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EXPERIMENTAL RESEARCH ON THE RECYCLING OF REFRACTORY WASTE OF MULCORITE IN THE PRODUCTION OF COMPONENT PARTS OF THE CAST IRON NETWORK

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Abstract: Ceramic and refractory materials are needed in many fields of activity, necessary for the development of human society, such as steel, non–ferrous metallurgy, chemistry, construction. The raw materials for obtaining refractory products are in short supply on the world market. In the activity carried out by leading economic agents, significant quantities of refractory waste are continuously generated, which must be neutralized and efficiently utilized. In this paper, the authors present the results of experimental research aimed at finalizing a technology for recycling mulcorite waste generated by an economic agent producing porcelain products. The physicochemical characteristics of this type of waste were determined and several technological variants of using mulcorite waste were tested, after an initial processing, in the manufacturing recipes of some pieces of refractory material component of the cast iron casting network from a manufacturer of gray iron castings. After testing eight variants of the composition of the manufacturing recipes, the best variant was chosen, which ensures the use of a maximum percentage of waste but also good values of the quality properties of the products obtained. For this, the main qualitative properties of the finished products were determined by measurements in the laboratory for the absorption coefficient, density and porosity, as well as by optical microscopy investigations on the structure of the finished products. The results of the conducted research demonstrated the possibility of efficient utilization of mulcorite waste, with beneficial effects on manufacturing costs and the environment.
Keywords: refractory materials, ceramic materials, industrial waste, mulcorite, cast iron, electron microscopy

1. INTRODUCTION

Technical ceramics and refractories are as important to modern society as they are to the steel industry. There is a wide variety of refractory materials in terms of raw materials, general composition and manufacturing method.[1–3]

The requirements for refractory materials are very diverse, they must withstand:
- temperatures with low values, well below the threshold of zero degrees, or at very high temperatures, up to 2200°C;
- to sudden temperature changes that cause high tensile stresses that can cause thermal shock resulting in cracking or fracturing;
- to abrasive forces at both high and low temperatures;
- to the corrosive action of the slag, with an acidic or basic character;
- at high pressures exerted on the refractory materials by the metal melt;
- to the action of gases, including CO, SO_2, Cl, CH_4, H_2O and oxides and volatile salts of metals, all of which are capable of penetrating and reacting with the refractory material.

Refractories are mainly made from metal oxides such as alumina, silica, magnesia. Refractory materials are usually mixtures of these oxides.

Refractories can also be made from non–oxide ceramics such as graphite or silicon carbide. The availability of raw materials is one of the key factors of the refractory ceramics market. [4–7]

The purity of the raw material has a direct impact on the quality of the ceramic product. The most used refractory raw materials are refractory clays.

The refractory industry is the third largest industry in Europe. Refractories represent 17% of the ceramic market in Europe [4,5]. The European Federation of Refractory Manufacturers (EPR) estimated that the European refractories industry employed 24,000 people in 2020. Refractories production was around 5 million tones and turnover was €3.77 billion in 2020. [5–7]

European refractories production represents 14% of the 40 million tons consumed worldwide. Thus, European refractories producers must ensure access to raw materials at a competitive price. One option for them is to develop another source of raw materials. [4–8] This could be achieved by implementing secondary raw materials or by recycling used refractory materials.

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Table 1 shows the worldwide consumption of refractory materials in 2020.

Raw materials represent 40–60% of the price of a finished refractory product [8].

The steel industry consumes a significantly larger amount of refractories compared to other industries. Refractory consumption has decreased from 25–30kg/ton of steel to 10kg/ton in Europe, Japan and the USA [6].

It should be taken into account that high consumption of refractories increases the cost of the manufactured product. Thus, the cost of refractory ceramics represents 1–3% of the cost added to the finished product; the cost is relatively small compared to the large quantities of refractory materials consumed.

