INVESTIGAȚII PRIVIND UTILIZAREA HIDROXIDULUI DE ALUMINIU CA MATERIE PRIMĂ LA OBȚINEREA MATERIALELOR CERAMICE
INVESTIGATIONS REGARDING USING OF ALUMINUM HYDROXIDE AS RAW MATERIAL AT THE OBTAINING OF CERAMIC MATERIALS

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In this paper the possibility of obtaining porous ceramics by using of two commercial sorts of aluminum hydroxide with different fineness (from Vimetco Alum SA Tulcea Romania) was investigated. Proposed method for obtaining a such ceramics is based on a very low-pressing (30kN) of powder mixtures, followed by a thermal treatment at 1550°C. The effect of aluminum hydroxide finesses on the apparent porosity and density, compressive strength and thermal expansion coefficient of the ceramics obtained was investigated. Also, the mineralogical and microstructural characteristics were evaluated by XRD and SEM analyses. The results showed that both samples of aluminum hydroxide led to improve of porosity, density and mechanical strengths properties compared to standard ceramics prepared with calcined alumina. The use of the coarser powder (below 45µm) in the proportion of 25% leads to the obtaining of ceramics with physical-mechanical characteristics close to those of ceramics made with finer powder (less than 10µm) in a proportion of 25-50%, with beneficial effect on production costs. The properties of porous ceramics had suggested that they may be used in filtering applications.

În această lucrare a fost investigată posibilitatea obținerii ceramicii poroase prin utilizarea a două sortimente comerciale de hidroxid de aluminiu cu finețe diferită (de la Vimetco Alum SA Tulcea România). Metoda propusă pentru obținerea unei astfel de ceramici se bazează pe o presare foarte scăzută (30kN) a amestecurilor de pulberi, urmată de un tratament termic la 1550°C. A fost investigat efectul fineții hidroxidului de aluminiu asupra porozității și densității aparente, rezistenței la compresiune și coeficientului de dilatare termică ale ceramicii obținute. De asemenea, caracteristicile mineralogice și microstructurale au fost evaluate prin analize XRD și SEM. Rezultatele au arătat că ambele probe de hidroxid de aluminiu au condus la îmbunătățirea proprietăților de porozitate, densitate și rezistență mecanică în comparație cu ceramica standard preparată cu alumină calcinată. Utilizarea pulberii mai grosiere (sub 45µm) în proporție de 25% conduce la obținerea unor ceramici cu caracteristici fizico-mecanice apropiate de cele ale ceramicilor realizate cu pulbere mai fină (sub 10µm) în proporție de 25-50%, cu efect benefic asupra costurilor de producție. Proprietățile ceramicilor poroase au sugerat că pot fi utilizate în aplicații de filtrare.

Keywords: *aluminum hydroxide, porous ceramics, microstructure, physical and mechanical characterization*

1.Introduction

The ceramics occupies an important place within modern materials, due to its physical and chemical properties, such as high resistance at corrosion, thermal resistance, and biological compatibility. From this category, a special role has the materials of aluminum oxide and the composites based on this, due to the highest capacity to keep the structure and properties then when they are exposed at aggressive environments [1- 3]. In the range of technical ceramics, one direction of development is the obtaining of porous ceramic materials. The ceramics with porous structure may be used in order to produce filters, thermal isolations, supports of catalyst, membranes etc. [4-6]. Porous ceramics are, also, interesting for medical applications due of its compatibility with the bony tissues [4, 7].

Porous ceramic products may be classified depending on the following characteristics [8]:

- porosity: moderate porosity (30 – 50%), high porosity (60 – 75%), and super-high porosity (over 75%);
- physical state of products: on pieces, continuous, filling;
- inside structure: granular, cellular, fibrous;
- refractoriness correlated with service temperatures: low melting point (below 1350°C), high melting point (1350 – 1580°C), refractory (1580 – 1770°C), very refractory (1770 – 2000°C), super refractory (over 2000°C);
- range of application: thermo-isolator, thermal screening and permeable.

