PHYSICO-MECANICAL AND STRUCTURAL CHARACTERIZATION OF SOME SOUND-ABSORBING OXIDE MATERIALS MADE OF DIFFERENT SOLID WASTES

CARACTERIZAREA FIZICO-MECANICĂ ŞI STRUCTURALĂ A UNOR MATERIALE OXIDICE FONOABSORBANTE OBŢINUTE DIN DIVERSE DEŞEURI SOLIDE

MIHAI BRATU[1], OVIDIU DUMITRESCU[2*], OVIDIU VASILE[3], LUOANA FLORENTINA PASCU[1], CAROL LEHR[1]

[1] National Research & Development Institute for Industrial Ecology – ECOIND Bucharest, 71-73 Drumul Podul Dâmboviţei Str., 060652, Bucharest, Romania,

[2] University POLITEHNICA of Bucharest, Faculty of Applied Chemistry and Materials Science, Department of Oxide Materials Science & Engineering and Nanomaterials, 1-7 Gh. Polizu Str., 011061, Bucharest, Romania,

[3] University POLITEHNICA of Bucharest, Faculty of Biotechnical Systems Engineering, Department of Mechanical Engineering, 313 Independentei Spl., 060042, Bucharest, Romania

Pollution, in all its current views, is a permanent concern of the researchers. Therefore, making sound-absorbing materials that include waste is a very useful approach.

The paper deals with two environment related issues: noise pollution due to different sources in industry and transport, as well as waste storage, also affecting the quality of the environment.

The paper presents the researches on obtaining some oxide materials containing various solid wastes that may affect the environment. The schema of obtaining process and the actual composition of these materials are described.

Some physico-mechanical properties have been measured to characterize these oxide materials: particle size distribution of starting materials, compressive strength, apparent density and apparent porosity and sound absorbing properties.

The microscopy analyses (SEM and EDAX) have been used to study the morphology, homogeneities and existing pores could be highlighted in order to explain the obtained physico-mechanical properties, especially those of sound absorption.

The values of the measured properties allow for the determination of the best oxide materials obtained from solid wastes in terms of sound absorption.

Poluarea, sub toate aspectele ei actuale, reprezintă o preocupare permanentă şi actuală a cercetătorilor. De aceea, realizarea de materiale fono-absorbante care să înglobeze deşeuri este o abordare foarte utilă.

Lucrarea atinge două aspecte legate de mediu şi anume: poluarea fonică datorată diferitelor surse din industrie şi transporturi, dar şi depozitarea deşeurilor care afectează calitatea mediului înconjurător.

Lucrarea prezintă cercetări privind obţinerea unor materiale oxidice ce conţin diverse deşeuri solide. Este descris schematic procedeul de obţinere, precum şi compoziţiile efective ale acestor materiale.

Au fost măsurate o serie de proprietăţi fizico-mecanice pentru caracterizarea acestor materiale: distribuţia granulometrică a materialelor de start, rezistenţă mecanică la compresiune, densitatea aparentă şi porozitatea aparentă, precum şi capacitatea de absorbţie a undelor sonore.

Analizele de microscopie (SEM şi EDAX) au fost utilizate pentru a studia morfologia, neomogenitaţile şi porii existenţi, cu scopul de a explica proprietăţile fizico-mecanice obţinute, în special cele de fonoabsorbanţă.

Valorile proprietăţilor măsurate permit stabilirea celor mai bune materiale oxidice obţinute din deşeuri din punct de vedere al absorbţiei undelor sonore.

Keywords: *solid wastes, physico-mechanical and microstructural properties, sound absorption coefficient*

1. Introduction

This work touches on two aspects of the environment: noise pollution from various sources in industry and transport, as well as waste storage, both affecting the quality of the environment. Thus, due to the economic development, some concerns were raised in the field of environmental protection in general, and in particular, the reduction of the level of noise pollution.

Noise is a risk factor that has harmful effects on the human body, which can lead to: disturbances of the auditory organ; disorders of various organs and systems of the body; reducing labor productivity; lesser capacity to understand speech [1-6].

Thus, the rapid evolution of materials science and engineering in the last decade requires the production of new, high performance materials with functional and aesthetic qualities capable of reducing the level of noise pollution. Obtaining some sustainable, economical and ecological materials is a top priority in the field. Issues related to the ecosystem are permanent problems of the

* Autor corespondent/*Corresponding author*,
E-mail: ovidiu.dumitrescu@upb.ro

century, therefore the necessity for applying sustainable development, defined as follows: "The sustainable development is the development that aims to meet the needs of the present, without compromising the possibilities of future generations to meet their own needs."

The idea of the paper was to obtain sound-absorbing oxide materials based only on solid waste whose storage has a negative impact upon the environment [7-10]. The paper is a research on the physico-mechanical characterization of these oxide materials with sound absorbing properties obtained from various solid wastes. Through a series of laboratory tests, an optimal technological process has been achieved for obtaining various oxide material samples. The samples were tested for mechanical compressive strength, apparent density and apparent porosity, sound absorption coefficient. Based on microstructural analyses (SEM and EDAX), the physico-mechanical and sound absorbing properties are confirmed. Microstructural analyses (SEM and EDAX) confirmed the evolution of physico-mechanical and sound absorbing properties, depending on the waste used.

