortars. These are highly porous mortars applied to accessible surfaces of the masonry being remediated. Gradually, water and water-soluble salts migrate from the masonry into the porous system of the remediation mortars. The masonry materials are gradually dried and desalinated, and the overall condition of the building structure improves[3-7].

Currently, there are many repair mortar products available on the market. Their effectiveness in construction is usually ensured by monitored parameters. Repair mortar mix is defined in the ČSN P 730610 as an "industrially manufactured repair mortar mix." This standard also defines a "repair plaster system," which is classified in this standard as an indirect as an indirect repair method in this standard repair method. The system of requirements for restoration plasters is available, for example, in WTA Guideline No. 2-9-04/D[8]. Further criteria, requirements, and procedures for verifying the properties of mortars are specified in the EN 1015 series of standards and in the EN 998-1, Ed. 3[9]. The key properties of restoration mortars, in accordance with the above standards and regulations, include high porosity, resistance, and durability in relation to the effects of water-soluble salts, frost resistance, and diffusion openness.

The raw material composition of the current range of restoration mortars varies and usually consists of a high proportion of high-quality non-renewable raw materials and materials with high impact category values. In general, the desired goal is to replace these raw materials and materials with suitable secondary raw materials, thereby reducing the impact category values of these widely used building restoration materials.

Iron and steel products remain among the most commonly used materials in a wide range of industries in the 21st century. This is evidenced by steel production in the EU, which amounted to 129.5 million tons in 2024[10]. Approximately 120 – 130 million tons of iron ore were consumed for this production[11]. The volume of production is one of the reasons the metallurgical industry is one of the most significant sources of waste. The following types of wastes are generated: sintering dust and sludge from the sintering process; blast furnace/steelmaking (convertors, electric art furnace) dust and sludge from the blast/steel (convertors, electric arc furnace) process; Ceramic debris; sludges from wet dedusting of burned gases and melting losses[12-14].

The pressure to reduce the amount of solid particles released into the air has led to the introduction of effective gas cleaning technologies. However, dry (dust) and wet (sludge) waste gas cleaning processes generate considerable amounts of waste. For example, a blast furnace with a capacity of approximately 2 million tons of raw iron per year produces up to 20,000–30,000 tons of dust per year[15]. Across the EU, it is estimated that hundreds of thousands of tons of dust are produced annually, but accurate aggregate data are not publicly available.

Although both dust and sludge contain not only iron in oxide and metallic form, but one of the main difficulties in handling such waste is its physical structure. Fine particles have a very small size and low density, which makes them easy to spread by air and difficult to collect. The fine-grained waste can only be recycled after testing, usually in combination with a suitable binder.

This article presents the results of an experimental study in which metallurgical waste and a low-temperature binder, PROMPT natural (Roman) cement (Vicat, France), were used to produce mortar samples. The purpose of this experimental study was to verify the usability of selected metallurgical waste for the development of a new building material, remediation and filling mortar. The values of selected properties of hardened mortars were monitored: bulk density, flexural strength, compressive strength, frost resistance, and porosity.

Experimental part

Equipment and methods

The chemical composition of the samples was determined using X-ray fluorescence spectroscopy (XRFS) with an energy-dispersive X-ray fluorescence spectrometer SPECTRO XEPOS using the pellet method. The pellets were prepared from the samples after grinding them to a grain size of less than 0.06 μm.

The contents of selected elements were then confirmed by atomic emission spectroscopy with inductively coupled plasma (AES-ICP) after decomposition of the material in a mixture of acids. The determination was performed on a SPECTRO ARCOS device using the calibration curve method with Merck standards.

The phase structure of the samples was measured by powder X-ray diffraction (XRPD) using a Rigaku Ultima IV diffractometer (Rigaku Corporation, Japan). X-ray diffraction records were obtained using CuKα radiation at an acceleration voltage of 40 kV, a current of 40 mA, a scanning speed/duration of 1°/min, a step width of 0.02° and a scanning range of 10-100°. The phase composition was evaluated using the ICDD PDF-2 2022 database.

The free CaO content was determined according to EN 459-2[16]. The principle of determination is based on dissolving free calcium oxide in water in a sucrose solution and titrating the resulting solution with hydrochloric acid. The free CaO content is calculated from the volume of hydrochloric acid used to neutralise the solution.