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In conventional way, porous aluminous ceramics may be manufactured through partial sintering of the powders of Al_2O_3 with very fine granulation [3, 6]. Also, in order to obtain porous ceramics may be used additives, formator of pores at burning, which allow the control of pores volume, but present the inconvenient of the carbon traces on inside surfaces of pores, inacceptable thing for bio-implants manufacturing [2, 9, 10]. In order to form a structure of pores without adding the additives formator of pores, the method of components decomposition may be used, for example, hydroxides in oxides, accompanied by gas release, and, consequently, porosity formation. The use of $Al(OH)_3$ for obtaining of porous ceramics is an usual method [6,10,11]. An aqueous colloidal suspension of $Al(OH)_3$ was used to obtain porous aluminous foams [6]. Also, Levkov R and Kulkov S [10] obtained promising porous ceramics in medical applications such as inorganic bone matrix, by using aluminates solutions as a starting component to obtain aluminum hydroxide with gibbsite modification $Al_2O_3 \cdot 4H_2O$. Another study [11] was approached a combination of Al_2O_3 - $Al(OH)_3$ and Al_2O_3 - $Al(OH)_3$-ZrO_2 for porous Al_2O_3 ceramics that can be widely used as catalyst supports in industry, especially for high-temperature catalysis. Chakravarty et al. [12] had demonstrated the possibility of obtaining aluminous ceramics through sintering in plasma using Al $(OH)_3$ nano-crystalline exclusively as raw material, without additives or dopants. Another way to obtain porous Al_2O_3 ceramics starting from a very fine $Al(OH)_3$ powders (2.5μm) is by cold-press of raw materials at a pressure between 30-200 MPa followed by a thermal treatment up to 1600 ^oC [1,11]. Zkuhov and al. [1] pressed the powders at 200 MPa and found that increasing sintering temperature of the Al_2O_3 ceramics leads to increasing compressive strength from 6 MPa to 800 MPa at the temperature of sintering between 1,300 and 1,500 °C, respectively. Analysis of literature shows that currently there is a lack of systematic studies of porous ceramics obtained by applying a very low-press on the Al_2O_3-$Al(OH)_3$ powder mixtures. To the best of our knowledge, no data were found in the literature for the Al_2O_3 -$Al(OH)_3$ powder mixtures starting from commercial aluminum hydroxide powders with a particle size up to 45 μm. So, our research is focus to obtain porous ceramic using as raw materials two commercial components, namely calcined alumina and hydroxide aluminum powders. The proposed obtaining method is based on the homogenization of powder mixtures, followed by a very low pressing of them in the presence of polyvinyl alcohol solution (as binding agent) and sintering at 1550^{o}C, with a beneficial effect on the production costs due to the simplification of the manufacturing process, while maintaining the specific performance (porosity, coefficient of thermal expansion, compressive strength). The paper presents the preliminary laboratory research regarding the possibility of obtaining porous ceramics by using of aluminum hydroxide powders produced at ALUM S.A. Tulcea. The paper is the result of technical-scientific cooperation between Vimetco Alum SA Tulcea, Romania and CEPROCIM SA Bucharest, Romania.

2.Materials and Methods

2.1.Materials

In order to investigate the possibility of porous ceramics obtaining, the raw materials resulted in the technological installations of ALUM Tulcea were used, as follows: a sample of calcined alumina (AC) and two samples of aluminum hydroxide with different fineness. In the case of aluminum hydroxide, the first sample presented a content of minimum 95% particles below 10μm (AH10), while the second sample shown a content of minimum 95% particles below 45μm (AH45).

2.2. Preparing of ceramic samples

The raw materials, dosed according to the recipes presented in the Table 1, were homogenized in a planetary mill for a period of 60 minutes. The compositions were mixed for binding with 20% solution of polyvinyl alcohol (of 10% concentration) and then granulated on the sieve with mesh screen of 0.8 mm. The pressing was made at a force of 20 kN with hydraulic press Meyer + SCHNEGG A.G, obtaining cylindric test pieces with the diameter of 11 mm and height of 6...8.5 mm. The test pieces were dried in oven at 100^{o}C and then were subject to thermal treatment in an NABERTHERM electric oven, in air atmosphere, at the temperature of 1550^{o}C with keeping of a level of 2 hours. Increasing speed was of 1^{o}C/minute in the interval of temperature of 0-350^{o}C and 4^{o}C/minute in the interval of 350-1550^{o}C.

