EXPERIMENTAL RESEARCH ON THE RECOVERY OF SLAG FROM SECONDARY ALUMINIUM PRODUCTION FOR THE DEVELOPMENT OF CERAMIC AND REFRACTORY MATERIALS

Abdulkader JANPOLAT[1], Radu Vasile BUZDUGA[2], Constantin - Domenic STĂNCEL[1*], Mihai BUȚU[1], Nicolae CONSTANTIN[1], Maria Camelia MATACHE[1], Costinela OPREA SZIBLIK[1]

This study presents a technology for recycling secondary aluminium slag waste into ceramic block formulations. Using an experimental design matrix, eight recipes with variable slag proportions and particle sizes were analysed. Key properties - water absorption, density, porosity, and microstructure - were evaluated to identify the optimal variant. The results confirmed a formulation that ensures high waste incorporation without compromising quality. The findings demonstrate that secondary aluminium slag can be valorised efficiently and sustainably, reducing manufacturing costs and environmental impact.

Keywords: refractory materials, ceramic materials, industrial waste, aluminium slag, electron microscopy.

1. Introduction

Refractory materials are mainly manufactured from metal oxides such as alumina, silica, and magnesia. Typically, refractory products consist of mixtures of these oxides. Refractories can also be produced from non-oxide ceramic materials, including graphite or silicon carbide.

The availability of raw materials is one of the key factors influencing the refractory ceramics market [1-3, 4]. European refractory manufacturers must secure access to raw materials at competitive prices. One strategic option for European producers is the development of alternative raw material sources [2, 5-8]. This can be achieved through the integration of secondary raw materials or by recycling spent refractory materials.

Aluminium and its alloys are widely used in the manufacturing of various products. The production of aluminium-based alloys is undergoing continuous development and innovation. Newly engineered aluminium alloys are progressively

* Corresponding author: Constantin - Domenic Stăncel, e-mail: domenic.stancel@upb.ro
[1] National University of Science and Technology POLITEHNICA Bucharest, Romania
[2] SC CCPPR SA, Alba Iulia, Romania

replacing other metals in the fabrication of goods designed for medium- and long-term service life.

Aluminium recycling prevents approximately 90 million tons of CO_2 emissions annually. According to studies in the scientific literature regarding the importance of recycling, this process avoids the direct greenhouse gas emissions associated with primary aluminium production and results in energy savings of up to 95%. The reduction in emissions achieved through aluminium recycling has doubled since 1990.

Slag generated during secondary aluminium production contains compounds such as magnesium aluminate ($MgAl_2O_4$), aluminium nitride (AlN), quartz (SiO_2), metallic aluminium, alumina (Al_2O_3), periclase (MgO), and spinel phases of the (Mg, Si)Al_2O_4 type. Hot water washing enables the conversion of AlN to aluminium hydroxide ($Al(OH)_3$), which subsequently reacts with Na and K compounds, facilitating the removal of chloride contaminants from the waste material. Aluminium losses can easily reach 50%, and the final loss rate remains substantially high, which makes conventional recovery processes extremely inefficient.

At AS Metal, large amounts of secondary aluminium are produced through the recycling of a wide range of aluminium waste streams. Besides aluminium, the manufacturing process also generates gases and slag. In this study, an advanced valorisation pathway for secondary aluminium slag is proposed, consisting of its incorporation into formulations for producing refractory and ceramic blocks intended for various applications.

The slag resulting from the production process of secondary aluminium alloys at SC AS METAL COM SRL was chemically analysed in order to determine its composition and subsequently evaluate its potential for valuable reuse. The main compounds identified by X-ray diffraction analysis are: Al, Al_2O_3, MnO_2, $MgAl_2O_4$, Mg_2SiO_4, $CuAl_2O_4$, Fe_2O_3, $(Mn_2O_3)_3MnSiO_3$, $NiAl_2O_4$, and $ZnAl_2O_4$.

The results of the chemical analysis performed at the UNSTP București determining the elemental composition of secondary aluminium slag for two different particle size fractions are presented in Table 1.