The moisture content of the analytical sample was determined by drying at 105 °C gravimetrically according to EN ISO 17892-1[17] for waste. The results of the determination of individual analyses are further related to their content in the dry matter of the sample.

The mortar samples were made in accordance with EN 196-1[18]. A standardised programmable laboratory mixing device of the type HOBART was used to prepare the mortars, meeting the specific requirements of the EN 196-1 standard. Slow speed: rotation around the axis of the broom 140±5/min, rotation around the axis of the container 62±5/min. Compared to the EN 196-1, the mixing time was 5 minutes.

The bulk density, flexural strength, compressive strength, porosity, and frost resistance were determined on samples of hardened mortar. Water absorption and frost resistance. The samples were tested at 14, 28, and 56 days of age. The bulk density of the hardened mortar was determined according to EN 1015-10[19]. The flexural strength and compressive strength were determined according to EN 1015-1120.The water absorption of the hardened mortars was determined according to EN ISO 1514821. Water absorption values were used to determine porosity. Porosity was determined according to the formula:

$$V_p = \frac{\rho \times m_{F1}}{\rho_{F1} \times m_d} \times\ 100\ [\%] \qquad (1)$$

Where: V_p porosity [%]
ρ bulk density of the dried test specimen [kg/m^3]
m_{F1} = m_s - m_d
ρ_{F1} density of the liquid, in this case water [kg/m^3]
m_{F1} weight of the liquid (water) absorbed by the test specimen
m_d weight of the dried test specimen [kg]
m_s weight of the water-soaked test specimen [kg]

The frost resistance of the mortars was tested in accordance with ČSN 72 2452[22], but the environmental parameters were adjusted. The samples were subjected to 3 cycles. One cycle lasted 6 hours, with the climate chamber set as follows: freezing at -8 °C for 4 hours, then thawing at +10 °C for 2 hours. The cycle was adjusted based on the assumption that the material would be used in a building structure below ground level, where the minimum and maximum temperatures fluctuate within a range with lower limit values than those recommended by the standard.

The pore size and pore size distribution were measured in a liquid nitrogen atmosphere by means of Thermo Scientific Surfer (Milan, Italy). The pore size was evaluated from the desorption curve of individual samples. Before determining the pore size, all samples were dried at 50 °C for 12 hours and degassed at 50 °C for 24 hours. The measurements were performed under two basic conditions:

- in the original compact state;
- on crushed experimental samples.

Wastes

Steel dust (O) and fly ash (P) from heat plant separators were selected for the experimental work. These are very fine-grained materials, as can be seen in figure 1. From the two types of fine-grained waste mentioned above, mixture No. 1 was prepared with a content of 80% steel dust and 20% fly ash. The mixture was homogenized by shaking it upside-down, which was used in experiments. The granulometry and chemical composition of the waste are shown in Tables 1 and 2. Content of Fe phases in dry sample of steel dust is shown in Table 3.

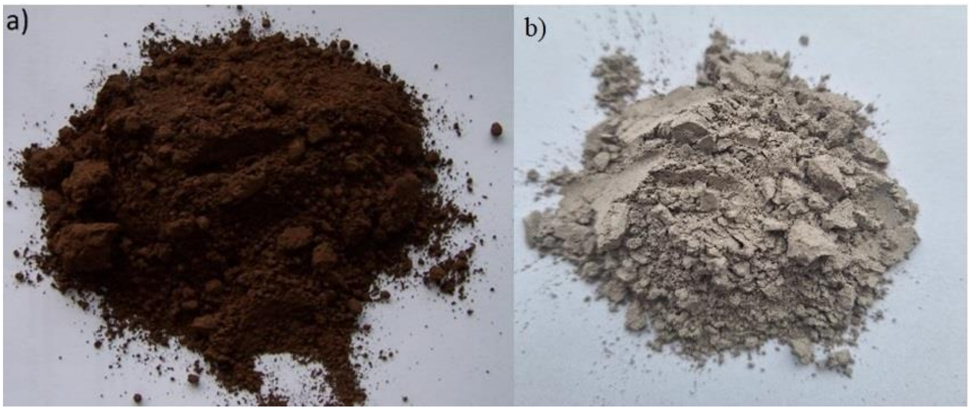

Figure 1: Photographs of waste materials, a) steel dust and b) fly ash.

