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EXPERIMENTAL RESEARCH ON THE RECYCLING OF FINE–GRANULATED REFRACTORY WASTE IN THE PRODUCTION OF HEAT–RESISTANT CERAMIC BLOCKS

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Abstract: As a result of the activity carried out in the field of production of ceramic and refractory materials, important quantities of silico–aluminous waste with fine granulation are generated. To comply with environmental legislation, recycling of this waste is mandatory. In this paper, the authors present the experimental research carried out in order to recycle these wastes, by using as raw materials for the production of heat–resistant ceramic blocks. In this research, the authors studied different types of waste, likely to be used as raw materials in the manufacture of heat–resistant ceramic blocks. The manufacturing recipes included different proportions of refractory waste collected from two economic agents producing refractory materials. The authors tested several manufacturing recipes that resulted in obtaining ceramic blocks resistant to high temperatures. The authors presented the results of their own laboratory research for the determination the physico–chemical properties of the obtained products. Comparative analyses of the manufacturing recipes and characteristics of the heat–resistant ceramic blocks obtained; we established the optimal recipe for their production. Based on this optimal recipe, a pilot batch of ceramic blocks was obtained, whose behavior in operation will be followed in the beneficiaries.
Keywords: refractory materials, ceramic materials, industrial waste, recycling, solid pollutants

1. INTRODUCTION

Scientific research has as very important objectives in the current stage of development to find technological, efficient, economic and ecological solutions.

Humanity cannot afford to neglect the environmental effects of sustainable development.

Economic agents must produce quality products, but which are no longer accompanied by hard–to–use by–products or landfilled waste.

However, the concept of zero waste is far from being real. It is mandatory to find technologies that make better use of own production waste. For this, it is necessary to identify all types of industrial wastes, to determine their characteristics and to establish valorization solutions.

These principles are also valid in the industry producing ceramic and refractory materials. On the territory of Romania there are still and it is essential to have economic agents in this field.

A good knowledge of their activity, the identification of pollution sources, the utilization of dusty waste and the application of minimally polluting technologies will allow the preservation of the objects of the companies' activity, the preservation of jobs and their sustainable development.

Refractory products, regardless of whether they are shaped products (with precise shapes and sizes) or unshaped products, in bulk (mortars, concretes, molding materials, casting materials, refractory putties, etc.) are composed of two basic components: refractory aggregate (degreaser), main component that generally represents over 70% of the material composition and forms the structure of the product and the binder that binds the aggregate into a monolith. [1–5]

Refractory products manufactured in the country at this moment with clay binders reach a maximum of 90–92% Al_2O_3, the remaining 8–10% representing impurities of the refractory product by foreign ions that come especially from the clay binder introduced in the refractory mass. [2–5]. Refractory products are natural or artificial refractory products, whose refractoriness is at least 1500oC, (150 IP) [1–4].

Refractory products, in general, can be classified into manufactured, semi–manufactured, unshaped products, and these are classified taking into account different criteria, such as: chemical composition, refractoriness, shaping method, proportion of binder material – degreaser, their shape. Also, according to appearance, refractory products are classified into shaped products and granular or powdery products. [1–4]

According to the standards in force, refractory products are classified according to: refractoriness, chemical and mineralogical composition, porosity, shape, bonding method and manufacturing method. [1,4]

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The classification of refractory products according to refractoriness is presented in Table 1. The classification of refractory products by chemical composition is presented in Table 2.

Table 1. Classification of refractory products by refractoriness [1]

Type of products	Refractoriness		
	Pyroscopic indicator	Seger Con	Temperature, [oC]
Products with normal refractoriness	158 – 175	26 – 34	1580 – 1750
Products with high refractoriness	177 – 200	35 – 42	1770 – 2000
Superrefractory products	> 200	> 42	> 2000

Table 2. Classification of refractory products according to chemical composition [1]