Table 1

Compositional recipes / Rețete compoziționale

Raw material	Code compositions				
	A0	**A10-1**	**A10-2**	**A45-1**	**A45-2**
AC, %	100	75	50	75	50
AH10, %	0	25	50	-	-
AH45, %	-	-	-	25	50

2.3.Methods of characterization

Granulometric analyze – granulometric distribution of raw materials was performed with laser granulometer MERVEL MASTERSIZER 2000E, according ISO 13320:2009 [13], SR ISO 9276-1:2001 [14].

Mineralogic constituents of raw materials and of sintered samples were identified through X-ray diffraction, by exposure of samples in angular interval 2θ = 10-80 Å, using the diffractometer DRON 3 (CuKα, λ=1.5405Å).

Microstructural analysis of sintered samples, in order to visualize the microstructure and distribution of pores size, was performed by scanning electron microscopy (SEM) with scanning electron microscope QUANTA INSPECT F provided with a gun of electrons with emission in field – FEG (field emission gun) with resolution of 1.2 nm and spectrometer of X-rays, dispersive in energy (EDS) with resolution at MnKα of 133 eV. The samples were covered with gold and were analyzed both in fracture and on surface.

Density and apparent porosity of sintered samples was performed according SR EN 60672-2:2003 [15] based on Archimedes principle (immersion in water). Presented values are average values of 3 measurements.

Apparent density was, in g/cm^3, calculated with the relation:

$$\rho_a = \frac{(m_0 \cdot \rho_{H_2O})}{(m_2 - m_1)} \qquad (1)$$

where:

- ρ_{H2O} is the density of distilled water at working temperature, g/cm^3;
- m_0 is the weight of dried specimen, g;
- m_1 is the weight of the specimen saturated with water, weighted in water, g;
- m_2 is the weight of the specimen saturated with water, weighted in air, g.

Apparent porosity, in %, was calculated with the relation:

$$P_a = \frac{(m_2 - m_0)}{(m_2 - m_1)} \cdot 100 \qquad (2)$$

Compression strength was determined according to ASTM E9-09 [16] at room temperature using a universal press for the testing in static regime of the materials, model LFM 30kN. The loads were applied in the period of compression tests at a transversal speed of 0.20 mm/min until the samples were cracked. All given values are average values of 3 measurements.

Young's modulus and ***yield strength*** were calculated from the compression test according to ASTM E9-09 [16], paragraph 9.2 and 9.3, respectively].

Thermal expansion coefficient was determined according ASTM E831-14 [17], standard method of testing of linear thermal expansion of solid materials through thermo-mechanical analyze.

3.Results and discussion

3.1.Characteristics of the raw materials

Chemical and granulometric characteristics of the calcined alumina and aluminum hydroxide samples are presented in the Table 2.

The two samples of alumina hydroxide used in the preparation of ceramics showed an advanced purity, the content of Al $(OH)_3$ being over 99%.

Average diameter of the samples of aluminum hydroxide highlight that these are very fine, 50% vol. being below 5.472 μm (in the case of AH10) and below 11.214 μm (in the case AH45). Calcined alumina (AC) is coarser, d50 being of 89.265 μm.

Table 2

Physical-chemical characteristics of the raw materials / *Caracteristici fizico-chimice ale materiilor prime*

Composition	M.U	Calcinated alumina (AC)	Alumina hydroxide	
			AH10	AH45
Chemical characteristics				
Al $(OH)_3$	%	-	99.69	99.64
Al_2O_3	%	98.73	65.18	65.15
Fe_2O_3	%	0.011	0.010	0.012
SiO_2	%	0.011	0,009	0.009
Na_2O_T	%	0.30	0.18	0.20
Moisture	%		0.211	0.323
Granulometry				
>150 μm	%	8.64		
<45 μm	%	10.84		99.90
<10 μm	%		72.00	
d10	μm	42.503	1.387	1.797
d50	μm	89.265	5.472	11.214
d90	μm	161,844	14,790	41,026