2. Experiments

The raw materials used are: fly ash, thermal power plant slag, steel works slag, sterilized waste garbage and ash resulted from burning seed shells.

Fly ash and thermal power plant slag are the result of coal combustion in the boilers of thermal power plants. The sterilized waste garbage is a mixture of household waste from the Făgăraş municipal warehouse. This waste was used in the laboratory after sterilization at 120^{0}C made at SGE SMART GEO ENERGY SRL. The steel works slag is a by-product of the process electrically melting metallic wastes with graphite electrodes; following the melting process, above the metal bath slag separates, which is then cooled and stored in waste dumps. The burned seed ash results from burning seed shells in the combustion boilers at the oil making factories.

The chemical composition of the used solid wastes was determined using X-ray Spectrofluorimetry (ED-XRF) type Rigaku CG (Rigaku, Japan) equipped with a 50W X-ray source with a Pd anode and secondary targets of Al, Mo, Cu and RX9 (HOPG polarizer). Detection was performed using a silicon drift detector (SSD) maintained at optimal temperature with Peltier system.

The chemical composition of these wastes (mean values % weight) is shown in Table 1.

Due to the storage and transport conditions, the raw materials described above are dry. Also, the steel works slag and the thermal power plant slag have been subjected to milling operations.

The solid wastes used for obtaining the samples analyzed in this study were measured from the point of view of the particle size distribution. For coarse fractions, the sieves were used according to SR ISO 2591-1: 2003 and for fine fractions were analyzed using the laser diffraction particle size analyzer MALVERN, MASTERSIZER 2000 (range 0.02÷2000μm) according to ISO 13320-1: 2009 [11,12]. Table 2 presents the particle size distribution for coarse fractions to the following wastes: ash resulted from burning seed shells, sterilized waste garbage, steel works slag and thermal power plant slag.

In Figure 1 are presented the particle size distributions for fine fractions on the following types of wastes: ash resulted from burning seed shells (fig. 1a), fly ash (fig. 1b), sterilized waste garbage (fig.1c), steel works slag (fig.1d) and thermal power plant slag (fig. 1e).

According to the optimized technological process [13,14] shown in the diagram in Figure 2, the oxide materials were made only from solid waste. In the first phase, the wastes used as raw materials are weight dosed at the established proportions.

Table 1

Chemical composition of the utilized solid waste, % weight (mean values)

Compoziţia chimică a deşeurilor solide utilizate, în % gravimetrice (valori medii)

Oxide / *Oxid*	% weight / *% gravimetrice*				
	Fly Ash *Cenuşă de termocentrală*	*Steel works slag* *Zgură de oțelarie*	Sterilized waste garbage *Deșeu steril de gunoi*	Thermal power plant slag *Zgură de termocentrală*	Ash resulting from burning seed shells *Cenușă de la arderea cojilor de semințe*
SiO_2	3.13	18.69	34.05	49.63	9.00
Na_2O	17.79	1.55	10.86	1.76	0.00
K_2O	0.30	0.00	5.67	1.17	23.85
MgO	0.42	4.02	5.21	1.35	17.95
CaO	76.00	38.27	28.33	6.84	35.67
Al_2O_3	1.01	6.92	7.75	29.28	1.65
Fe_2O_3	0.86	25.29	2.77	7.23	0.98
P_2O_5	0.18	0.44	3.87	1.08	10.63
Other oxides *Alți oxizi*	0.31	4.83	1.49	1.65	0.27
Total	100	100	100	100	100

Table 2

Particle size distribution for the coarse fractions of the solid wastes / *Distribuţiile granulometrice pentru fracţiile grosiere ale deşeurilor*

Sieve size / *Dimensiune sită* [mm] / Waste / *Deşeu*	0.25	0.5	2	4
	Percent passing / *Trecerea cumulată* [%]			
Ash from burning seed shells *Cenuşă de la arderea cojilor de semințe*	52.33	89.71	95.38	100
Sterilized waste garbage *Deşeu steril de gunoi*	41.97	79.19	94.87	100
Steel works slag *Zgură de oţelărie*	15.05	57.87	89.98	100
Thermal power plant slag *Zgură de termocentrală*	16.72	73.80	92.49	100