Table 1

Constituent Elements of Secondary Aluminium Slag

Element in slag	**Slag with particle size below 0.4 mm, composition in wt%**	**Slag with particle size above 0.4 mm, composition in wt%**
Al	69.8	71.59
Mg	9.65	7.037
Na	8.06	6.58
Si	5.061	6.289
Cu	2.075	2.871
Ca	1.314	1.314
Fe	1.135	1.497

Cl	1.048	0.7049
K	0.3534	0.3247
S	0.3216	0.3433
Ti	0.3132	0.3643
Zn	0.2424	0.3123
Mn	0.1547	0.2003
Ni	0.1187	0.132
P	0.1127	0.1199

The compositional analyses were performed using an energy-dispersive spectrometer (EDAX – Saphire) at an accelerating voltage of 30 kV, with an electron beam spot size of 5.5 µm and a distance of 10 mm between the pole piece and the sample surface, at an angle of 35° between the sample surface and the X-ray detector. The analyses were conducted at a magnification of 100× over five different fields, and the results reported in this study represent the average of the individual quantitative measurements.

It should be noted when interpreting the data that microanalysis is a method for characterizing the composition of microvolumes. To determine the chemical composition of heterogeneous material volumes, additional analyses using atomic or mass spectrometry are required. Figures 1 - 3 present the EDAX analyses for slag with different particle size fractions.

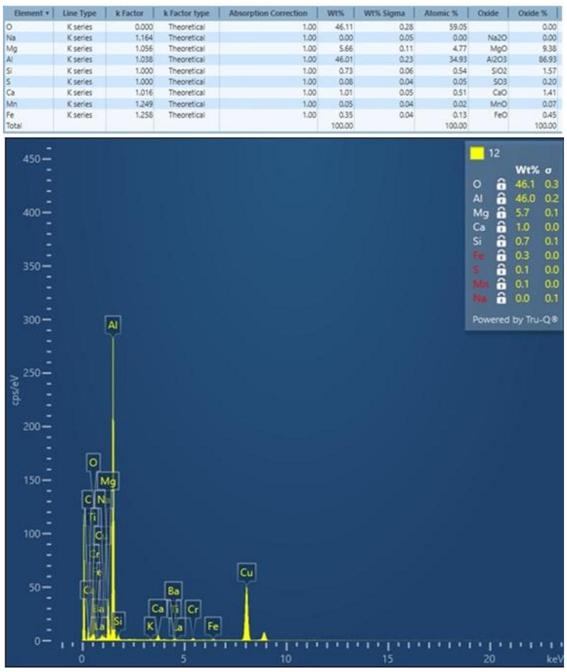

Fig. 1. EDAX analysis of slag with particle size below 0.5 mm

Element	Line Type	k Factor	k Factor type	Absorption Correction	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %
O	K series	0.000	Theoretical	1.00	47.01	0.30	60.00		0.00
Na	K series	1.164	Theoretical	1.00	0.00	0.00	0.00	Na2O	0.00
Mg	K series	1.056	Theoretical	1.00	0.46	0.07	0.38	MgO	0.76
Al	K series	1.038	Theoretical	1.00	51.58	0.27	39.03	Al2O3	97.46
Si	K series	1.000	Theoretical	1.00	0.50	0.07	0.37	SiO2	1.08
S	K series	1.000	Theoretical	1.00	0.09	0.05	0.06	SO3	0.22
Ca	K series	1.016	Theoretical	1.00	0.22	0.04	0.11	CaO	0.31
Mn	K series	1.249	Theoretical	1.00	0.05	0.05	0.02	MnO	0.06
Fe	K series	1.258	Theoretical	1.00	0.08	0.05	0.03	FeO	0.11
Total					100.00		100.00		100.00