Name	Symbol	Main component	The content		Raw materials and manufacturing method
			Min.	Max.	
Siliceous	S	SiO2	93	–	Masses of quartz rocks (quartzite, quartz, etc.) ground, agglomerated with lime or other binder, shaped and then burned.
Silica– alumina	SA	Al2O3 SiO2	25	75	Mixture of silico–aluminous raw materials (refractory clays, fireclay, bauxite, calcined alumina, corundum, quartzite, etc.)
Corundum	A	Al2O3	75	98	Corundum or electro corundum agglomerated with a binder of refractory clay, kaolin, chemical binder, shaped then fired
Magnesitic	M	MgO			Masses of natural and artificial magnesium raw materials burnt, crushed and granulated, shaped by agglomeration with binders, hydraulically pressed and dried without burning
Spinelic		MgO Al2O3			Masses of mixtures of magnesite with bauxite or with calcined alumina, fired, ground, agglomerated with a binder, dried and then fired
Chromite		Cr2O3	30		Chromite masses, with a quantity of additions (magnesite, bauxite), bound with a binder, shaped, dried, fired
Chromo– magnesitic		Cr2O3 MgO	10	30	Masses of chromite and magnesite, fired, bound with a binder, shaped, dried, fired
Carborundic		SiC	50	95	Silicon carbide (sometimes mixed with fireclay) bonded with a binder, shaped, dried, fired
Specials Carbonic	C	C	90	–	Carbon–containing masses of low–ash, granulated metallurgical coke

Silico–aluminous refractory products are the most widely used refractory products. From the point of view of apparent porosity, refractory products can be superdense, dense and light. Dense products are products that have an apparent porosity below 30%, and light shaped ones (thermal insulators) have an apparent porosity above 30%. The classification of refractory products according to porosity is presented in Table 3 and by shape, in Table 4.

Table 3. Classification of refractory products according to porosity [1]

Classes and subclasses	Apparent porosity, [%]
Superdense class, with subclasses super dense 1 superdense 2 superdense 3	0 – 16 0 – 3 3 – 10 10 – 16
Dense class, with subclasses dense 1 dense 2	16 – 30 16 – 22 22 – 30
Light Class, with subclasses easy 1 easy 2 easy 3	upwards of 30 30 – 45 45 – 75 upwards of 75

Table 4. Classification of refractory products by shape [1]

Type of refractory materials	Domains of use
Shaped refractory products – Refractory bricks – Refractory blocks – Complex refractory products – Special refractory products (crucibles, retorts, tubes, plugs, holes, etc.)	Masonry and linings for thermal aggregates
Unshaped refractory products – Refractory mortar – Refractory concrete – Refractory masses – Formation mixtures and other products	– Realization of refractory masonry – Lining of thermal aggregates – Refractory linings, hot repairs – Molds for foundries.

The classification of refractory products according to the bonding method is presented in Table 5 and in Table 6 the classification of refractory products according to the manufacturing method.

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Table 5. Classification of refractory products according to the bonding method [1]

Binding mode	The technique of realization
Fired (ceramic bond)	Heat treated to sintering temperature
Burnt and impregnated with organic binders	Heat treated up to the sintering temperature and impregnated with organic binders after firing
Burnt and glazed	Thermally treated up to the sintering temperature and the application of a protective layer of glaze
Chemically bonded	Bound with inorganic chemical binders (cements, aluminum chloride, phosphates
Bound with organic binders	Bound with organic binders (tar, pitch, plastics)

Table 6. Classification of refractory products according to the manufacturing method [1]

The type of refractory products according to the main technological characteristics	The technique of realization
Shaped products	
– plastic shaped;	– molded from plastic materials by pressing or extrusion;
– semi–dry or dry beans;	– made from semi–dried or dry masses by pressing, vibration, compaction;
– cast from slush;	– obtained by casting in molds
– foamy;	– obtained by pouring into molds, with the prior addition of sparkling wine
– with additions of combustible materials;	– with the addition of combustible substances
– hot pressed;	–pressing the refractory mass heated beforehand or during pressing;
– taxed and armed;	– pressed in metal boxes with metal fittings;
– glazed;	– applying a protective layer of glaze after shaping;
– melted and cast;	– obtained by melting the refractory mass and pouring it into molds
Powder products	obtained by granulation from natural or synthetic refractory materials

Currently, there are numerous companies that manufacture ceramic blocks, but from the documentation carried out, there is none that includes refractory brick waste in the manufacturing recipe.

The main objective of the research was refractory brick waste, which in a small proportion is reused in the technological flows of the refractory sector, but a large part can be utilized in the construction materials sector.

At the same time, from the refractory materials used in the construction of the furnaces and thermal aggregates of melting and which come into contact with the respective melt (metal, slag, glass, etc.), waste heavily impurity with slag and traces of melts is obtained. [6–9] .

This waste can only be reused in the refractory industry after an arduous cleaning process, a process that raises its cost price to almost the value of new raw material.

Under these conditions, at steel plants, glass factories, etc., large amounts of such wastes are collected that do not find their use. [10,11–14]

By grinding them, a uniform composition is achieved, obtaining a base of raw materials for the products used in the construction materials industry [3,15–19].