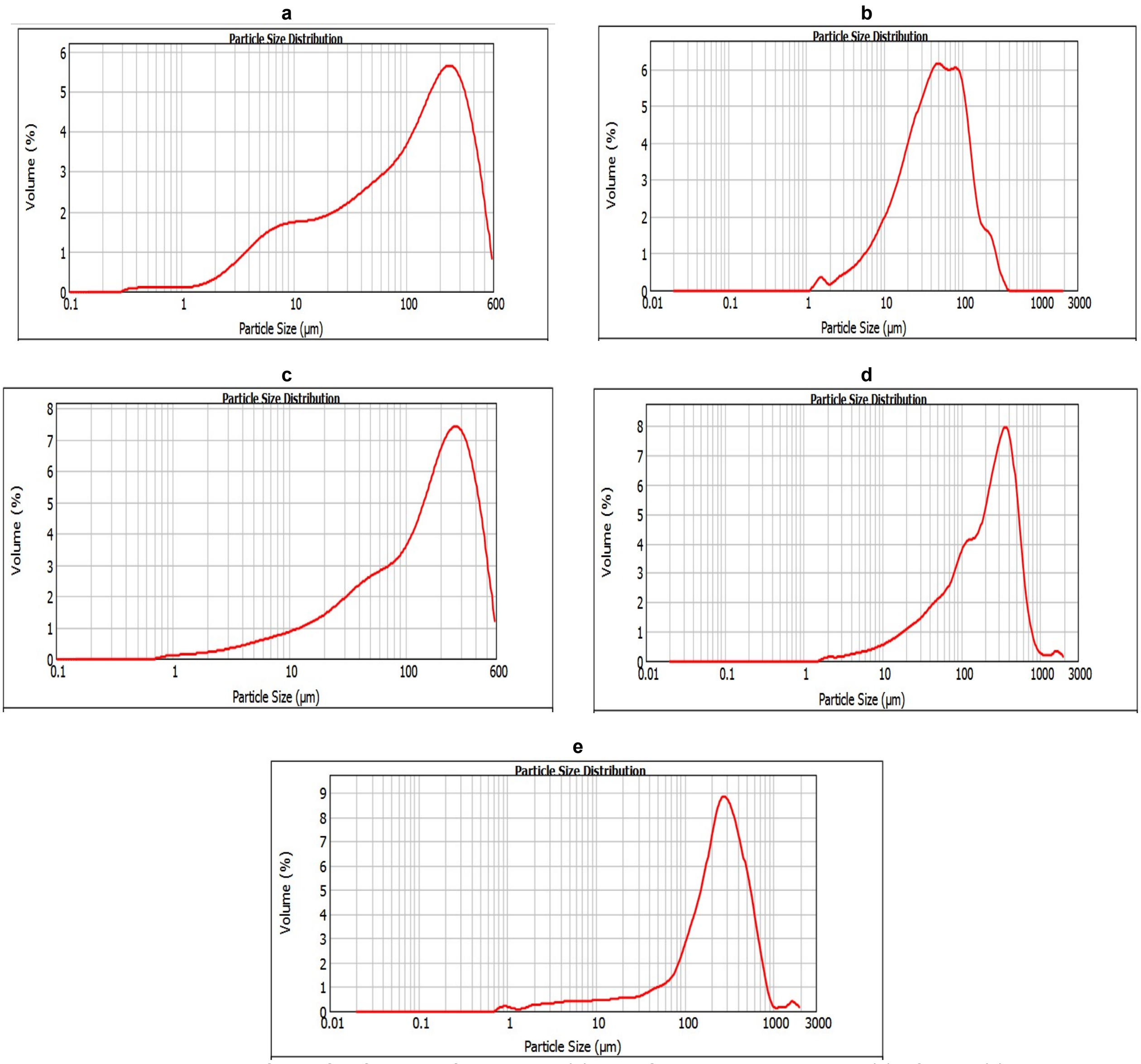

Fig. 1. - Particle size distribution for the fine fractions of the wastes: (a) – ash from burning seed shells; (b) – fly ash; (c) – sterilized waste garbage; (d) – steel works slag; (e) – thermal power plant slag / *Distribuțiile granulometrice pentru fracțiile fine ale deşeurilor: (a) – cenuşă coji; (b) – cenuşă termocentrală; (c) – deşeu steril menajer; (d) – zgură de oţelărie; (e) – zgură de termocentrală.*

The homogenization was carried out in a mechanical shock resistant container - stirrer with cross pallet Heidolph type RZR 2021 - in which the other materials were added together with water. Also, a mixture 2% CaO + $CaCl_2$ [15-18] was added as an activator to achieve the hardening of the obtained oxide materials. In the first stage, the fly ash was mixed with water until a consistency paste was obtained, then the other materials were added, according to the established proportions. Table 3 shows the proportions of solid wastes, of CaO + $CaCl_2$ activator as well as the amount of water used to prepare the oxide materials studied in this paper [19].