Binder

The binder in the tested mortars was PROMPT natural cement[23-24]. This is a low-temperature, highly belitic cement. This type of cement is currently being researched for its unique properties and compared to ordinary Portland cement (OPC), it has a lower carbon footprint. It is interesting for use in repair mortars due to its excellent resistance to water-soluble salts. This material has significantly higher durability in damp and saline masonry environments than comparable OPC based materials. PROMPT natural cement is characterized by the chemical and mineralogical composition shown in Tables 4 and 5.

Mortars

Two different mortars were tested; the recipes are listed in Table 6. The components were dosed by weight. Drinking water from the tap was used. The filler was laboratory-grade silica sand with a fraction of 0.8 – 2 mm. The dosage according to Table 6 provides the amount of mortar for 3 samples measuring 40x40x160 mm.

Results and Discussion

Waste and cement PROMPT

Result of the granulometric analysis steel dust and flay ash shown in Table 1. The chemical composition of the waste samples used is in Table 2, the content of the main elements is expressed as oxides. Content of different phases of Fe shows Table 3.

Table 1: Granulometric composition of waste materials used for the preparation of composites.

Particle size [mm]	Particle size distribution [%]	
	O	P
1 – 0.2	14.2	0.5
0.2 – 0.125	18.4	0.4
0.125 – 0.04	45.6	25.9
<0.04	21.9	73.3

Table 2: Chemical composition of the waste samples.

Element	Dry matter content [wt.%]		
	O	P	Mix No. 1 (O+P)
Al_2O_3	5.29	11.5	6.53
CaO	18.6	33.6	21.6
Fe_2O_3	27.3	4.69	22.8
K_2O	4.29	<0.01	3.43
MgO	14.9	0.939	12.1
MnO	5.56	0.016	4.45
Na_2O	12.0	1.44	9.89
P_2O_5	0.135	0.053	0.119
SiO_2	4.62	32.0	10.1
SO_3	3.51	5.12	3.83
TiO_2	0.089	0.776	0.23
CaO(free)	6.23	20.0	8.98
LOI	9.61	9.05	

Table 3: Content of Fe phases in dry sample of steel dust.

Fe phase	Fe (total)	Fe (metal)	FeO	Fe_2O_3
Content [wt.%]	19.1	0.06	0.98	26.1

Chemical composition of PROMPT natural cement expressed in oxides is summarized Table 4. The main element of the PROMT is Ca and Si, which created different mixed oxide compounds, as show phase composition in Table 5.

Table 4: Chemical composition of PROMPT natural cement by manufacturer in mass %.

LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	MnO	TiO_2
8.14%	18%	7.68%	3.2%	55.1%	3.69%	2.38%	0.97%	0.14%	0.072%	0.065%	0.28%

Unlike the binder in the input waste materials, X-ray diffraction proved that the majority of the phases present were also chlorides, sulphates, hydroxides, or carbonates, as shown in Table 5.

Table 5: Mineralogical composition of PROMPT natural cement by manufacturer in mass %.

C_3S	C_2S	C_3A	C_4AF	$C_{12}A_7$	C_4A_3S	Periclase	Free lime	Calcite	Sulfates	Others, incl. amorphous phases
5 – 15%	40 – 60%	6 ± 2%	9 ± 2%	3 ± 1%	3 ± 1%	4 ± 1%	2 ± 2%	10 – 15%	3 ± 1%	10 – 5%

Mortars

The average values of the bulk density of hardened mortars are shown in the graph in Figure 2. The values were determined on samples in a dry state. The bulk density decreases with the age of the samples, which is more noticeable in mortar 2-E/1, and this effect is related to the formation of a hydrated structure in PROMPT natural cement. The higher bulk density values for mortar 2-E/3 are related to the higher filler content.

Table 6: Recipes of Mortars.