Table 1. World consumption of refractory materials [1,4,5]

Raw materials	Proportion of world consumption of refractory raw materials, [%]
Refractory clays	46.0
Magnesia	26.0
Recycled refractories	7.0
Calcined bauxite	4.0
Molten alumina	3.0
Dolomite	3.0
Tabular alumina	2.0
Calcined alumina	2.0
Other (consumption < 1.0%)	7.0

Refractory consumption is highest in applications such as furnaces and converters. The weight of the types of refractories and the consumption of refractories in different applications in the steel industry are presented in Table 2 and in Table 3 – the types of refractory materials and their applications in the steel industry are presented.

The refractory industry is highly dependent on the availability of raw materials. The availability of raw materials is a concern for European and North American industries. However, finding new sources of raw materials is difficult.

Therefore, the use of secondary raw materials and recycled refractories for refractories production has been an important research topic. Recycling spent refractory materials for refractory raw material is an opportunity for companies to reduce the amount of imported raw material used in their processes.

Table 2. Average specific consumption and product shares of refractory materials in different applications in the steel industry [1,4,5]

Application	Average consumption, (kg/ton)	Product share	
		Formed, (%)	Unformed, (%)
Blast furnace	1.2	75	25
Oxygen converters	5.1	30	70
Electric arc furnace	6.2	92	8
AOD–Converter	3.3	90	10
Steel treatment ladle	1.3	50	50
RH and DH units	1.2	10	90

Table 3. Refractory materials and their applications in the steel industry [1,4,5]

Refractory material	Application
Silica (> 93% SiO_2)	Acid slag Induction Furnaces, Electric Arc Furnace, Vaults, Continuous casting bricks, Coking Batteries
Chamotte (30–45% Al_2O_3)	Blast Furnaces, Furnaces for heat treatment, Liners for ovens
Alumina (45–56% Al_2O_3)	Steel Treatment Pots, Electric Arc Furnace vaults
High purity alumina (> 56% Al_2O_3)	Refractory masonry furnaces
Magnesia (> 80% MgO)	Electric Arc Furnaces, Steel Treatment Pots, LD and OD Converters
Chromite–magnesite (25–55% MgO)	Electric Arc Furnace vaults
Dolomite (> 36% and < 60%MgO)	Electric Arc Furnaces, Steel Treatment Pots, LD and OD Converters

Recycling of refractory materials has taken off due to increased environmental awareness and increased costs for waste disposal. [11–16] One of these applications was the reuse of spent refractories as recycled raw material for the manufacture of refractories.

Mulcorite is part of the group of aluminous refractory materials, it is a very valuable type of material that is found spread over a very small area in nature, but it can be made artificially through a complicated technology. [1,13–15] The chemical composition and physical characteristics are presented in Tables 4 and Table 5.

This paper will present the experimental research carried out within the economic agent SC CCPPR SA Alba Iulia, with the objective of obtaining the component parts of the cast iron casting assembly from SC SATURN SA Alba Iulia, using them as raw material in the manufacturing recipes waste mulch taken from SC APULUM Alba Iulia. The company SC APULUM SA Alba Iulia [17], has been producing a diversified range of ceramic products, various porcelains for over 50 years. This company uses mulcorite slabs as a bed for burning raw products. After a number of cycles of use, mulcorite tiles lose their properties and break, become unusable and are considered waste.

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Table 4. Chemical analysis of mulcorite [1]

Oxide type	Percentage composition, (%)
SiO_2	32.0 – 35.0
Al_2O_3	57.0 – 60.0
TiO_2	0.3 – 0.4
Fe_2O_3	0.6 – 0.7
MgO	4.0 – 5.0
Alkalies ($Na_2O + K_2O$)	0.3 – 1.2

Table 5. Physical characteristics of mulcorite [1]

Physical characteristic	Value
Linear thermal expansion, ($10^{-7}K^{-1}$)	26.0 – 30.0
Bulk density, (g/cm^3)	2.1 – 2.5
Water absorption, (%)	12.0 – 14.0
Apparent porosity, (%)	25.0 – 28.0
Modulus of compressive strength, (daN/cm^2)	360.0 – 400.0
Modulus of cold rupture, (daN/cm^2)	140.0 – 170.0
Modulus of rupture after heating at 100°C with cooling in cold water at 20°C, (daN/cm^2)	90.0 – 100.0

2. MATERIALS AND EQUIPMENT

Materials

SC SATURN S.A. Alba Iulia is one of the most important Romanian foundries for lamellar and nodular gray cast iron parts. The cast iron casting assembly at this commercial company is shown in Figure 1.