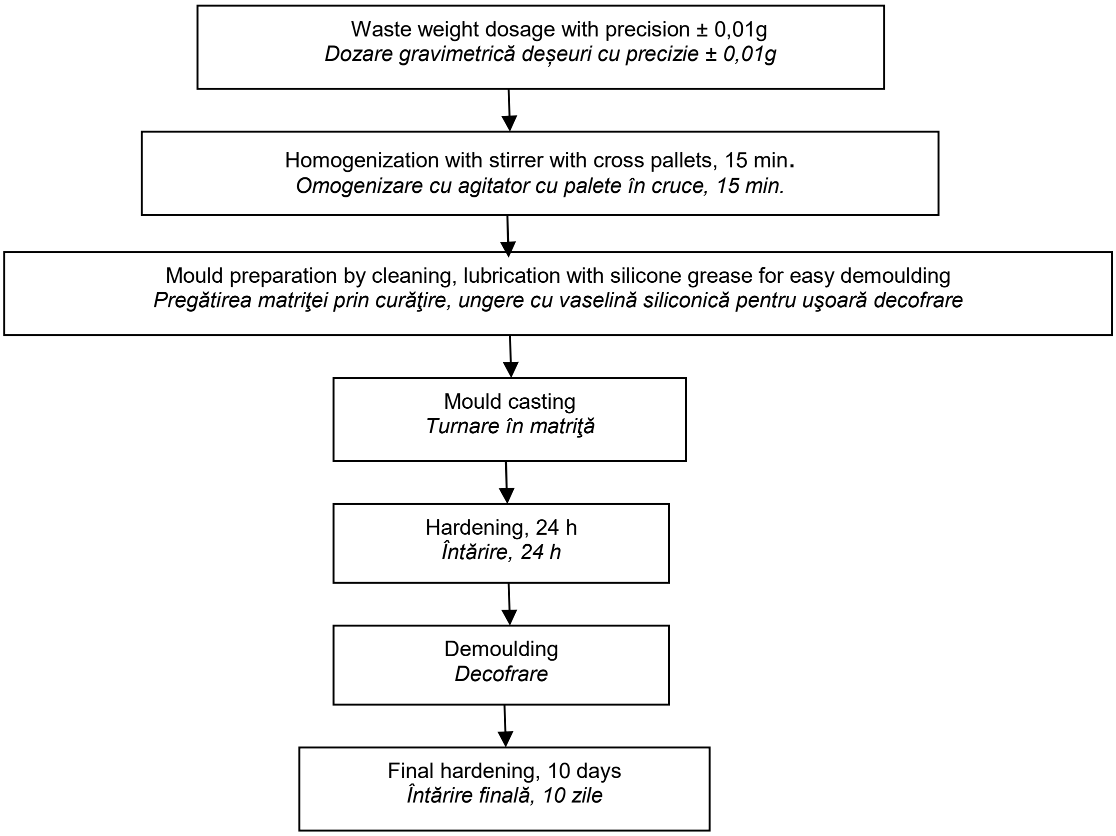

Fig.2 – Schema of the technological process for obtaining the oxide materials / *Schema procedeului tehnologic de obţinere a materialelor oxidice.*

Table 3

The proportion of solid wastes of activator and the amount of water used to prepare the studied oxide materials
Proporţiile de deşeuri solide de activator și cantitatea de apă utilizate pentru prepararea materialelor oxidice studiate

Used materials / *Materiale utilizate*	No. sample / *Nr. probă*				
	P1	P2	P3	P4	P5
Fly Ash / *Cenuşă de termocentrală*	60%	60%	60%	60%	98%
Steel works slag / *Zgură de oțelarie*	38%	---	---	---	---
Sterilized waste garbage / *Deșeu steril de gunoi*	---	38%	---	---	---
Thermal power plant slag / *Zgură de termocentrală*	---	---	38%	---	---
Ash from burning seed shells *Cenușă de la arderea cojilor de semințe*	---	---	---	38%	---
$CaO+CaCl_2$	2%	2%	2%	2%	2%
Solid material: water ratio / *Raport material solid : apă*	1: 0.30	1: 0.33	1: 0.30	1: 0.35	1: 0.30

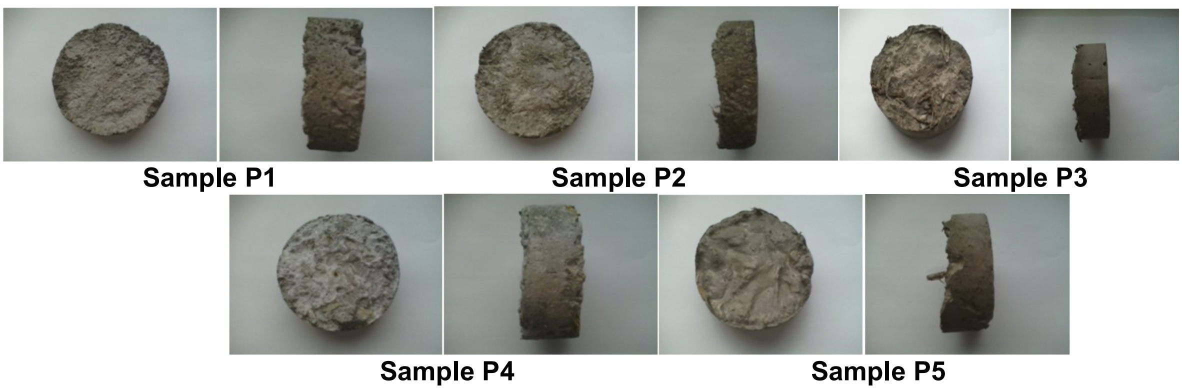

Fig.3 - Images of obtained oxide materials / *Imagini cu materiale oxidice elaborate.*

After homogenizing the mixture, it was cast into the mold that has been pre-prepared to allow easy demoulding. After a 24 hours hardening period, the sample is demoulded and then left for 10 days for final hardening. For each composition six specimens were prepared. Samples were 60 mm in diameter and 20 mm in thickness.

Figure 3 presents images of the five compositions elaborated and studied in this paper, in terms of physical and mechanical characteristics [19].

For characterizing the obtained oxide materials known methods were used, according to the current standards. Thus, the determination of the sound absorption coefficient was achieved with a Kundt acoustic interferometer according to SR EN ISO 10534-1; SR EN ISO 10534-2 [20,21,22].

Also, compressive strength, apparent density and apparent porosity were measured in accordance with the respective standards [23,24].