Following the study carried out on the construction materials used today, the idea emerged that those based on hydraulic binder are widely spread, considering a number of advantages that the products obtained from this binder have:

- wide spectrum of the field of applicability;
- an increase in productivity, as a result of the fact that construction elements can be produced on an industrial scale outside the place of construction;
- the good machinability of the mass based on hydraulic binder, allows the creation of shapes with a high degree of complexity and with a superior surface finish, giving it a special aesthetic appearance;
- their physico–chemical properties place them on a higher level, in a hierarchical scale of quality and quality function, compared to other construction materials;
- low cost prices compared to other materials used in the construction industry, taking into account the wide area of spread on the surface of the earth of the raw materials used;
- 6) the possibility of using waste from different sectors of activity, with the effect of eliminating the aspect of storing this waste and at the same time replacing natural raw materials, thus protecting the ecosystem. [20–22].

2. MATERIALS AND EQUIPMENT

Materials

Starting from the main groups of raw materials that contribute to the manufacture of a ceramic block, binder, aggregate, additives or fluidizers, or chosen for tests:

1. Binding raw materials:

- ceramic binder raw materials, clay and kaolin;

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— inorganic binder raw materials, sodium silicate, colloidal silica, aluminum chloride;
— hydraulic binder raw materials, plaster, aluminous cement, Portland cement, which are shown in Figure 1.

2. Degreasing raw materials (aggregate), refractory waste, calcined alumina, fireclay, slag, diatomite, perlite, vermiculite, which are shown in Figure 2.

These 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, K_2O. The results are presented in Table 7.

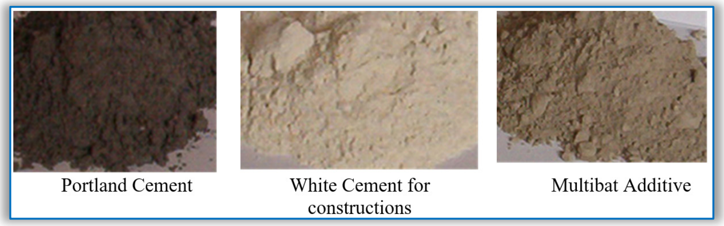

Figure 1. Hydraulic binder raw materials used in the manufacture of ceramic blocks [1-3]

Figure 2. Degreasing raw materials used in the manufacture of ceramic blocks [1-3]

Table 7. Chemical compositions of raw materials

Raw material	Chemical composition, [%]						
	SiO2	Al2O3	Fe2O3	CaO	MgO	Na2O	K2O
Slag	60.56	24.72	1.76	1.30	1.20	1.00	1.00
Waste 28A	24.00	59.45	0.65	0.80	4.50	0.25	0.35
Sand (Colloidal Silica)	87.50	–	–	–	–	3.70	8.80
Portland Cement	22.50	6.50	2.50	63.40	4.50	0.25	0.35
White Cement	25.75	6.75	0.75	64.55	1.50	0.40	0.30
Multibat	24.50	3.50	2.00	65.50	4.00	0.25	0.25

Based on the experience of the manufacturer of ceramic and refractory materials, as well as the information from the specialized literature, eight different samples were created for the experimental research, the composition of which is presented in Table 8.

Table 8. Manufacturing recipes for ceramic blocks

Experimental recipe	Slag, [%]	Waste 28A, [%]	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 graphical variations of the raw material components in the experimental recipes are shown in Figure 3.

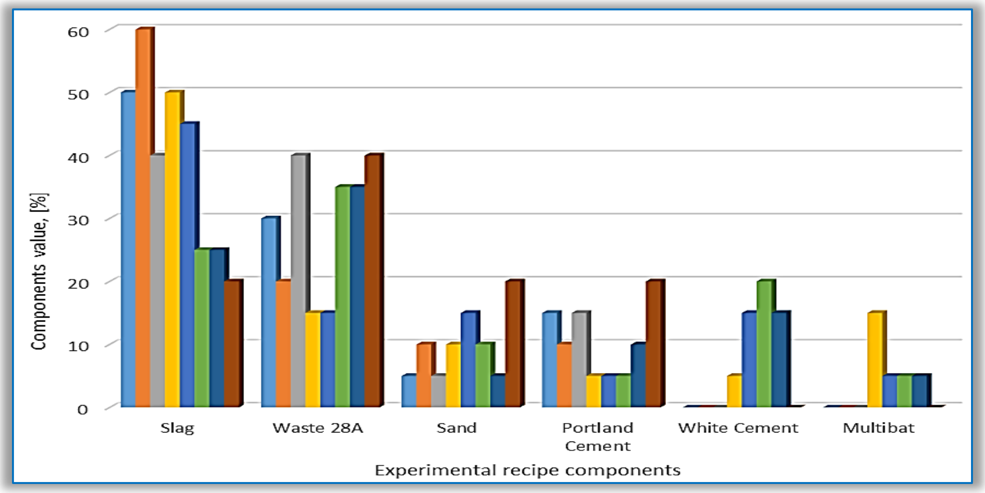

Figure 3. The graphical variations of the raw material components in the experimental recipes