Figure 1. Cast iron assembly from SC SATURN SA Alba Iulia and its component parts

Some subassemblies of the casting network are surface protected by adding a special fireclay that gives the appearance of glaze, as seen in Figure 2.

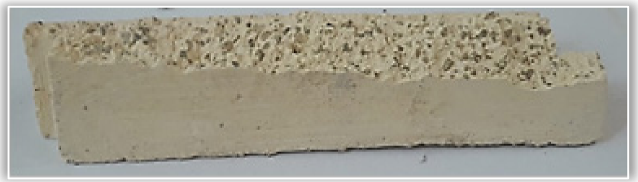

Figure 2. Cast iron assembly component part with glazed surface

Ceramic binder raw materials, clay, inorganic binder raw materials, colloidal silica, hydraulic binder raw materials, white cement and Portland cement, degreasing raw materials (aggregate), mulcorite refractory waste, fireclay were used in the manufacturing recipes of these parts and slag (fired clay in the deposit). Figure 3 shows the appearance of a special fireclay that gives the surfaces a rough, glazed appearance.

Figure 3. Special chamotte used in manufacturing recipes

Laboratory equipment for tests on raw materials and the finished product

The laboratory equipment used is intended to determine the main quality characteristics of raw materials and finished products. These devices can perform measurements for:

- chemical analysis/ X–ray fluorescence spectrometry (calibration – with certified reference materials, with internal standards);
- water absorption;
- porosity;
- dimensions / dimensional tolerance;
- thermal shock resistance;
- resistance to breaking and resistance to compression.

The main laboratory equipment used are shown in Figure 4.

Figure 4. Laboratory equipment for testing raw materials and finished products: (a) High resolution spectrometer; (b) High temperature compact oven; (c) AS 200 Control sieve analyzer; (d) Microwave system for mineralization under pressure; (e) Analytical balance type AL204, Mettler–Toledo [11,12,18]

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The industrial equipment components of the technological flow

The industrial equipment components of the technological flow of production of ceramic and refractory blocks and parts existing at the economic agent where the research and their characteristics were carried out are: [11,12,18]

— raw material storage hopper, productivity 15t/h, vat diameter 2400mm, 2 crushing drums, diameter 1440mm, width 500mm, weight 1200kg, electric motor drive 30kw/1000rp;
— crusher with rollers, throughput 6÷18t/h;
— bucket elevator, lifting height 11m, throughput 5÷18t/h;
— belt conveyor, width 500mm, length 8m;
— reversible belt conveyor, width 500mm, length 4m;
— conveyor with plates, throughput 3÷10t/h, inclination 20°, length 7500mm;
— vibrating sieve with a cloth, the active surface of the cloth 1200x2500mm;
— dosing scale, maximum dose 500kg, bucket volume 0.6m^3;
— gravimetric dispenser, maximum capacity 500kg;
— dispenser with level glass, capacity 120 l;
— jaw crusher, type 6A, throughput 8÷12t/h;
— Eyrich mixer, capacity 700 l;
— friction press, pressing force 1177N;
— electric hoist, maximum load 1.6tf.

Method

Following an experimental research using the matrix experiment 2^3, 8 variations of the percentages of mulcorite waste in the manufacturing recipes were made.

Considering the fact that the production technology of the components of the cast iron casting assembly requires a preparation of raw materials identical to that for the production of refractory bricks according to the classic technology, all the machines in that flow exist within SC CCPPR SA Alba Iulia and the location of the machinery does not undergo any change compared to the existing flow at the present time. [18]

The raw materials were analyzed from a chemical point of view, determining the percentage of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O and K_2O. Also, the calcination losses, grain sizes, density and moisture were determined. The results of the analyzes are presented in Table 6.