The compressive strength was performed on a compression testing machine with automatic record force – deformation curve, Tinius Olsen H10KT type, measuring range: 0-10 kN with a control routine. Before testing, the specimens (disc-shaped – see Fig. 3), three for each composition, were kept (conditioned) for 24 hours in an enclosure at (20 ± 2)°C. The compression test complies with EN 826: 2013. The specimens were placed centrally between the two rectangular plates.

From the force-displacement record curves, the values of the maximum breaking forces were selected, and then, depending on the diameter of the samples, the compressive strengths were calculated (in MPa).

For the measurement of apparent density and apparent porosity, the immersion in liquid method was used (according to SR EN 993-1: 1997) [24]. The samples used were 20 x 20 x 10 mm (3 for each composition).

The electronic microscopy images, showing the structure of the obtained oxide materials, were performed using the QUANTA INSPECT F scanning electron microscope (SEM) equipped with field emission gun (FEG) with a resolution until 4 nm and an energy dispersive X-ray spectrometer (EDS).

3. Results and discussions

To characterize the physico-mechanical properties, the obtained oxide materials were analyzed in terms of sound absorption. Figure 4 shows the variance of the determined sound absorption coefficient, α, versus the sound wave frequency variation, between 16 and 3150 Hz.

The P1 sample has a better sound absorption property than the other samples. This achieves a maximum of α = 0.93 at 1650 Hz. Also, the P3 sample shows a sound absorption coefficient α = 0.90 at the 2400 Hz frequency and a good absorption in the high frequency range between 2000-2800 Hz. The P2 sample shows a slightly lower sound absorption than sample 1 and 3 so that on the frequency range between 1200-2000 Hz it reaches a maximum of α = 0.8 to 1600 Hz. Samples P4 and P5 have the lowest sound absorption coefficient.

These determinations show that from the point of view of sound-absorbing property, the best oxide materials obtained are those which, in a fly ash matrix, have embedded steel works slag (P1), thermal power plant slag (P3) or sterilized waste garbage (P2).

Samples 1-3 have high sound-absorbing properties on medium and high frequency ranges, similar to the existing materials on the market, such as the following materials: wood, rubber [25], textile materials and polyurethane [26,27,28].

The samples were then tested for compressive strength as well as apparent density and apparent porosity, and the results of the measurements are shown in Figures 5-7.

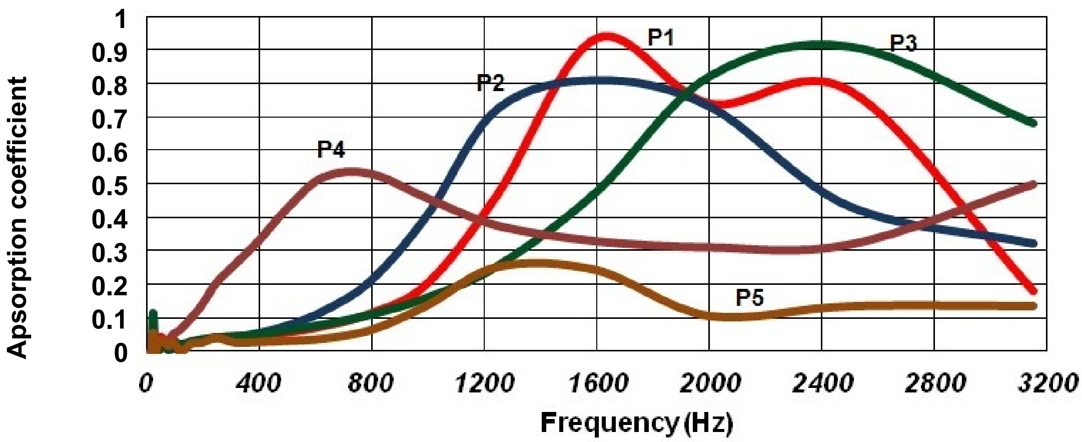

Fig. 4 - Sound absorption coefficient versus the sound wave frequency, for the obtained oxide materials / *Coeficientul de absorbție a undelor sonore în functie de frecvența undelor sonore, pentru materiale oxidice elaborate.*

From the analysis of these results it can be stated that the P1, P3 and P4 oxide materials have lower compressive strength values than the other compositions, which is also explained by the apparent density and apparent porosity. However, these compressive strength values allow the use of these compositions in the future: this will be possible for making sound-absorbing panels, depending on their sound absorption coefficient, by improving them with the appropriate additions that would increase their mechanical resistance.

An interesting variation is shown in Figure 8, namely the correlation between the apparent porosity and the sound absorption coefficient, phenomenon also found in other studies [27,28], namely that as the apparent porosity increases, the sound absorbing properties of the material (on a wider range of the sound frequency) also increase. The shape of the microstructural surface, the density of the material, as well as the percentage of perforations in relation to the total surface, results in the variation of the peak of the central frequency where the sound absorption coefficient has a maximum value, for variable frequency domains.

This evolution confirms that the best oxide materials in terms of sound-absorbing are those that have a high apparent porosity, P1, P3 and P2, because the sound absorption coefficient depends on the apparent porosity value of the oxide material analyzed.

Figures 9-13 show SEM micrographs (at 2 different magnifications) for the obtained oxide materials.