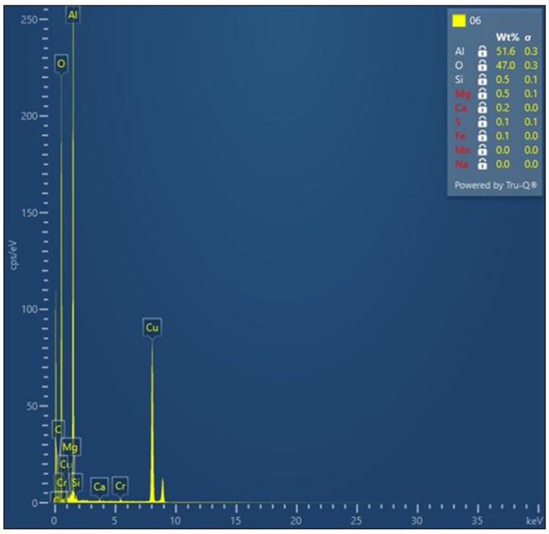

Fig. 2. EDAX analysis of slag with particle size between 0.5 and 1 mm

Element	Line Type	k Factor	k Factor type	Absorption Correction	Wt%	Wt% Sigma	Atomic %	Oxide	Oxide %
O	K series	0.000	Theoretical	1.00	47.01	0.30	60.00		0.00
Na	K series	1.164	Theoretical	1.00	0.00	0.00	0.00	Na2O	0.00
Mg	K series	1.056	Theoretical	1.00	0.46	0.07	0.38	MgO	0.76
Al	K series	1.038	Theoretical	1.00	51.58	0.27	39.03	Al2O3	97.46
Si	K series	1.000	Theoretical	1.00	0.50	0.07	0.37	SiO2	1.08
S	K series	1.000	Theoretical	1.00	0.09	0.05	0.06	SO3	0.22
Ca	K series	1.016	Theoretical	1.00	0.22	0.04	0.11	CaO	0.31
Mn	K series	1.249	Theoretical	1.00	0.05	0.05	0.02	MnO	0.06
Fe	K series	1.258	Theoretical	1.00	0.08	0.05	0.03	FeO	0.11
Total					100.00		100.00		100.00

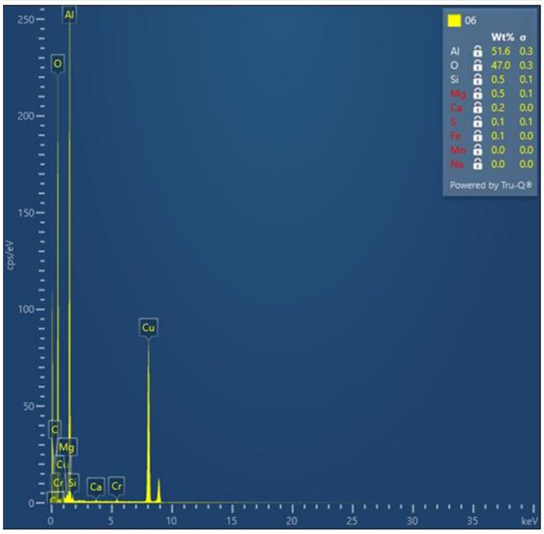

Fig. 3. EDAX analysis of slag with particle size between 1 and 2 mm

The valorisation of secondary aluminium slag involves converting waste into valuable products through a multi-stage process that recovers metals and salts while producing new materials [1]. These wastes contain approximately 3 - 9 wt% metallic aluminium, 20 - 50 wt% oxides such as aluminium oxide (also referred to as non-metallic products), 20 - 75 wt% flux salts, and other components in smaller proportions.

Due to their composition and potential reaction with water, saline slag is classified as hazardous waste and is included in the European Waste Catalogue, meaning it must be disposed of in landfills or secure storage facilities [1]. Aluminium slag waste can be used as a base material for the production of construction bricks by incorporating it in varying proportions into the manufacturing formulations of refractory bricks.

This approach efficiently manages hazardous industrial waste while simultaneously producing bricks that meet or exceed standard requirements for compressive strength and other performance properties. Improper disposal of raw slag can release toxic and flammable gases, such as ammonia and methane, upon contact with water, posing a serious risk to both the environment and human health [1, 9-14].

To enable the efficient valorisation of slag waste generated by a secondary aluminium producer, experimental research was conducted to assess the feasibility of using this waste as a raw material for the production of ceramic blocks, specialty ceramics, or refractory bricks. These materials can be used in civil or industrial construction or as masonry for thermal units where internal temperatures do not exceed 1000 °C.