Table 6. Chemical composition of raw materials [1]

Raw materials	Chemical composition mass, (%)							CL, (%)
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	
Clay	60.56	24.72	1.76	1.30	1.20	1	1	9.30
Mulcorite waste	34.00	59.45	0.65	0.80	4.50	0.25	0.35	8.50
Sand (Colloidal Silica)	87.50	–	–	–	–	3.70	8.80	7.70
Portland cement	22.50	6.50	2.50	63.40	4.50	0.25	0.35	2.20 (SO_3)
White cement	25.75	6.75	0.75	64.55	1.50	0.40	0.30	2.35 (SO_3)
Multibeam	24.50	3.50	2.00	65.50	4.00	0.25	0.25	2.50

From the raw materials analyzed from a physico–chemical point of view, recipes were established according to Table 7.

Figure 5. Examples of experimental cubes for different recipes [2]

Table 7. Shares of raw materials in manufacturing recipes, (%) [1]

Recipe	Clay	Mulcorite waste	Sand	Portland cement	White cement	Multibat
R1	50	30	5	15	–	–
R2	60	20	10	10	–	–
R3	40	40	5	15	–	–
R4	50	15	10	5	5	15
R5	45	15	15	5	15	5
R6	25	35	10	5	20	5
R7	25	35	5	10	15	5
R8	20	40	20	20	–	–

The components of each recipe were dosed gravimetrically and then mixed in the laboratory in the paddle mixer. In these first recipes, the use of hydraulic binder based on plaster was tried in the first phase in a larger proportion. Wetting of the mixture was done at a humidity of 10–15%.

The obtained mixture was pressed in the press with a friction of 120tf/m^2, in 4 stages, aiming at the most advanced deaeration of the mass. 100x100mm cubes were obtained, 3 cubes for each recipe. The experimental cubes were dried, naturally differentiated 7, 10 and 14 days. The experimental cubes are

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presented in Figure 5. The graphical variations of the components of the raw material in the experimental samples are shown in Figure 6.

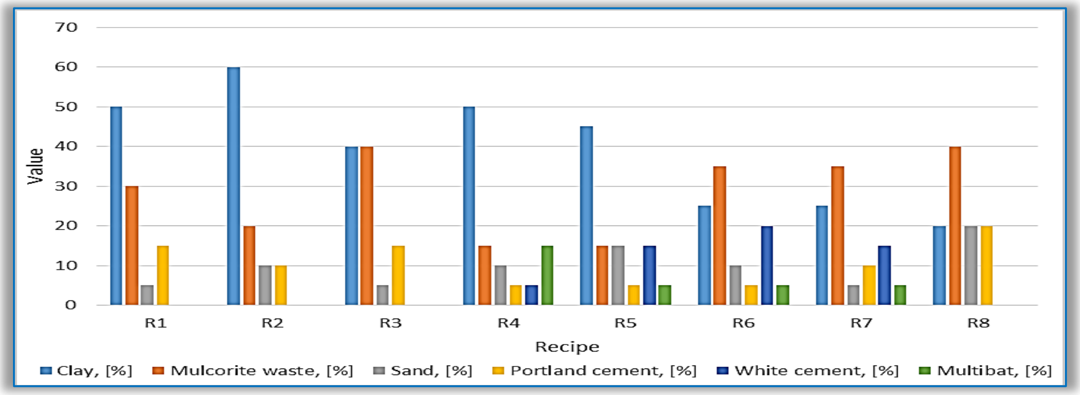

Figure 6. Variation of the mineral components used in the experimental recipes

It can be observed that the batch recipes are diversified; they are made based on the experience of the economic agent SC CCPPR SA Alba Iulia, where the experimental researches were carried out.

3. RESULTS

The values of the main quality characteristics of the obtained refractory products

For all the experimental cubes, three for each of the 8 recipes, the main characteristics were determined through physical–chemical analyses.

Determining the physical characteristics of the resulting products, the values recorded in Table 8 were obtained. For each recipe in this table, the average of the determinations made for the 3 cubes, dried differently, is presented.

Analyzing the results, it is considered that the R6 recipe is the most suitable for the production of the cast iron casting assembly from SC SATURN SA Alba Iulia.