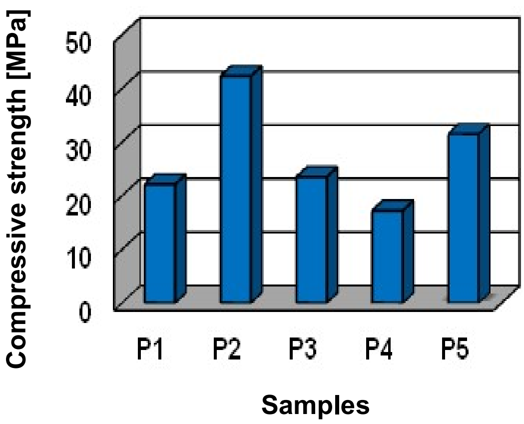

Fig. 5- Compressive strength values for the five obtained oxide materials / *Valorile rezistenţei mecanice la compresiune pentru cele 5 materiale oxidice elaborate.*

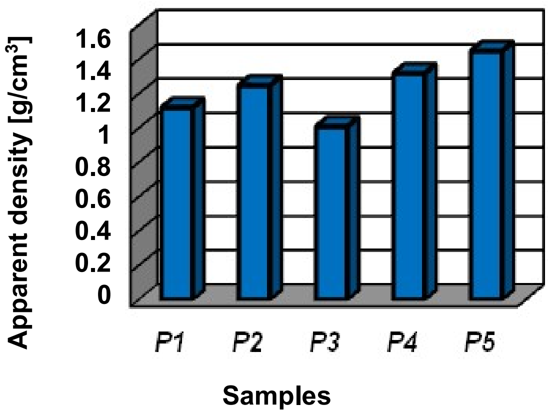

Fig. 6 - Apparent density values for the five obtained oxide materials / *Valorile densităţii aparente pentru cele 5 materiale oxidice elaborate.*

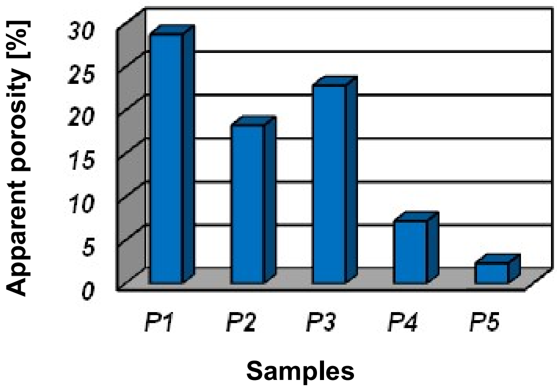

Fig. 7- Apparent porosity values for the five obtained oxide materials / *Valorile de porozitate aparentă pentru cele 5 materiale oxidice elaborate*

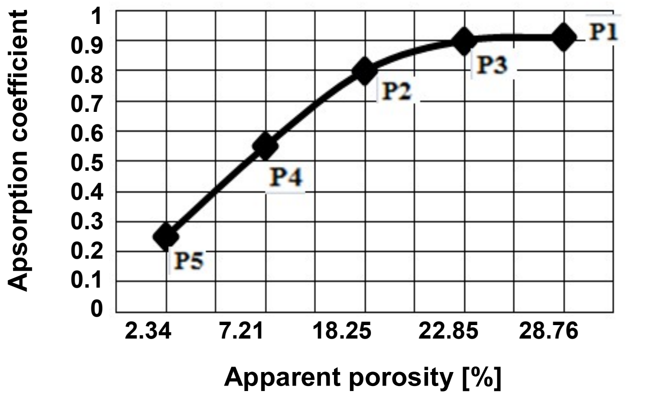

Fig.8 - The variation of the sound absorption coefficient versus the apparent porosity for the 5 obtained oxide materials / *Variaţia coeficientului de absorbţie a undelor sonore versus porozitate aparentă pentru cele 5 materiale oxidice elaborate.*

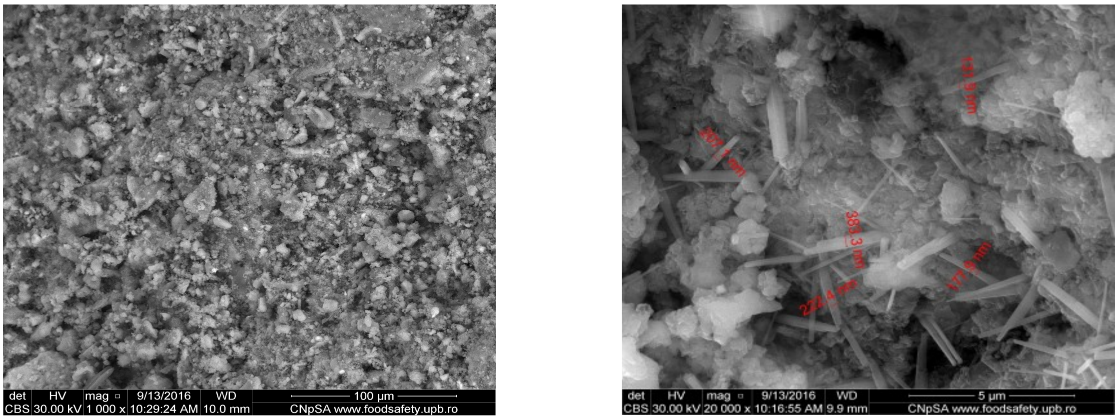

Fig. 9 - SEM micrographs (magnification 1000X and 20000X) for P1 composition / *Inregistrările SEM (mag.1000X și 20000X) pentru compoziţia P1*