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The experimental equipment used for experimental research of the characteristics of the raw materials used to make heat–resistant ceramic blocks

The testing of the raw materials was done in the Laboratories of SC CCPPR SA Alba Iulia using special equipment for laboratory chemical analyzes and the determination of the physical properties of the raw materials [1–3].

Laboratory equipment for tests performed on raw materials and finished products.

Raw materials and finished products can be tested according to a number of valid standards.

The laboratory equipment for testing raw materials allowed the following investigations to be carried out:

- chemical analysis/ X–ray fluorescence spectrometry (calibration – with certified reference materials, with internal standards), presented in Figure 4;
- determining the loss on calcination (1000oC);
- particle size distribution – the residue on the sieve (wet; dry);
- granulometric analysis on the site (wet; dry);
- determination of humidity;
- determining the content of solid substance;
- determination of pH (10% solid substance);
- plasticity (Pfefferkorn);
- resistance to breaking in the dry state (at 100oC);
- determination of flow properties (Lehmann viscometer): optimal addition of electrolyte, flow time (volume of 100ml), formation speed (30 min);
- contraction (drying, burning, total);
- refractoriness;
- compactness characteristics (fired samples): water absorption, apparent porosity, apparent density; the actual density.

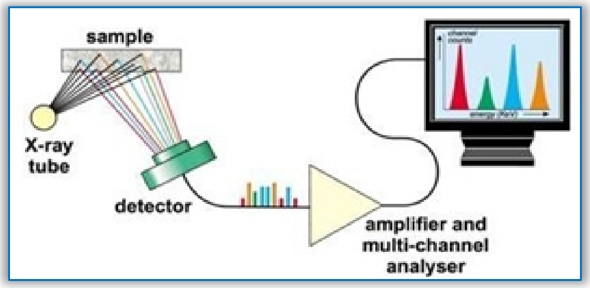

Figure 4. Scheme of apparatus for chemical analysis / X-ray fluorescence spectrometry [1,3]

Laboratory equipment for tests on the finished product consists of equipment that can determine:

- 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.

3. RESULTS

Because the technology of production of heat–resistant ceramic blocks requires a preparation of raw materials such as natural slag, refractory waste, identical to that to produce refractory bricks according to classical technology all equipment in that stream exists within S.C. CCPPR S.A. Alba Iulia [1,5,9].

The location of the equipment does not undergo any change compared to the existing one at present, the flow of raw materials being the one indicated in Figure 5.

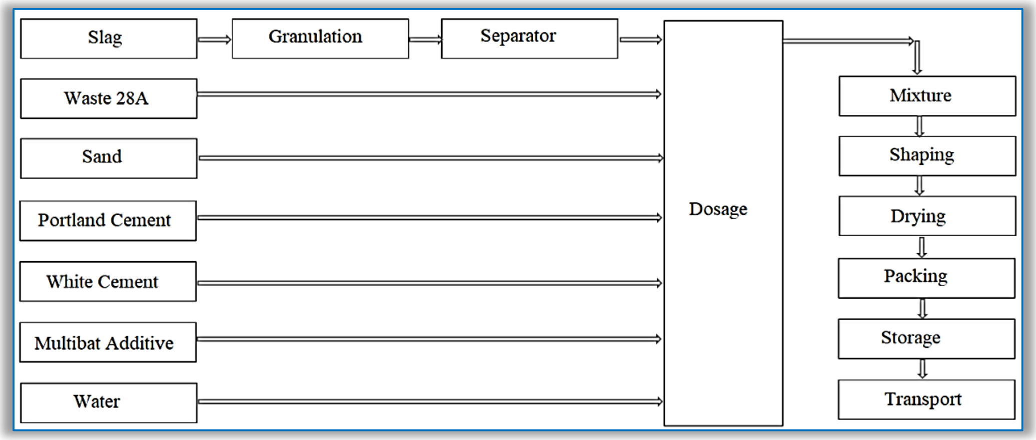

Figure 5. Technological flow to produce heat–resistant ceramic blocks using refractory waste [2,3,5].