2. Materials, Equipment and Method

2.1. Materials

The research was carried out at an industrial enterprise specializing in the design and production of refractory and ceramic materials, SC CCPPR SA in Alba Iulia. The study focused on the valorisation of slag with two different particle sizes as a raw material in the formulations for producing ceramic blocks.

The research was conducted using a 2 experimental design matrix, resulting in the development of eight compositional variants of manufacturing recipes, each containing variable proportions of secondary aluminium slag.

2.2. Equipment

Laboratory Equipment for Testing Raw Materials and Finished Products. The laboratory equipment used is intended to determine the main quality characteristics of the raw materials and the manufactured products. This equipment is capable of performing measurements for: Chemical analysis / X-ray fluorescence spectrometry (calibrated with certified reference materials and internal standards), Water absorption, Porosity, Dimensions / dimensional tolerance, Thermal shock resistance, Flexural strength and compressive strength.

The main laboratory equipment used is shown in Figure 4.

a b c d e

Fig. 4. Laboratory equipment for testing raw materials and finished products: a) High-resolution spectrometer, b) Compact high-temperature furnace, c) AS 200 Control sieve analyser, d) Microwave system for pressure-assisted mineralization, e) AL204 analytical balance. [4,11,12, https://www.ccppr.ro/]

2.3. Method

Following an experimental study using a 2-factorial design, eight variants of slag proportion were developed in the manufacturing recipes. Considering that the technology for producing ceramic and refractory materials using industrial wastes, such as slag from secondary aluminium production, requires raw material preparation identical to that used in conventional refractory brick manufacturing, all the necessary equipment for this process is available at SC CCPPR SA, Alba Iulia [4, 11, 12].

The raw materials were chemically analysed to determine the percentage composition of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, and K_2O. Loss on ignition, particle size distribution, density, and moisture content were determined. The results of the analyses are presented in Table 2.

Table 2

Chemical composition of the raw materials

Raw material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	PC %
Chamotte SA 40A	77.15	18.25	0.75	-	-	-	-	6.25
Secondary Al Slag	5.28	69.09	3.84	2.92	12.20	0.24	0.07	14.28
Portland cement	22.50	6.50	2.5	63.4	4.5	0.25	0.35	2.20 (SO_3)

Based on the physicochemical analysis of the raw materials, the formulations were established as shown in Table 3.

Table 3

Manufacturing formulations of the experimental ceramic samples

Raw material	**Alum. cement %**	**Fine slag %**	**Coarse slag %**	**Chamotte SA 40A fine %**	**Chamotte SA 40A granules %**	**Water ml**
R1	25	2.5	-	12.5	60	350
R2	25	5	-	10	60	260
R3	25	10	-	5	60	260
R4	25	15	-	2	60	290
R5	25	-	2.5	15	57.5	250
R6	25	-	5	15	55	250
R7	25	-	10	15	50	300
R8	25	-	15	15	45	270