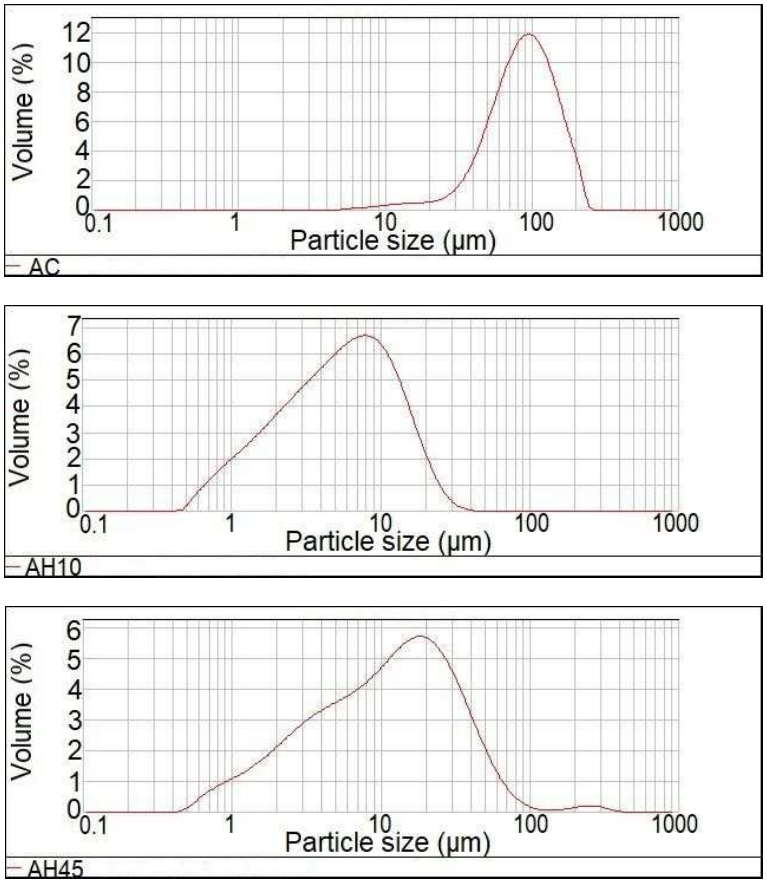

Fig. 1 - Diagrams of size distribution for powders particles of: a – calcined alumina (AC); b – aluminum hydroxide <10 µm (AH10); c – aluminum hydroxide <45 µm (AH45) / *Diagramele dispribuției dimensiunii particulelor: a – alumina calcinată (AC); b – hidroxid de aluminiu < 10 µm (AH10); c – hidroxid de aluminiu < 45 µm (AH45).*

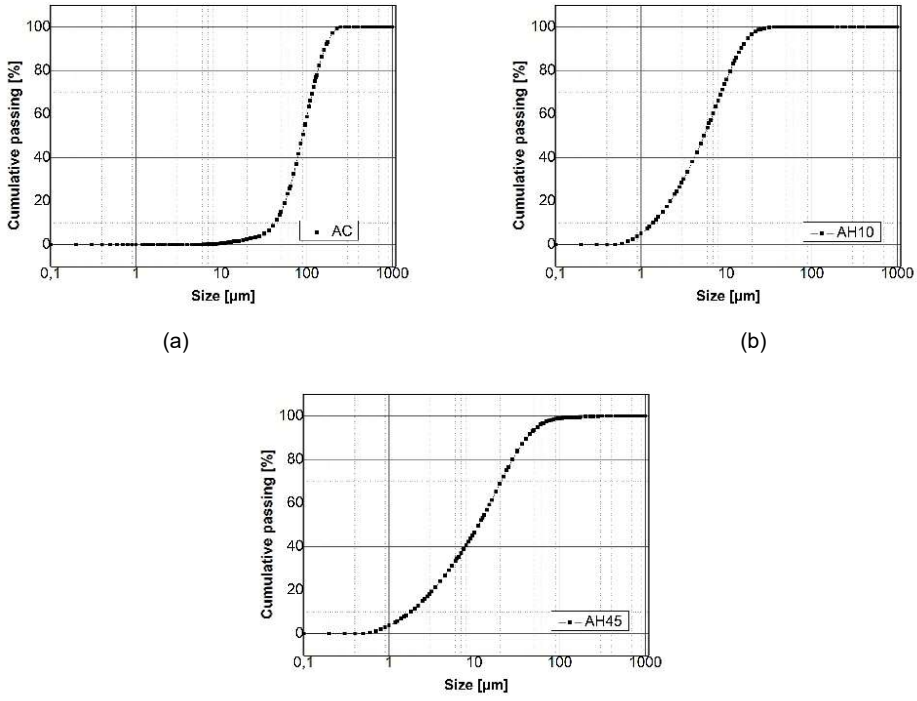

Fig. 2 - Diagrams of cumulative passing on grain size classes of the powders of: a – calcined alumina (AC); b – aluminum hydroxide <10 µm (AH10); c – aluminum hydroxide <45 µm (AH45) / *Diagrame ale trecerii cumulate pe clase granulometrice ale pulberilor: a – alumina calcinată (AC); b – hidroxid de aluminiu < 10 µm (AH10); c – hidroxid de aluminiu < 45 µm (AH45)*