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The calcination losses, grain sizes, density and moisture were determined. The results of the analyzes are shown in Table 7. From the raw materials analyzed from a physico–chemical point of view, recipes were established according to Table 8. 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 gypsum was tried in the first phase in a larger proportion.

Wetting the mixture was done at a humidity of 10–15%. The obtained mixture was pressed in the press with friction of 1.2MPa, in 4 stages, following the most advanced disaggregation of the mass. 100x100mm cubes were obtained. The marked cubes were dried, naturally 7–14 days. For all tested recipes, the characteristics of the finished product were determined by physico–chemical analysis. In Table 9 you can follow the results obtained.

Table 9. Results of determining the physical properties of recipes 1-8

Recipe	Absorption, [%]	Density, [g/cm3]	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

Analyzing the results, the recipe R1 for the manufacture of ceramic blocks was chosen.

Presentation of technological operations for obtaining ceramic blocks

Depending on the nature of the raw material, the preparation of the raw material includes:

— Preparation of raw materials

Depending on the desired fraction, grinding takes place in different machines. For the fine fraction (0.2–0.5mm) the slag is introduced with the help of a conveyor belt to the Kollergang. The initial size varies between 20–30mm. After grinding, the slag is lifted with the bucket elevator to the upper height of the hoppers, it is sifted through the vibrating screen and then directed to the intended hopper. For the coarse fraction (1–3mm), the slag is transported to the plate conveyor, to the roller crusher, from where the fine is sent with the bucket elevator to the Kollergang, after separation by sieving, and the course to the storage bunker.

— Dosage of raw materials

Dosing is the operation by which the quantities of material per product (manufacturing recipe) are determined. For the components of the previously established ceramic block recipe, a gravimetric dosage was chosen using the dosing scale. For slag and sand, a volumetric dosing with a dosing scale was chosen, since such high precision is not needed, while for cement, taking into account the fact that it also has a constant humidity, a gravimetric dosing was chosen using dispenser with four doses. In the case of refractory waste, since in the first phase it could be purchased in gravimetric fractions, only the storage bunkers are needed, from where dosing is also done through the dosing scale.

— The mixture of raw materials

The dosed material is picked up by a winch barge and taken to the Eyrich mixer. The materials are fed through the upper part by means of a funnel and the water or wetting solution is dosed from a tank, whose taps are automatically operated by a cylinder–piston assembly. Inside the vat, thanks to the mixing stars, particularly good mixing is achieved. After the mixing operation, which lasts about 10min through the discharge door the prepared mixture is discharged into a buffer hopper.

— Shaping of ceramic blocks

From the buffer hopper, the mixture is taken over by the bucket, which feeds the press with friction of 1.2MPa. The pressing mixture, with a humidity of 5–10% well homogenized, is placed in metal molds. The specific pressing pressure is used to compress the mixture until the required compaction; for overcoming the frictional forces between the mixture and the mold walls; for overcoming the frictional forces between the particles of the mixture. The dimensions to which the products were pressed were 240mm x 115mm x 63mm.

— Drying the products

The ceramic blocks made according to the previously recorded dimensions are subjected to a natural drying in the open air in dry places at an average temperature of 180–200oC, with the aim of strengthening the mechanical properties, taking into account the fact that the hydraulic binders still work overtime.

— Packaging and storage of products

Following the model of the other construction materials, the ceramic blocks are placed on EURO pallets, stacked and tied with metal bands.

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

In the continuation of this work, the results of experimental research carried out with the help of a scanning electron microscope Philips XL 30 ESEM TMP, located in the equipment of UPB–CEMS, are presented.

In advanced vacuum, using an electron beam acceleration voltage of 30kV, a spot size on the sample surface equal to 3, and a distance between the microscope polar piece and the sample surface of 10mm, secondary electron images were obtained (morpho), through which the morphological characterization of all samples was possible, at magnifications between 100X and 25000X.

The results of the compositional analyzes were obtained using an energy dispersive spectrometer, EDAX – Saphire, at acceleration voltages of 30kV, 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 25X, 100X, 500X and 2000X.SEM electron micrographs – backscattered electron images – for the six experimental samples made are shown in Figures 6–11. The figures at point f) show the characteristic X–ray emission spectra of some identified phases, as well as the respective local elemental analysis [14].