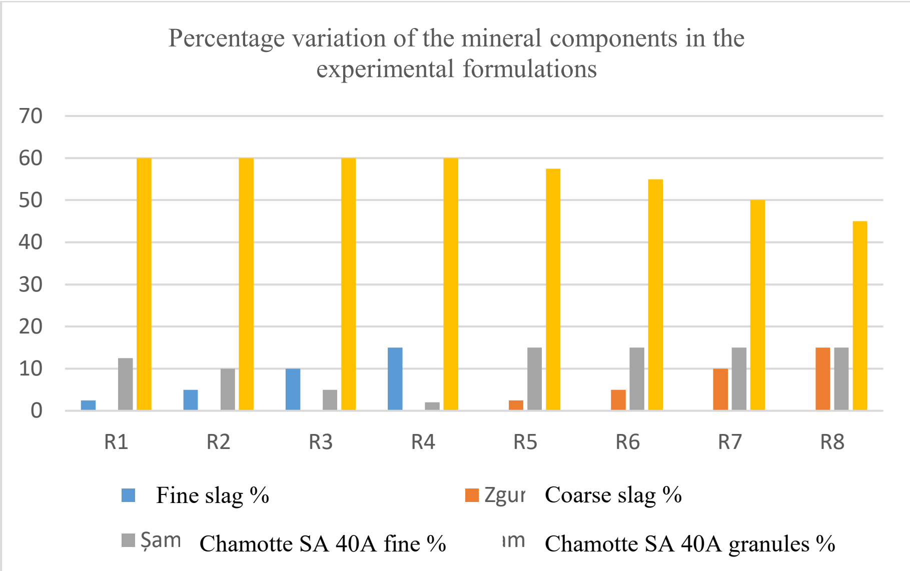

Fig. 5. Proportion of components in the experimental manufacturing formulations

The components of each formulation were weighed gravimetrically and then mixed in the laboratory using a paddle mixer. In the initial phase, these formulations were tested with a higher proportion of hydraulic binder based on gypsum. The mixture was moistened to achieve a moisture content of 10–15%.

The resulting mixture was pressed in a friction press at 120 tF/m² in four stages, aiming for maximum deaeration of the mass. Cubes measuring 100 × 100 mm were produced. Examples of the experimental cubes are shown in Figure 6.

Fig. 6. Examples of experimental cubes for different formulations

The experimental cubes were naturally dried for 10 days. It can be observed that the batch formulations are diverse, developed based on the experience of the industrial operator SC CCPPR SA, Alba Iulia, where the experimental research was conducted.

3. Results

3.1. Values of the main quality characteristics of the obtained ceramic products

For all experimental cubes from the eight prepared samples, the main quality properties were determined through laboratory measurements of water absorption, density, and porosity, as well as optical microscopy investigations of their microstructure. The results of the physical property determinations for formulations 1 - 8 are presented in Table 4.

Table 4

Results of the physical property measurements for formulations 1 - 8

Formulation	Absorption %	Density g/cm3	Porosity %
R1	10.90	2.85	24.15
R2	10.04	2.77	25.74
R3	9.5	2.65	26.21
R4	7.8	2.56	26.70
R5	10.40	2.70	24.30
R6	9.70	2.65	25.20
R7	8.80	2.62	26.75
R8	7.65	2.59	27.9

The graphical variations of the main quality characteristics of the ceramic and refractory materials for the experimental samples are presented in Figure 7.

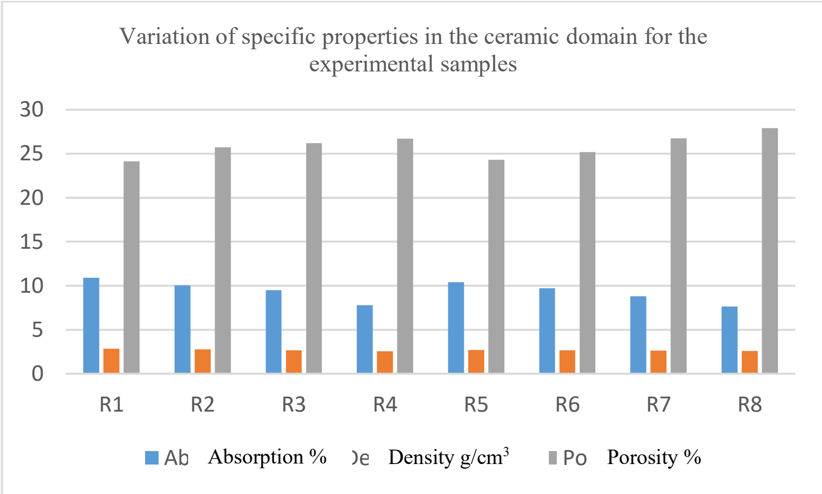

Fig. 7. Variation of specific quality characteristics of ceramic and refractory materials for the eight experimental samples

Analysing the results in terms of the main quality characteristics of the ceramic and refractory materials, formulation R5 is considered the most suitable for the valorisation of slag obtained from secondary aluminium production.

3.2. Laboratory Studies on the Compressive Strength of Experimental Samples

For the compression test, a Walter + Bai LFV 300 universal testing machine was used. The test was displacement-controlled at a rate of 5 mm/min. The results recorded for the eight experimental samples are presented in Figures 8 - 15, while the comparative behaviour of samples made with fine and coarse slag is shown in Figures 16 - 18.