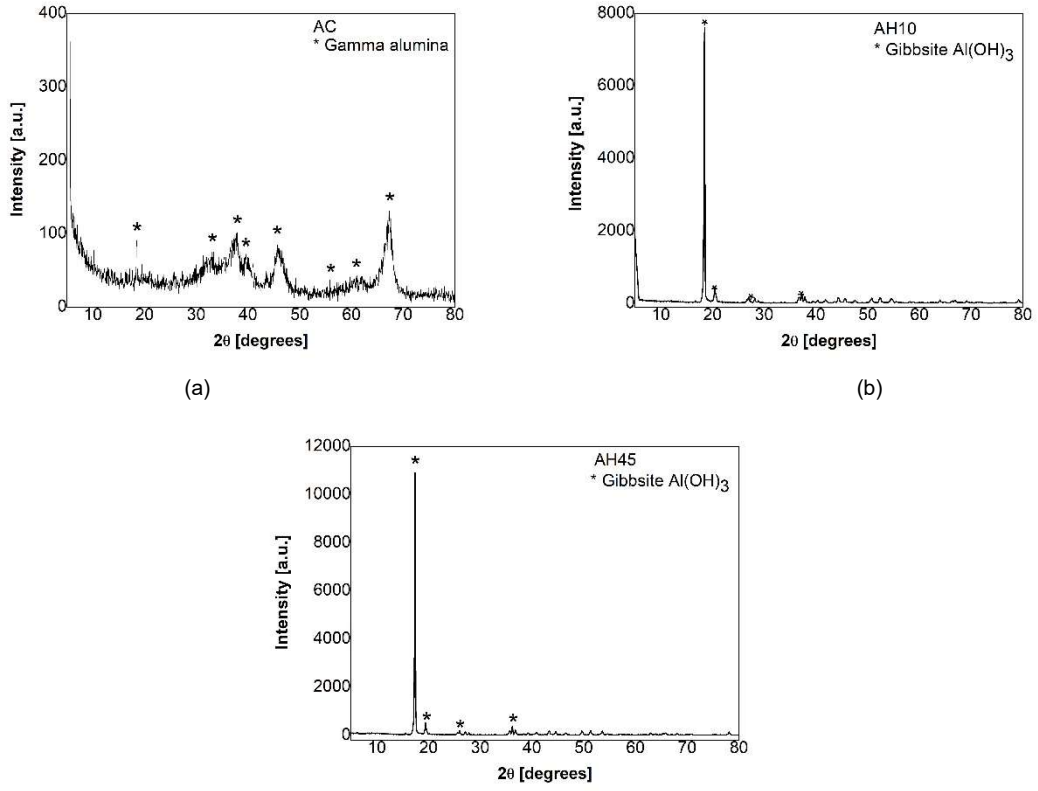

Fig. 3 - X-ray diffraction pattern of the powders of: a – calcined alumina (AC); b – aluminum hydroxide <10 μm (AH10); c – aluminum hydroxide <45 μm (AH45); *Difractograme de raze X ale pulberilor: a – alumina calcinată (AC); b – hidroxid de aluminiu < 10 μm (AH10); c – hidroxid de aluminiu < 45 μm (AH45)*

Grain size distribution and the passing cumulated on granulometric classes are represented in the diagrams from the Figures 1, and 2 respectively.

In the case of calcined alumina powder is highlighted a tight spectrum, almost uniform – monomodal, similar to a Gaussian distribution, with a maximum for granulometric fraction of 100 μm. Grain size distribution curves of alumina hydroxide powders present relatively large spectra of distribution, with asymmetric aspect, which emphasize high content of fine material (cumulative passing reaches values of 75.79% for the powder of aluminum hydroxide AH10 at a diameter of particle of 10 μm, and for the powder of aluminum hydroxide AH45 of 46.65% for a diameter of particle of 10 μm and of 91.83% for a diameter of particle of 45 μm).