Component	Mortar 2E-1	Mortar 2E-3
Waste mixture No. 1	439 g	384 g
PROMPT Natural cement (Vicat, France)	110 g	96 g
Sand	110 g	384 g
Water	439 g	384 g

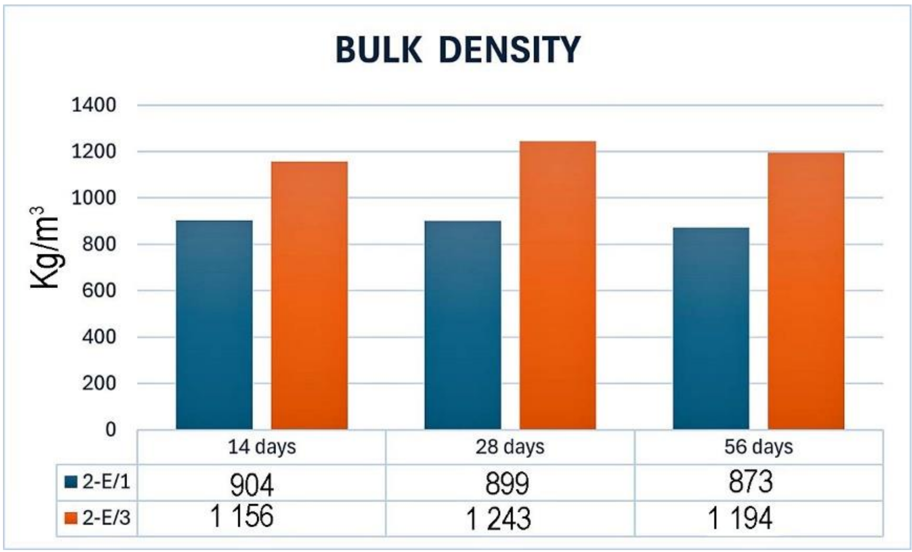

Figure 2: Average values of the bulk density of hardened mortars.

All mortar samples had very low flexural strength, which could not be measured using the standard method. The average compressive strength values are shown in the graph in Figure 3. Compressive strength increases with the age of the samples. Samples 2-E/3, achieve higher absolute compressive strength values. The anomaly of strength reduction in 28-day samples 2-E/1 is related to the dormant stage of PROMPT cement hydration and was more pronounced in this experiment in mortars with a higher binder content.

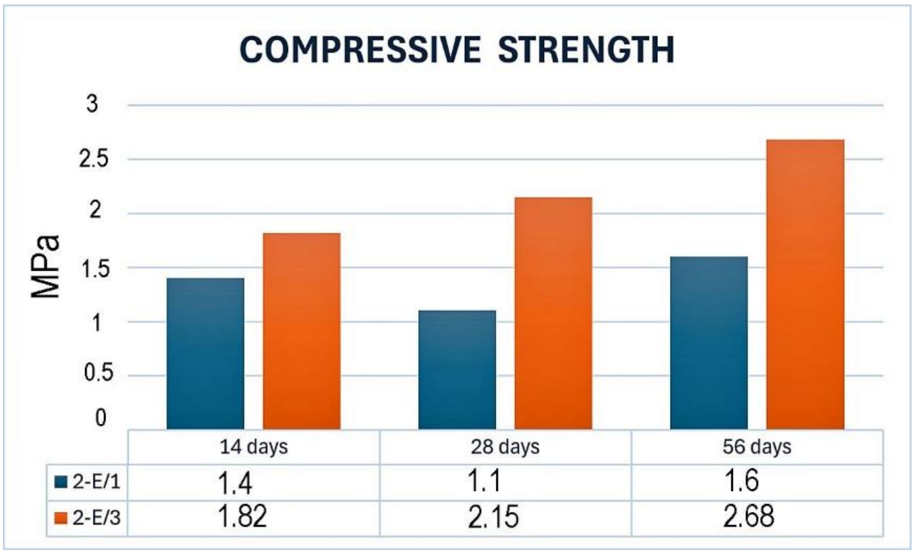

Figure 3: Average compressive strength values.

Absorbency was determined using a standard procedure, but the temperature in the dryer was lower, at 40 °C. The samples were dried to a constant weight. The gas porosimetry method for determining the nature of the pore structure was not suitable for this type of sample. Neither mode (compact and crushed sample) led to successful adsorption of gas (N_2) on the surface of the samples. Only sample 2E/1 showed successful adsorption of nitrogen on the surface, approximately 45%, after which adsorption did not proceed. Desorption did not occur at all. During the measurement, an indicative evaluation of the size of the surface pores was performed, with an average value of 7 nm. The porosity was therefore determined by calculation according to WTA Guideline No. 2-9-04/D. The results are shown in Figure 4.