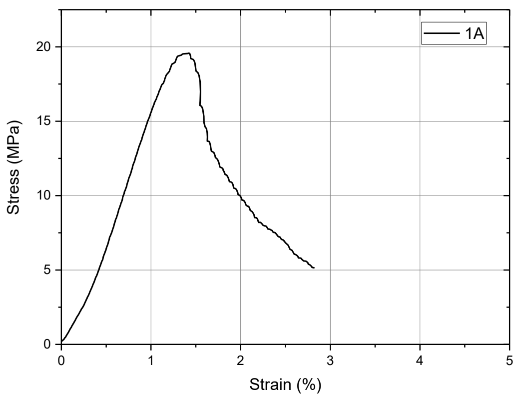

Fig. 8. Compressive behaviour of experimental sample R1

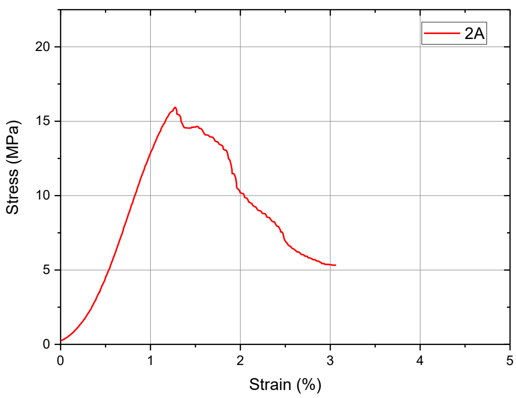

Fig. 9. Compressive behaviour of experimental sample R2(2A)

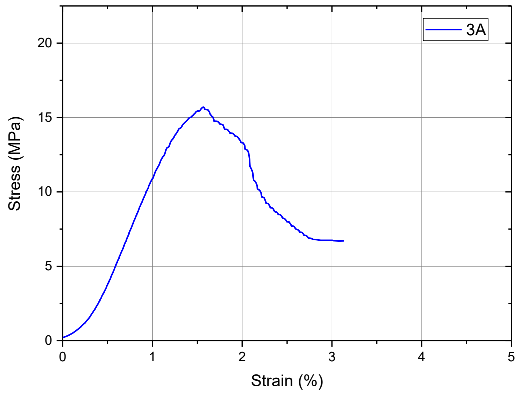

Fig. 10. Compressive behaviour of experimental sample R3(3A)

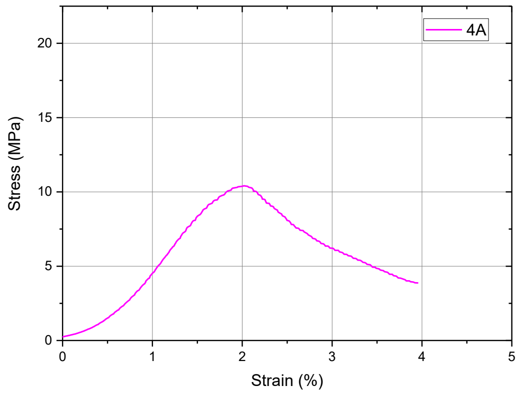

Fig. 11. Compressive behaviour of experimental sample R4(4A)

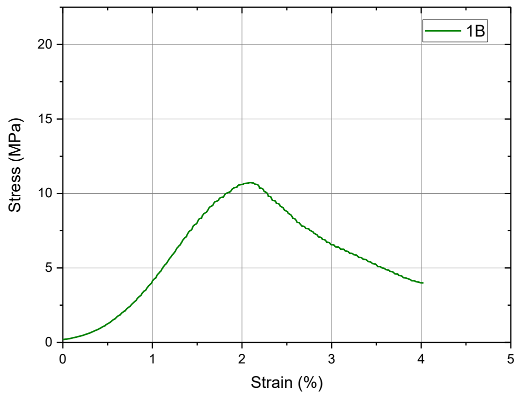

Fig. 12. Compressive behaviour of experimental sample R5(1B)