From mineralogic point of view, the spectra of X-ray diffraction corresponding to the samples of raw materials, presented in the Figure 3, highlight the presence of gamma alumina in the sample of calcined alumina (AC) and of gibbsite in the samples of aluminum hydroxide (AH10 and AH45), respectively.

3.2. Characteristics of obtained aluminous ceramics

The aluminous ceramics samples were characterized from physical, mechanical, mineralogical and microstructural point of view.

3.2.1. Physical characteristics

The investigated physical characteristics of ceramics samples were apparent density and porosity. The values obtained as consequence of the determination of apparent density and porosity are presented in the Figure 4.

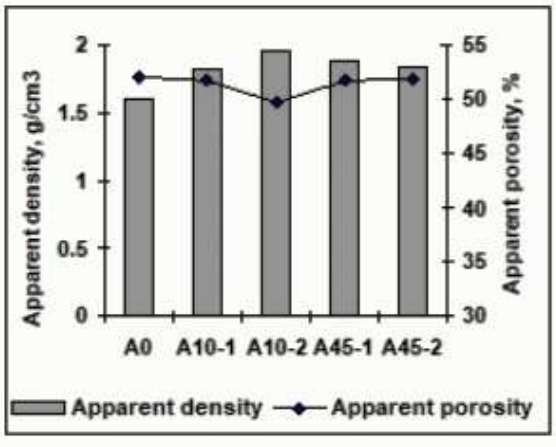

Fig. 4 - Variation of apparent density and porosity depending on the type and the proportion of alumina hydroxide / *Variații ale densității aparente și porozității aparente în funcție de tipul și proporția hidroxidului de aluminiu*

The presence in ceramic masses of alumina hydroxide determine the increasing of apparent density in comparison with standard composition (A0) with 100% calcined alumina. Apparent

porosity of the ceramics with aluminum hydroxide AH 10 dosed in proportion of 25% and of those with AH 45 dosed in proportion of 25 and 50% is comparable with that of the standard sample.

3.2.2.Mineralogic and structural characteristics

Diffractograms corresponding to standard ceramics (A0) and of those with 25% aluminum hydroxide, fraction below 10μm (AH10) and fraction below 45μm (AH45) are presented in the Figure 5.

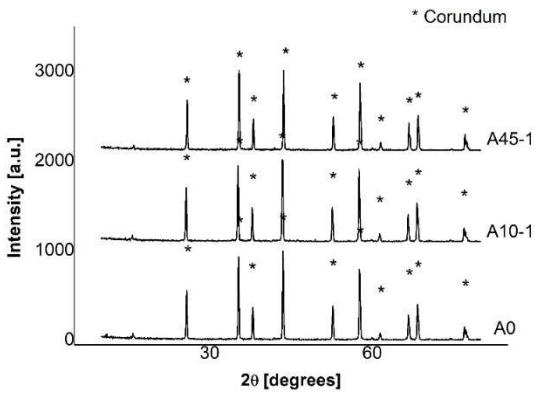

Fig. 5. X-ray diffraction patterns of aluminous ceramics: a – A0; b – A10-1; c – A45-1 / *Difractograme de raze X ale ceramicilor aluminoase: a – A0; b – A10-1; c – A45-1.*

Diffraction of X-rays had put into evidence, in all samples, the presence of alumina in the form of corundum. The intensity of corundum-specific interferences is similar in all samples. It can be said that the crystallinity of the samples is not influenced by the presence of aluminum hydroxide in the ceramic composition, nor by the size of its particles. A same behavior regarding the particle size of aluminum hydroxide on the crystallinity was noticed by Gan et al. [18], too.

The images of scanning electron microscopy put into evidence, in all analyzed samples, a relatively homogeneous structure, crossed by irregular cracks. The presence of cracks may be caused by volumetric contraction at burning of the test pieces, this being of 22 … 25%. Volumetric contraction can be explained both by the loss of the binder used to obtain the specimens (polyvinyl alcohol), and, in the case of the samples A10 and A45, by dehydroxylation of aluminum hydroxide [19, 20]. Another cause of sample's crack can be attributed to the technology of pressing.

Cracks wide is higher in the sample A0 (530 nm ... 12.96 μm), in comparison with those from the samples A10 and A45 (A10-1: 212 nm ... 12.27 μm; A10-2: 532 nm ... 2.55 μm; A45-1: 21 nm ... 10.6 μm; A45-2: 420 nm ... 10.63 μm).