Table 8. The main quality characteristics of the finished products [1]

Recipe	Absorption, (%)	Density, (g/cm^3)	Porosity, (%)
R1	13.90	1.66	26.40
R2	14.80	1.62	29.20
R3	13.75	1.71	28.60
R4	12.55	1.72	24.50
R5	11.30	1.95	18.30
R6	12.75	1.83	21.70
R7	13.00	1.75	19.15
R8	12.95	1.80	18.30

Based on this recipe, SC CCPPR SA Alba Iulia executes the cast iron casting networks entirely at SC SATURN SA Alba Iulia.

Experimental investigations by scanning electron microscopy of the structure of the main raw materials and the finished products made

The experimental researches were carried out with the help of a scanning electron microscope Philips XL 30 ESEM TMP, located in the equipment of UPB–CEMS and UPB ECOMET.

The results of the compositional analyzes were obtained using an energy dispersive spectrometer, EDAX – Saphire, at acceleration voltages of 30 kV, the spot size of the electron beam equal to 5.5, the distance between the polar piece and the sample surface of 10mm, at an angle of 35° formed between the surface of the sample and the X–ray detector. The compositional analyzes were obtained at 100x magnification, on five fields, the results presented in this report being the average of the individual quantitative results.

When interpreting the data, it must be taken into account that microanalysis is a method of compositional characterization of microvolumes, in order to determine the chemical composition of inhomogeneous volumes of substance, it is necessary to perform atomic or mass spectrometry determinations.

All samples were analyzed at magnification powers of 50X, 200X, 500X and 2000X.

SEM electron micrographs – backscattered electron images, of the mulcorite samples and the finished product, are shown in Figure 7 and Figure 8. The EDAX ratios of the mulcorite sample and the refractory ceramic block component of the iron casting network are shown in Figure 9 and Figure 10. In general, the studied ceramic materials present a heterogeneous microstructure, which contains several solid phases and, above all, closed pores. In the images showing the raw materials used, the presence of aluminum and silicon oxides, given by the peaks of the elements Al, Si, O, in the X–ray emission spectra corresponding to the analysis of the respective powders, can be noted. The granulation of the raw materials is relatively homogeneous without liquid phases, because the analyzed raw materials did not undergo prior melting of the solution.

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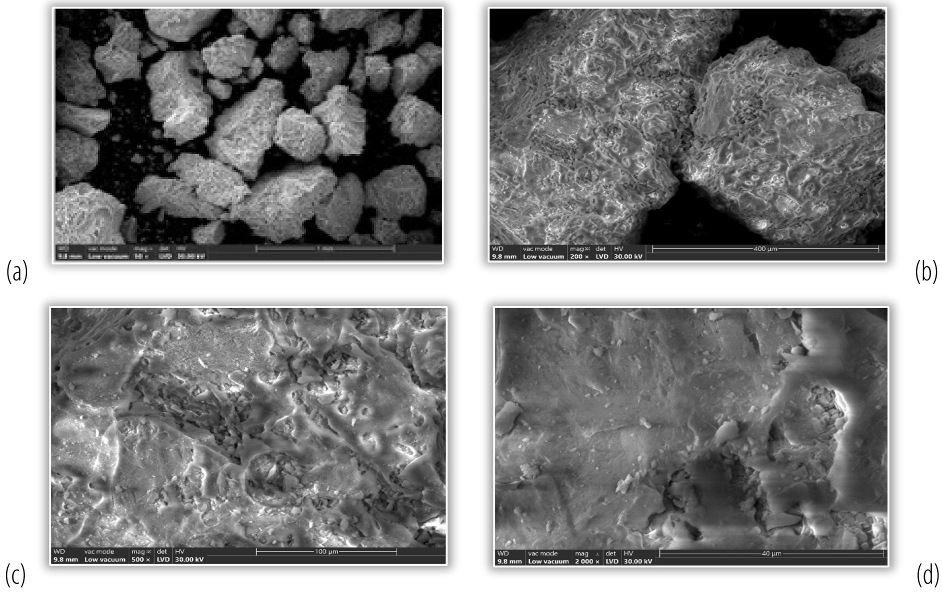


Figure 7. The morphology of the mulcorite powder, at different magnifications: (a) 50X; (b) 200X; (c) 500X; (d) 2000X.