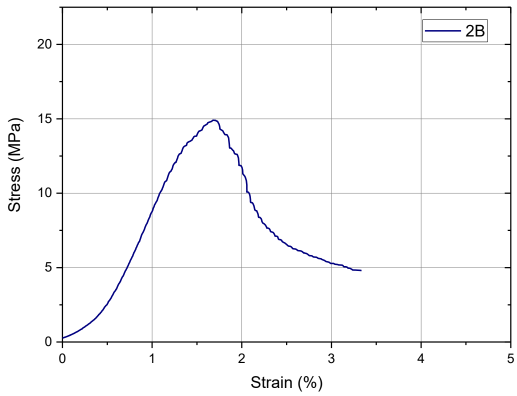

Fig. 13. Compressive behaviour of experimental sample R6(2B)

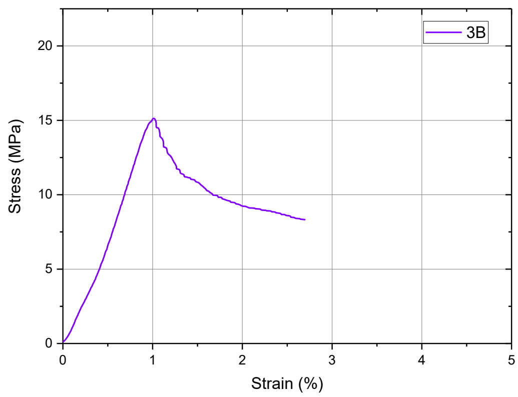

Fig. 14. Compressive behaviour of experimental sample R7(3B)

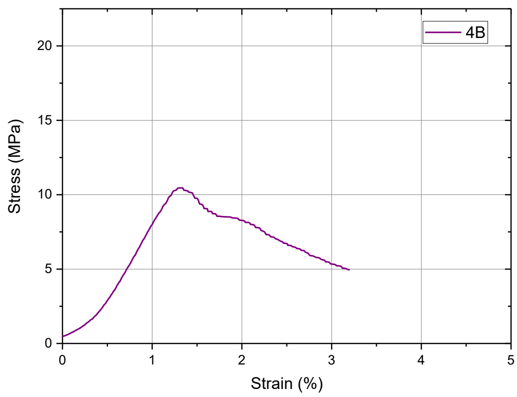

Fig. 15. Compressive behaviour of experimental sample R8(4B)

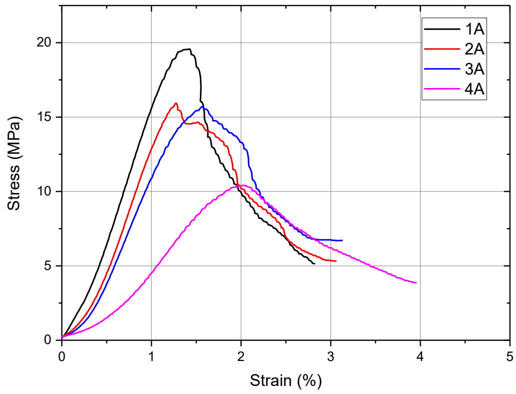

Fig. 16. Compressive behaviour of experimental samples R1(1A)-R4(4A)

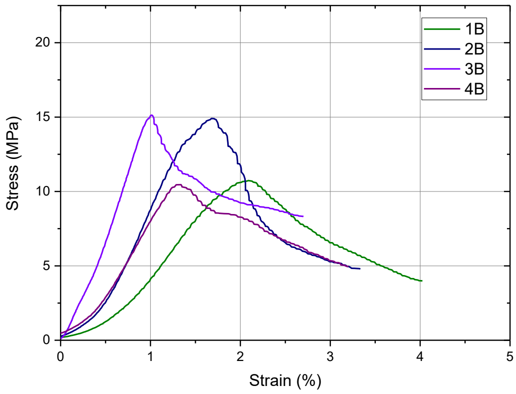

Fig. 17. Compressive behaviour of experimental samples R5(1B)-R8(4B)