In the images showing the raw materials used, the presence of aluminum and silicon oxides is noted, and in the EDAX report for the sample taken from the porous plug, the obvious peak of the Al element is observed due to the majority presence of Al2O3 in the composition of the recipes.

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.

A compaction of the granules due to the pressing of the raw materials on the technological flow is observed in all the experimental samples made.

The size of the granules depends on the initial composition of the mixture of raw materials, the size of the granules of the initial materials and their granulometric distribution. The grain size is larger in ceramic materials that have a higher glass phase content.

In all cases, the experimental samples have a uniform and relatively coarse microstructure.

Each additive used, both alone and in combination, causes an increase in the density of the sintered samples.

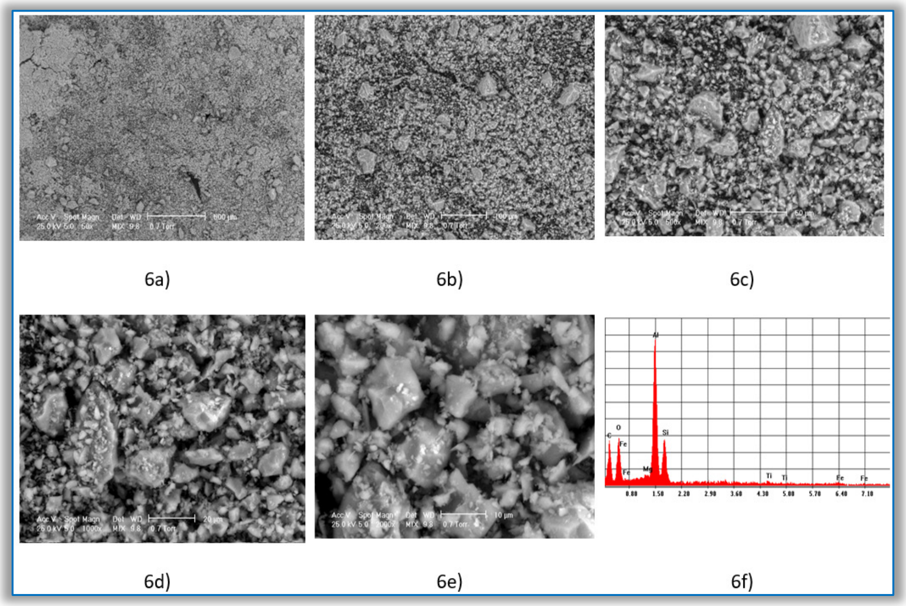

Figure 6. The morphology of the powder of the experimental sample R1, at different magnifications (a, b, c, d, e) and the X–ray emission spectrum corresponding to the analysis of the powder of the experimental sample R1 (f)

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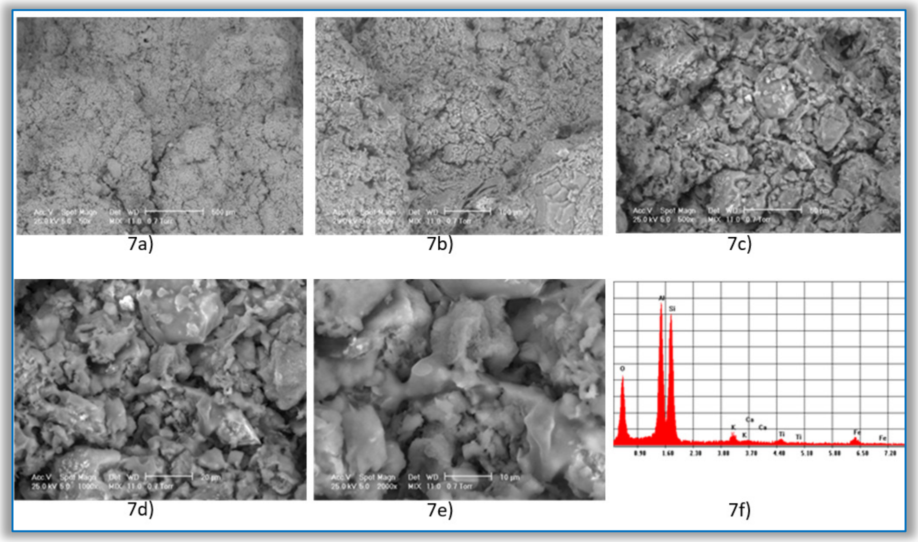

Figure 7. The morphology of the powder of the experimental sample R2, at different magnifications (a, b, c, d, e) and the X–ray emission spectrum corresponding to the analysis of the powder of the experimental sample R2. (f)