In the images performed at the magnitude of 20000x the grains of alumina of rounded shape, of different sizes are remarked, between which communicating bridges were formed. In the sample A0 (in which just alumina as raw material was used), granules dimension, in scanned zone, is of 540 nm …2.1 μm, the grain boundaries being apparent; the granules are intense inter-connected with low intergranular porosity. This structure was also highlighted by S. Lamouri et al. [21] in gamma alumina samples burned at 1700°C with a speed of 5°C/min, and they say that the cause is a well established particles rearrangement during the γ Al_2O_3→ α Al_2O_3 transformation process. In the samples A10-1 and A10-2 (in which aluminum hydroxide below 10 μm was used) individual granules inter-connected through communicating bridges have, in scanned zones, dimensions of 312 … 940 nm, and those powerful inter-connected reach at dimensions of 1.6 … 1.65 μm (especially in the sample A10-2). In the samples A45-1 and A45-2 (containing aluminum hydroxide below 45 μm) individual granules of dimensions of 480 … 950 nm are remarked, inter-connected through communicating bridges. The tendency of granules to be inter-connected through communicating bridges had created pores and irregular channels in the samples where aluminum hydroxide was used as raw material (A10 and A45), these showing a coral-like co-continuous morphology [19]. Some measurements in scanned zones showed that pore dimensions are from 100 nm to 24 μm (A10-1: 106 nm… 23.78 μm; A10-2: 138 …295 nm; A45-1: 145.7 nm …4.21 μm; A45-2: 219 nm…2.82 μm).

In the Figure 6 electron-microscope images of the samples with aluminum hydroxide below 10 μm and 45 μm respectively, near standard sample are presented.

3.2.3.Mechanical characteristics

In the Table 3 the values of mechanical strengths of studied ceramic samples are presented.

Significant increases of 77 ... 123% of the compressive strengths of all samples containing aluminum hydroxide are observed compared to the standard sample. The best mechanical properties are developed by ceramic mass A10-2 prepared with aluminum hydroxide having more advanced fineness, assuring a better compaction. Also, in the case of aluminum hydroxide compositions below 10 μm, there is a 52% increase in mechanical compressive strength with increasing proportion of aluminum hydroxide. In the case of the compositions with aluminum hydroxide below 45 μm the decreasing of mechanical characteristics at a higher dose of aluminum can be attributed to a weak densification of these, but all values are superior to standard sample. The use of the coarser powder (below 45μm) in the proportion of 25% leads to the obtaining of ceramics with physical-mechanical characteristics close to those resulting from the use in the proportion of 25-50% of the finer powder (below 10 μm), with beneficial effect on production costs.

The best mechanical properties are developed by ceramic mass A10-2 prepared with aluminum hydroxide having more advanced fineness, assuring a better compaction. The values of compression strength are correlated with apparent density. The yield strength and Young 's modulus is also improved in the case of samples containing aluminum hydroxide.