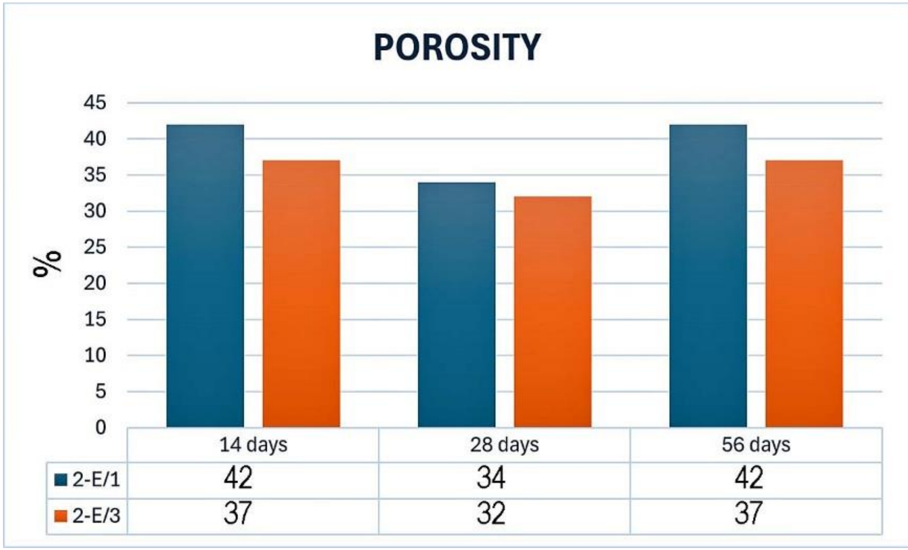

Figure 4: Average porosity values.

Table 7: Comparison of mortar properties with the requirements of WTA Guideline 2-9-04/D (Sheet 3). For samples aged 28 days.

Properties of hardened mortar	**Criterion value**	**Mortar 2E-1**	**Mortar 2E-3**
Bulk density (kg/m^3)	<1400	900	1 241
Compressive strength (MPa)	1.5 – 5	1.1	2.15
Porosity (volume %)	> 40	34	32

Table 8: Frost resistance. Average weight loss of test specimens at 28 days after cycling.

	Mortar 2E-1	**Mortar 2E-3**
Average weight loss (%)	8.6	9.2

The results of the frost resistance test showed that the mortars are frost resistant under the given conditions (see chapter Equipment and methods). Frost resistance is expressed as the average weight loss of test specimens in each set, and the data are shown in Table 8. Samples aged 28 days were tested.

As shown in Table 7, porosity values (Fig. 4) develop depending on the formation of the material structure. The formation of the structure of PROMPT cement-based mortars is specific and is not identical to the scenario of the formation of the structure of OPC-based mortars[25].

Waste mixture No. 1, as identified in Table 2, can be used to produce restoration and filling mortars for damaged, damp, and salt-contaminated masonry. The basic criteria for hardened mortars have been verified. The properties of fresh mortars will be tested in further studies. The properties of mortars related to pore structure formation deserve special attention in future research. This is particularly because the mechanism of structure formation differs from that of OPC-based reference materials.

Conclusion

This article discusses the results of an experimental study focused primarily on applied research, with the aim of identifying specific possibilities for the use of selected problematic (in terms of recycling) waste. The experimental study was the first step in the development of remediation mortars based on these specific fine-grained metallurgical wastes. The results of testing the properties of hardened mortars are acceptably consistent. This was the main concern of the authors, as both the natural binder and the waste mixture are naturally very heterogeneous materials in terms of their composition.

The compressive strength values for mortar 2-E/3 are satisfactory at 28 days of age, and contrary to expectations, the dormancy stage of PROMPT cement had no effect. Both mortars tested have very low, immeasurable flexural strength. For further development of the formulations, reinforcement of the mortar matrix with suitable particles and/or fibres can be considered. The vision for the next research is to fine-tune the recipe in relation to the properties of fresh and hardened mortar. The ultimate goal for further application research is to verify the basic requirements for construction products according to the relevant legislative and normative regulations for the product being developed.

The composition of the tested mortars reflects the requirements of sustainable development. The potential production of these mortars would help reduce the region's current environmental burdens. It would also contribute to the goals of the circular economy, resulting in a functional product with lower impact values according to the LCA methodology compared to products currently available on the building materials market.

The use of metallurgical waste for mortar production can result in new products with unique properties. This depends on the composition of the waste and its interaction with the binder system. This study verified the basic prerequisites for the use of selected metallurgical waste for the production of restoration and/or filling mortars for masonry.