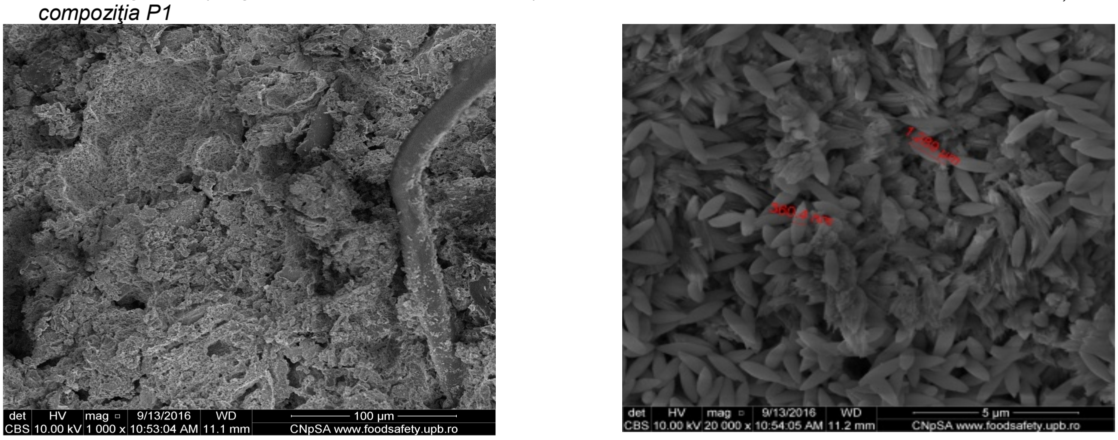

Fig. 10 - SEM micrographs (magnification 1000X and 20000X) for P2 composition / *Inregistrările SEM (mag. 1000X și 20000X) pentru compoziţia P2*

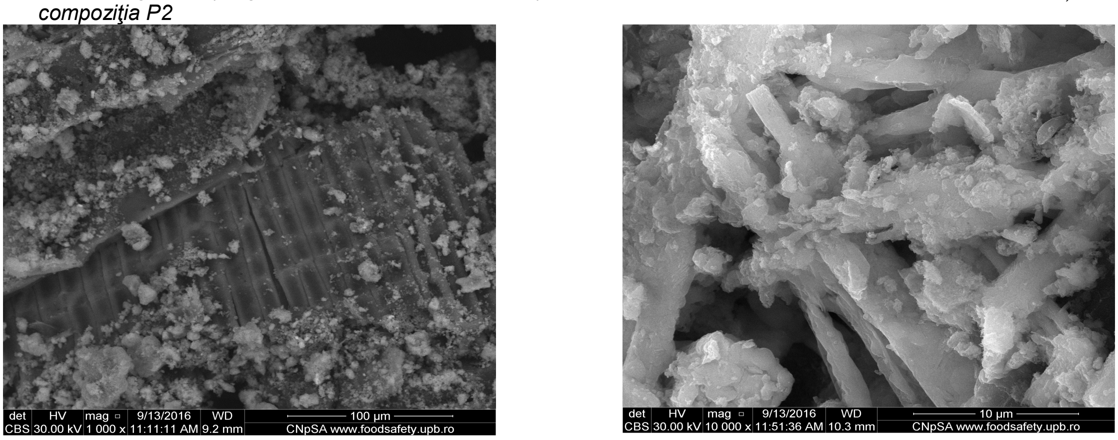

Fig. 11. - SEM micrographs (magnification 1000X and 10000X) for P3 composition / *Înregistrările SEM (mag. 1000X și 10000X) pentru compoziţia P3*

SEM analyzes have been used to study the microstructure and the state of the surface of the obtained materials, especially on breaking surfaces. Thus, the morphology, homogeneities and existing pores could be highlighted in order to explain the obtained physico-mechanical properties, especially those of sound absorption.

The SEM microscopies of the obtained materials highlights in Fig. 9÷13 irregular microstructural shapes, with pores of various sizes and shapes, which do not resemble each other, depending on the characteristic compositions used.

It can be seen that the SEM microscopies for the P1, P2 and P3 compositions shown in Fig. 9, 10 and 11, there is a higher percentage of *open pores* (varied holes / of “black” colour) compared to P4 and P5 (standard). The SEM micrographs for samples P4 and P5 (standard) show a lower pores fraction, greater compaction, and explain the lower sound absorption coefficient values.

For these reasons, the first three compositions are more effective in terms of sound absorption properties, confirming the results shown in Fig. 8, as well as those from other studies [27,28].

Table 4 shows the elemental compositions measured with the EDAX probe on the obtained oxide samples.