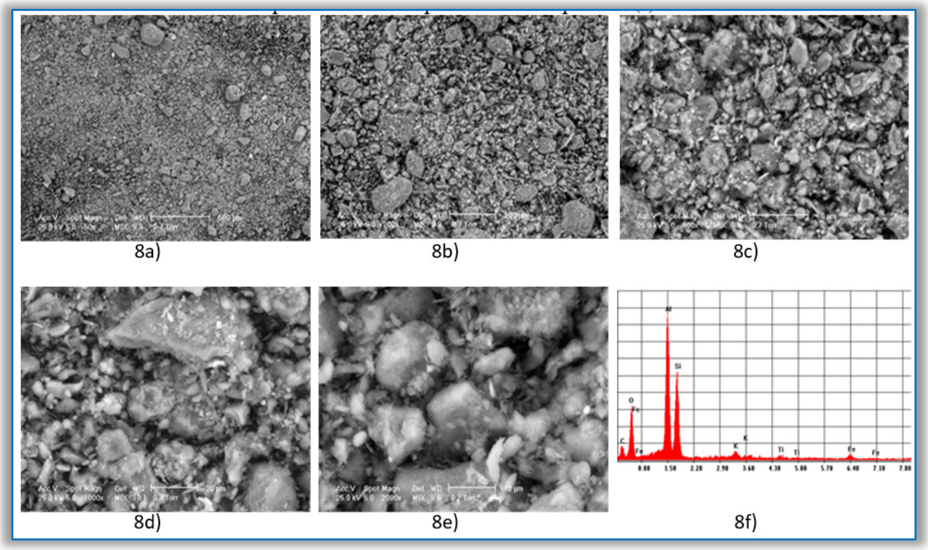

Figure 8. The morphology of the powder of the experimental sample R3, at different magnifications (a, b, c, d, e) and the X–ray emission spectrum corresponding to the analysis of the powder of the experimental sample R3. (f)

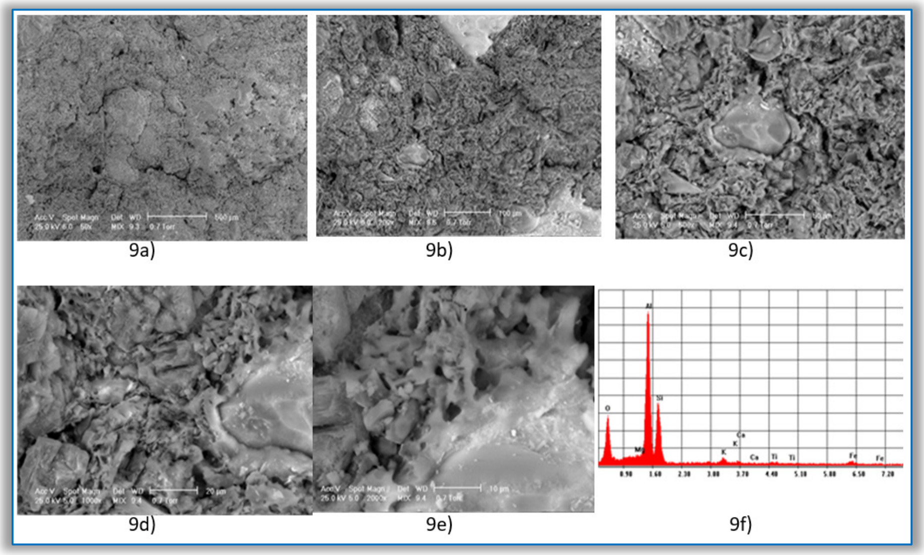

Figure 9. Morphology of the powder of the experimental sample R4, at different magnifications (a, b, c, d, e) and X–ray emission spectrum corresponding to the analysis of the powder of the experimental sample R4. (f)