In conclusion, waste mixture No. 1 has the potential for use in mortar mixtures with natural PROMPT cement. However, the development of the material is still in its initial phase.

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Sanační a výplňové malty na bázi metalurgických odpadů

Jana DAŇKOVÁ[a], Jana SEIDLEROVÁ[b, d], Adéla VALENTOVÁ[a], Petr BĚČÁK[b,d], Petr UNUCKA[c]

[a] *VŠB – Technická univerzita Ostrava, Fakulta stavební, Katedra stavebních hmot a diagnostiky staveb, Ludvíka Podéště 1875/17, 708 00 Ostrava – Poruba,*
[b] *VŠB – Technická univerzita Ostrava, Centrum nanotechnologií CEET, 17. listopadu 2172/15, 708 00 Ostrava – Poruba,*
[c] *ECOCOAL s. r. o,* Mrštíkova 4, 709 00 Ostrava 9,
[d] *VŠB – Technická univerzita Ostrava, Fakulta materiálově-technologická, Katedra chemie a fyzikálně-chemických procesů, 17. listopadu 15/2172, 708 00, Ostrava – Poruba*
e-mail: jana.dankova@vsb.cz, jana.seidlerovava@vsb.cz, adela.valentova@vsb.cz, petr.becak@vsb.cz, unuckap@plazkat.cz

Souhrn

V souladu s politikou udržitelného rozvoje je další směřování všech odvětví průmyslu a služeb směrem k cirkulární ekonomice velmi aktuálním tématem. Stávající analýzy na toto téma odhadují vysoký potenciál pro rozvoj cirkulární ekonomiky zejména v oblasti výroby oceli, cementu, betonu a stavebnictví. Tématem tohoto příspěvku je multidisciplinární experimentální analýza možnosti aplikace vybraných metalurgických odpadů jako hlavních surovin pro výrobu sanačních a výplňových malt pro zdivo. Snížení pevných částic v emisích v posledních letech vede k produkci tzv. jemnozrnných odpadů, které nelze bez úpravy recyklovat. Mezi takové patří především metalurgické odprašky ze suchého čištění odpadních plynů aglomerace, výroby železa a oceli, nebo popílky ze spalovacích procesů, cementáren aj. Hlavními složkami metalurgických odprašků jsou Fe_2O_3*, FeO,* Al_2O_3*, CaO, MgO, MnO,* SiO_2 *v různých hmotnostních poměrech, popílek ze spalovacích procesů obsahuje především* Al_2O_3*,* SiO_2*, CaO ve formě volného CaO, sírany. Oba druhy odpadů pak mohou obsahovat další prvky v množství menším než 1 hmot. %. Důležitou složkou pro recyklaci je obsah vápníku ve formě volného CaO, který slouží jako pojivo. Jemnozrnné metalurgické odpady – odprašky nebo směs odprašků a kalů – se smíchají s popílkem ze spalovacích procesů v hmotnostním poměru 4:1. Uvedenou směs lze zkusovět nebo přímo využít v dalších odvětvích průmyslu, a tedy i stavebnictví. V oboru stavebnictví je realizace sanačních opatření na konstrukcích a objektech zásadním přístupem pro eliminaci vzniku odpadů, prodloužení životnosti staveb a pro snižování hodnot environmentálních dopadů při výstavbě a užívání staveb. Výplňové malty jsou určeny pro vyplňování dutin a spár ve zdivu, případně pro sanaci degradovaných betonových a železobetonových konstrukcí. Sanační malty pro zdivo jsou materiály určené pro aplikaci sekundárních opatření pro snižování vlhkosti a zasolení zděných konstrukcí s cílem obnovit jejich funkce. Současný sortiment sanačních a výplňových malt je převážně založen na materiálech na bázi cementu, přírodních neobnovitelných surovin, případně se jedná o směsné polymercementové materiály. V této experimentální studii byly ověřovány vlastnosti malt, u kterých bylo 80 % cementu nahrazeno vybranými metalurgickými odpady a nebyly použity žádné přídavné polymerní látky pro úpravu jejich vlastností. Výsledky úvodní experimentální studie jsou perspektivní a budou použity pro další výzkum a vývoj řešené problematiky.*

Klíčová slova: *malty, přírodní cement, metalurgické odpady, stavební materiály, sanační malty pro zdivo*