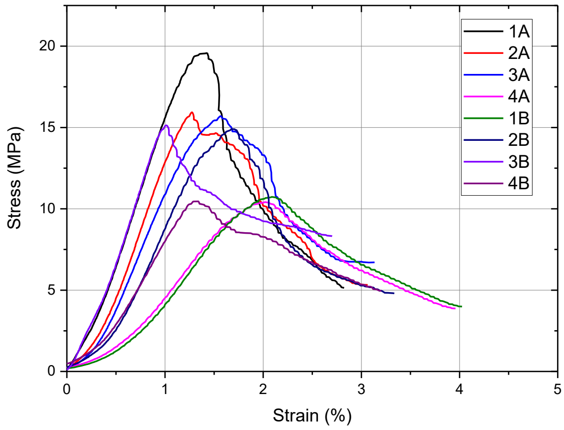

Fig. 18. Comparative compressive behaviour of all experimental samples

The following parameters were determined: the equivalent compressive modulus of elasticity (E), calculated as the slope of the linear portion of the stress–strain curve from the moment the specimen is seated; the compressive strength (Rc); and the strain at compressive strength (e@Rc).

Table 5

Measured mechanical parameters of the experimental samples

Samples	E[Mpa]	Rc[Mpa]	e@Rc [%]
R1(1A)	1870.74	19.58	1.42
R2(2A)	1732.87	15.94	1.28
R3(3A)	1445.29	15.70	1.57
R4(4A)	789.61	10.41	2.00
R5(1B)	811.85	10.74	2.09

R6(2B)	1373.55	14.91	1.73
R7(3B)	1874.30	15.13	1.02
R8(4B)	1055.74	10.46	1.34

Analysing the results of the laboratory compression tests for the eight experimental samples, the highest compressive strength was observed in sample R1 (1A). This sample was prepared using a formulation in which secondary aluminium slag accounted for 2.5%, while the largest proportion, 12.5%, consisted of fine-grained SA 40A chamotte.

Among the samples prepared using coarse secondary aluminium slag in the formulation, the highest compressive strength was observed in sample R7 (3B). This sample was made with a formulation containing 10% slag, 15% fine-grained SA 40A chamotte, and 50% coarse-grained SA 40A chamotte.

Regarding compressive strain, the highest values were recorded for experimental samples R4 and R5, while the lowest values were observed for samples R2 and R7.

Finding an optimal solution is very difficult, but with a reasonable approximation, good compressive strength values can be observed in samples R1 - R4, which were prepared using fine slag in their formulations. Among these, sample R1 exhibits the best performance. This can be partly explained by the fact that coarse slag in the formulations tends to generate higher porosity in the experimental samples.

4. Conclusions

The use of secondary raw materials and recycled refractory materials for refractory production was the focus of the experimental research. Slag waste generated during secondary aluminium production by a major industrial operator was used as a raw material in the formulations for experimental samples, with varying proportions of fine or coarse slag incorporated into the recipes.

Following an experimental study using a 2-factorial design, eight variants of slag proportions were developed: four using fine slag and four using coarse slag.

According to standards for defining the quality of ceramic and refractory materials, the analysed quality indicators are water absorption of the finished product (measured in %), density of the finished product (measured in g/cm³), and porosity of the finished product (measured in %).

It was found that slag waste can be successfully used in formulations for producing marketable refractory materials. The maximum proportion of slag, both fine and coarse, that can be incorporated into the recipes is 15%. The finished products exhibited good values for the main quality characteristics tested, considering their intended use, namely: 10.90% water absorption, 2.85 g/cm³ density, and 27.9% porosity.

The results of the experimental research demonstrated the feasibility of efficiently recycling slag from secondary aluminium production by incorporating this waste into the manufacturing formulations of products at SC CCPPR SA, Alba Iulia. These products include ceramic blocks used in refractory masonry for equipment operating at temperatures not exceeding 1000 °C.

The recycling of this waste has beneficial implications for both the manufacturing costs of refractory products and the protection of the environment. The use of aluminium slag (AS) in bricks provides a sustainable waste management solution, reducing the need for landfill space and the consumption of natural raw materials such as clay and sand. Ceramic and refractory products that incorporate specific amounts of slag in their raw material formulations will exhibit mechanical and physical properties suitable for industrial use.

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