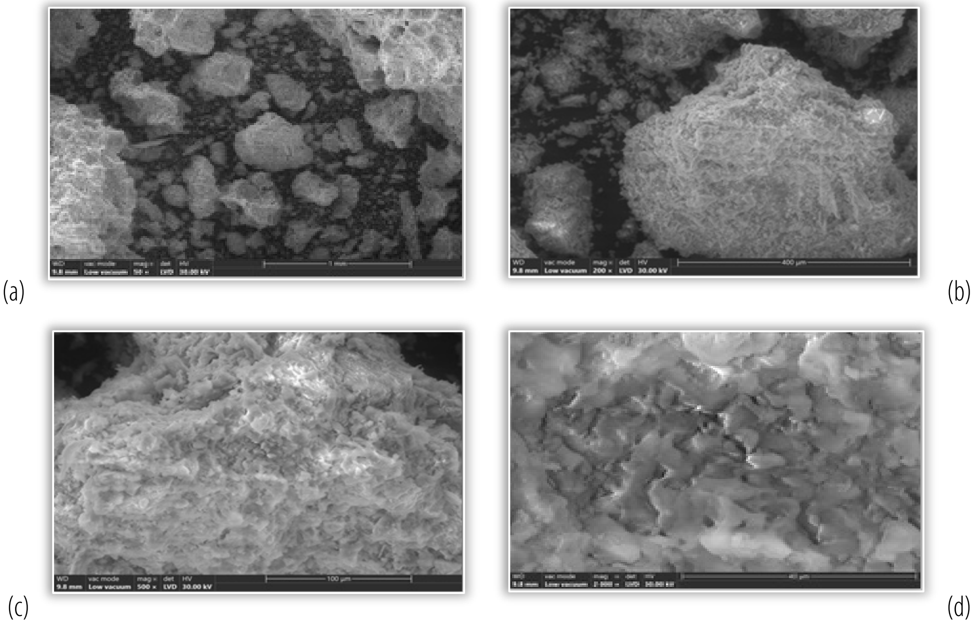
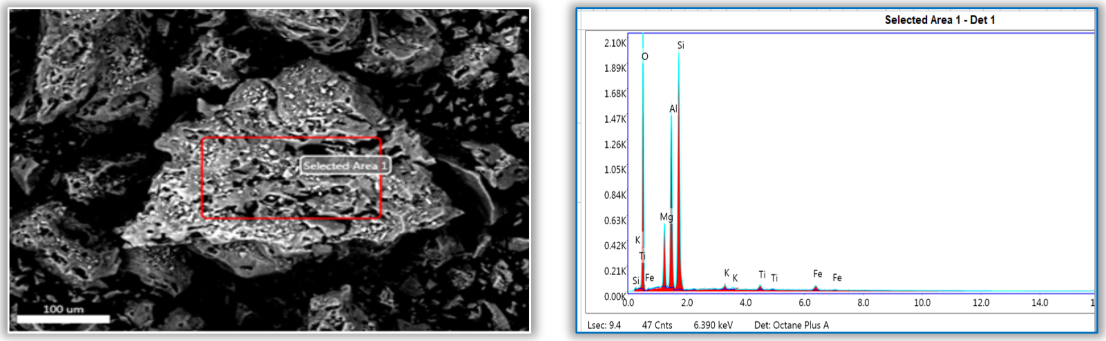


Figure 8. The morphology of the ceramic block powder, component of the casting network, at different magnifications: (a) 50X; (b) 200X; (c) 500X; (d) 2000X.