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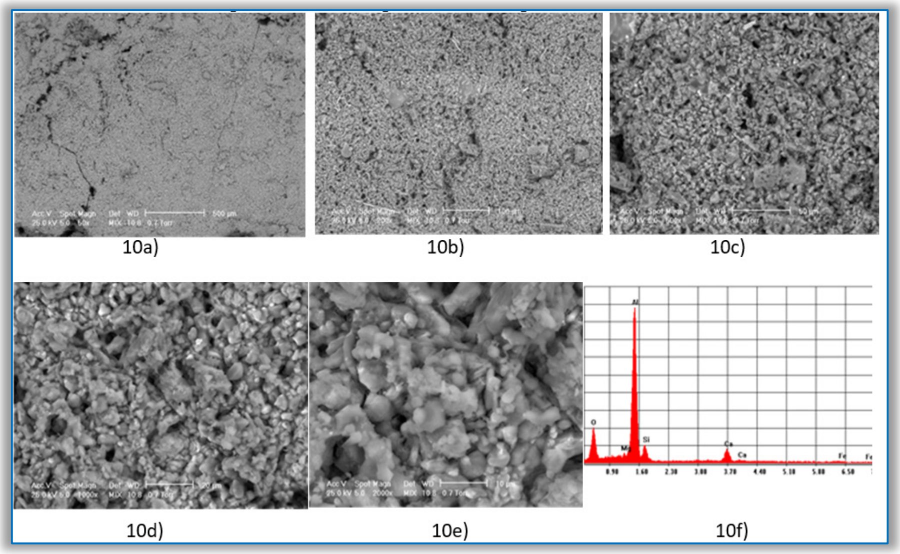

Figure 10. The morphology of the powder of the experimental sample R5, at different magnifications (a, b, c, d, e) and the X–ray emission spectrum corresponding to the analysis of the powder of the experimental sample R5. (f)

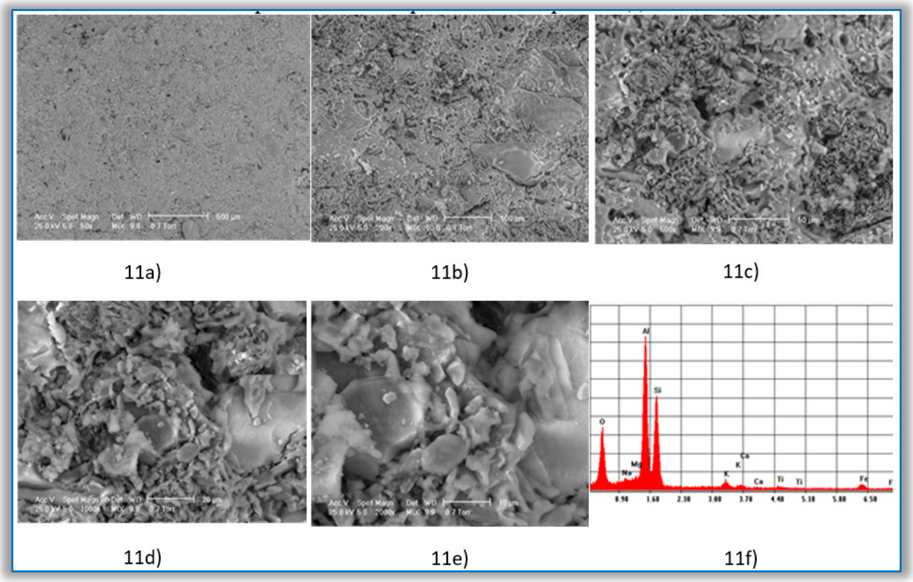

Figure 11. The morphology of the powder of the experimental sample R6, at different magnifications (a, b, c, d, e) and X–ray emission spectrum corresponding to the analysis of the powder of the experimental sample R6. (f)

5. CONCLUSIONS

The manufacture of ceramic blocks as a basic hydraulic binder requires the use of cement, as the strength of the product after hardening is better.

Regarding the use of slag, its good workability, accompanied by its low–cost price, recommends the use of up to 50% of the mass.

Particular attention must be paid to the granulometry of the slag because a too large proportion of fine, determines a decrease of the mechanical resistances, a deformation of the nuclei.

In the case of using refractory waste, the use of fractions that give a structural skeleton to the ceramic mass is considered. The use of refractory waste also aims at the fact that it is a burned product, and its absorption is very low, reducing the absorption capacity of the ceramic product.

The immediate effect is an increase in the freeze–thaw cycles of the final ceramic product, a very important goal to be achieved for a ceramic block used in construction.

The use of refractory waste involves another aspect, which is related to environmental protection, both by eliminating a more advanced exploitation of slag quarries and by eliminating waste that does not find a rational reuse in the economic circuit. This waste can no longer be reused in the refractory field due to impurities (slag) remaining from the furnaces or steel equipment to which it was built. Their storage at ground level or in pits is not possible, due to the sterile nature of the waste, with effect on the plant and

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animal kingdom. The manufacture of ceramic blocks with a basic hydraulic binder requires the use of cement, as the strength of the product after curing is better. The environmental effects of refractory waste recycling are obvious and extremely important.

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