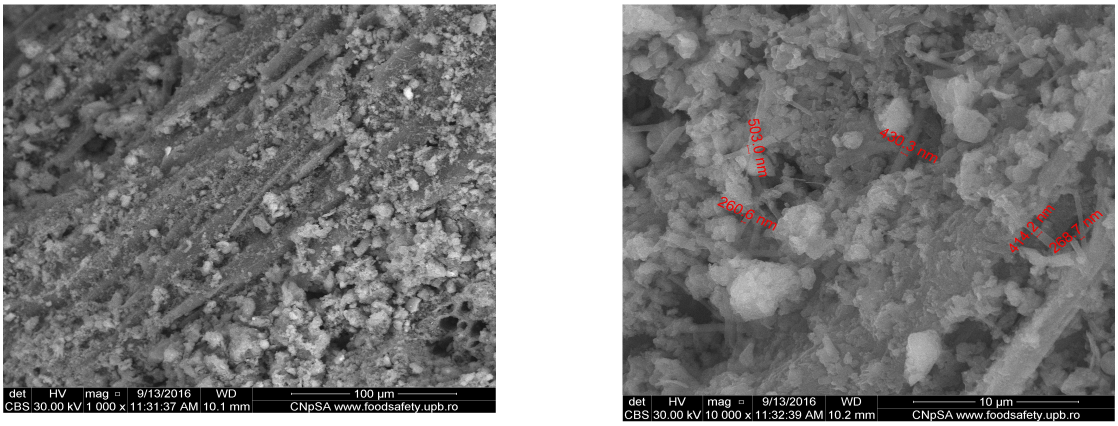

Fig. 12 - SEM micrographs (magnification 1000X and 10000X) for P4 composition / *Inregistrările SEM (mag. 1000X și 10000X) pentru compoziţia P4*

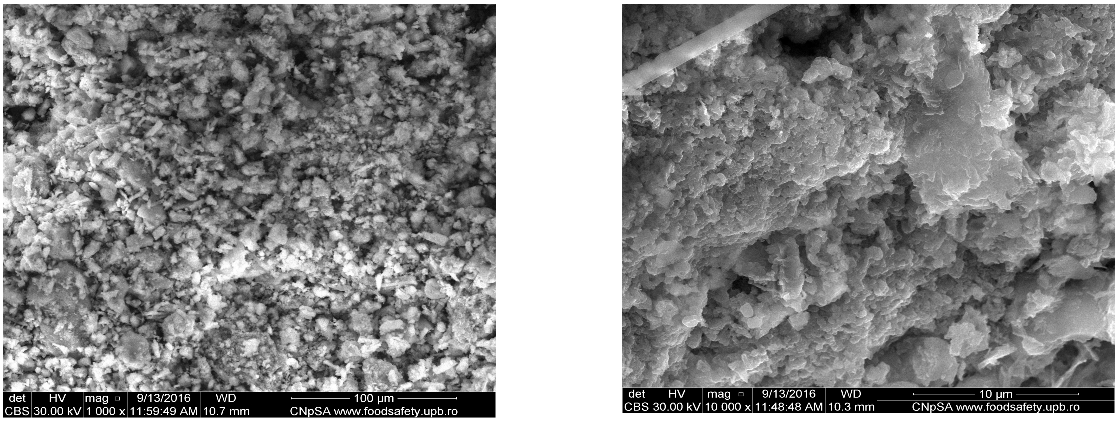

Fig. 13 - SEM micrographs (magnification 1000X and 10000X) for P5 composition */ Inregistrările SEM (mag. 1000X și 10000X) pentru compoziţia P5*

Table 4

EDAX elemental compositions of the obtained oxide materials / *Compoziţiile elementale EDAX ale materialelor oxidice obținute*

	% weight / *% greutate*									
No. sample *Nr. probă*	Si	Al	Mg	Ca	S	Cl	Mn	Fe	C	O
P1	4.11	0.96	0.82	35.51	3.1	0.46	1.63	9.62	0	43.79
P2	1.02	0.56	0	39.97	0.73	1.96	0	0	5.84	49.92
P3	3.37	2.46	0.39	23.87	0.74	3.11	1.47	3.91	12.5	48.18
P4	5.76	4.07	0.77	20.89	1.3	2.44	0.42	3.51	11.1	49.74
P5	3.58	0.44	7.66	24.34	1.58	0.78	2.05	0.49	7.95	51.13

Thus, the analysis of SEM micrographs reveals that the porosity of the samples is consistent with the apparent density and apparent porosity measurements, which indirectly confirms both the compressive strength and the sound absorption values.

The EDAX records showed the elemental compositions of the elaborated samples, which confirmed the relatively uniform distribution of the elements in the final composition of the obtained materials. This has shown that the technological process used for obtaining these materials is correct and optimal.

4.Conclusions

In this research we aimed to obtain oxide materials with sound absorbing properties using only waste based through an optimized technological process.

The main material was the fly ash and the other materials were wastes: thermal power plant slag, steel works slag, sterilized waste garbage, and ash resulted from burning seed shells.

Measurements of physico-mechanical properties have highlighted the values that determine the degree of usability of these materials. The values of the sound absorption coefficient indicated the best performing oxide materials in terms of sound-absorption. Microscopy analyses (SEM and EDAX) have confirmed the physico-mechanical and sound-absorption measured properties and allowed a well-chosen selection of the obtained materials.

We recall that the performance evaluation of sound-absorbing material in noise reduction devices must be performed for each type of material, according to current standards [29].

In conclusion, on the basis of the elaboration and characterization of these materials, it is possible to propose for future research the obtaining of sound-absorbing panels made with waste: the fly ash as base plus one of the wastes: steel works slag, sterilized waste garbage, thermal power plant slag, in this order.

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