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z
O K	52.05	65.09	1341.20	8.54	0.1663	1.0406
MgK	6.89	5.67	429.04	8.78	0.0273	0.9723
AlK	16.66	12.35	1251.59	7.09	0.0747	0.9383
SiK	22.76	16.21	1685.41	7.14	0.0973	0.9608
K K	0.37	0.19	33.50	31.73	0.0027	0.8983
TiK	0.52	0.22	45.76	22.15	0.0042	0.8330
FeK	0.75	0.27	53.08	18.18	0.0067	0.8256

Figure 9. EDAX report for the mulcorite sample

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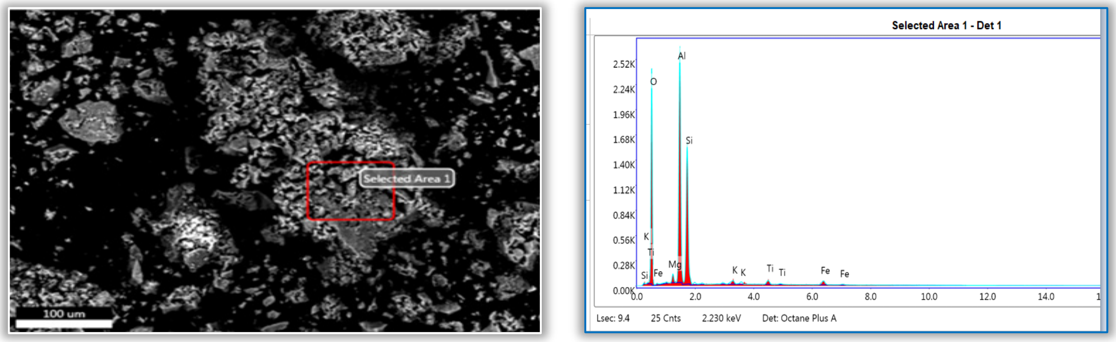

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z
O K	53.87	67.02	1524.65	8.47	0.1742	1.0418
MgK	1.39	1.14	91.91	12.21	0.0054	0.9734
AlK	24.36	17.97	2241.12	6.16	0.1233	0.9394
SiK	18.45	13.08	1392.97	7.74	0.0741	0.9619
K K	0.43	0.22	42.33	22.22	0.0032	0.8993
TiK	0.62	0.26	59.46	19.98	0.0050	0.8339
FeK	0.88	0.31	67.91	17.28	0.0079	0.8265

Figure 10. EDAX report of the sample from the refractory ceramic block component of the cast iron casting grid

5. CONCLUSIONS

The use of secondary raw materials and recycled refractories for refractories production has been a subject of experimental research. The mulcorite waste resulting from the current production of an important economic agent was used as raw material for the production of finished products, namely parts made of refractory materials that make up the cast iron casting assembly for an economic agent producing cast iron parts.

The batch recipes, with the use of waste mulcorite as a raw material for the creation of porous ceramic blocks, were diversified, they being made based on the experience of the economic agent SC CCPPR SA Alba Iulia, where the experimental researches were carried out.

Following an experimental research using the matrix experiment 2^3, 8 variations of the percentages of mulcorite waste were realized.

According to the standards for defining the quality of ceramic and refractory materials, the analyzed quality indicators are the absorption capacity of the finished product measured in %, the density of the finished product measured in g/cm^3 and the porosity of the finished product measured in %.

It has been found that mulcorite waste can be successfully used in the recipes for obtaining refractory materials, required in the market. The maximum percentage of mulcorite that can be used in recipes is 40%, the R6 experimental recipe containing this percentage of mulcorite along with 5% clay, 10% fireclay, 35% slag (fired clay), 5% colloidal silica and 5% Portland cement.

The finished products obtained had good values of the main quality characteristics tested, considering their destination, namely 12.75% absorption coefficient, $1.5g/cm^3$ density and 20.5% porosity.

The micrographs performed showed that the size of the granules depends on the initial composition of the mixture of raw materials, on the size of the granules of the initial materials and on their granulometric distribution. The experimental samples have a uniform and relatively coarse microstructure. The microstructure of the studied ceramic materials was influenced by the addition of Portland cement, it was highlighted that the presence of this additive favors the more abundant development of the liquid phase, as well as a greater growth of crystals.

The results of experimental research demonstrated the possibility of efficient recycling of mulcorite waste by using this waste in the manufacturing recipes of some benchmarks produced at SC CCPPR SA Alba Iulia, such as ceramic blocks that are components of the cast iron casting network.

The recycling of this waste has beneficial implications on the manufacturing costs of refractory products and on the protection of the environment.

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