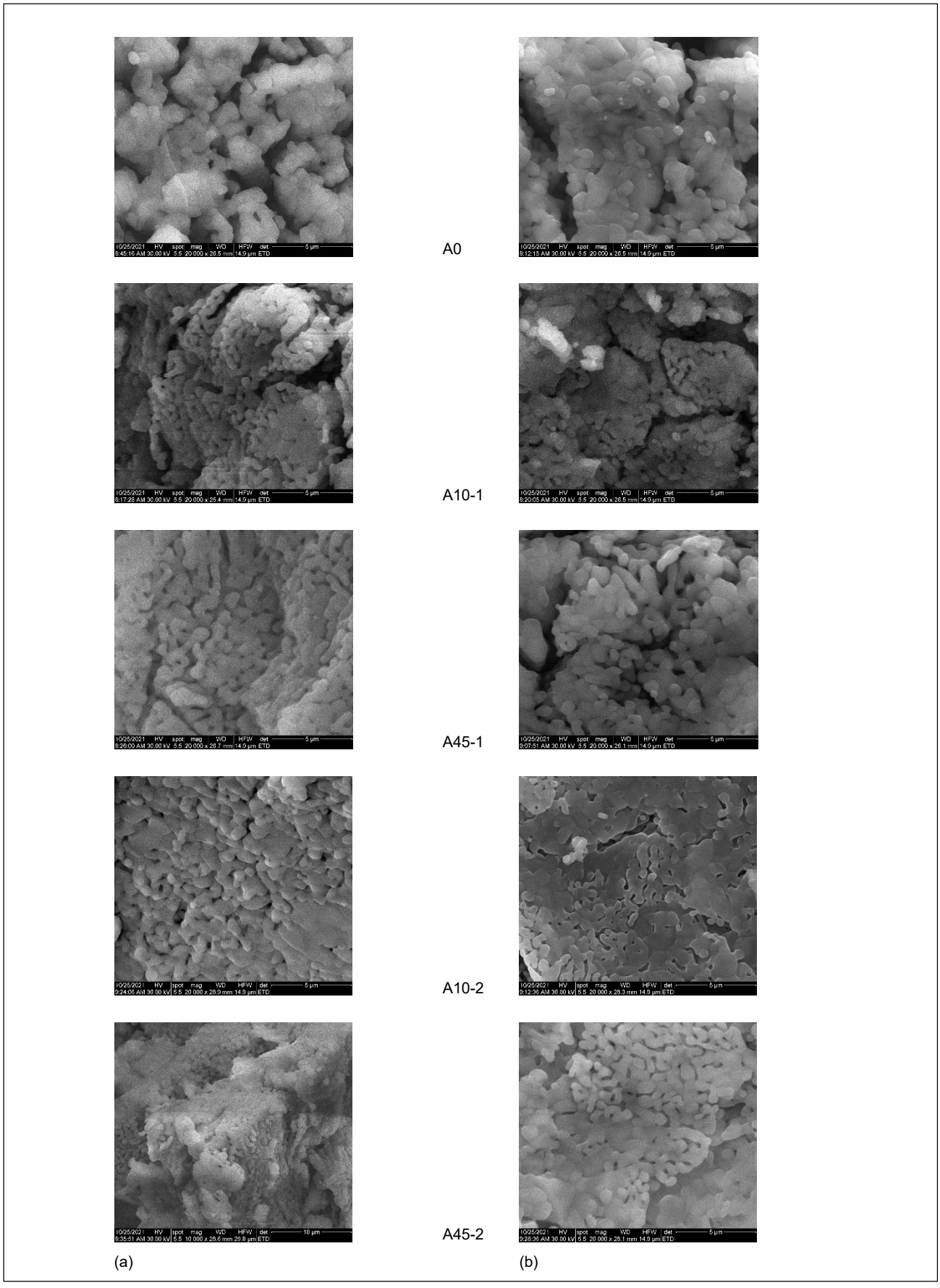

Fig. 6 **-** Images of scanning electron microscopy of the samples without aluminum hydroxide (A0), aluminum hydroxide below 10 μm (A10-1 and A10-2) and aluminum hydroxide below 45 μm (A45-1 and A45-2): a – on the fracture; b – on the surface of the sample / *Imagini de microscopie electronică ale probelor fără hidroxid de aluminiu (A0), cu hidroxid de aluminiu sub 10 μm (A10-1 și A10-2) și hidroxid de aluminiu sub 45 μm (A45-1 și A45-2): a – în fractură; b – pe suprafața probei.*

Table 3

Mechanical strengths of aluminous ceramics / *Rezistențe mecanice ale ceramicilor aluminoase*

Characteristic	M.U.	Code of compositions				
		A0	A10-1	A10-2	A45-1	A45-2
Mechanical strength at compression	MPa	4.44	9.92	15.09	11.00	7.87
Yield strength, at compression test	MPa	2.49	3.92	5.42	4.59	3.21
Young's module, at compression test	GPa	0.28	0.32	0.46	0.41	0.29

Table 4

The values of the thermal expansion coefficient depending on the temperature
Valori ale coeficientului de dilatare termică în funcție de temperatură

T, °C	α x 10^{-6} / K^{-1}		
	A0	A10-1	A45-1
100	8.667	8.806	9.130
200	10.691	11.746	12.569
300	11.759	12.463	12.944
400	11.274	11.422	11.613
500	10.252	11.407	11.205
600	10.531	12.139	12.771
700	11.375	13.210	12.716
800	11.857	13.714	13.316
900	12.388	14.400	13.864
1000	11.894	13.222	15.139
1100	10.251	10.346	13.566
1198	6.569	2.569	7.890

Table 5

Average values of the thermal expansion coefficient in temperature range of 200 – 1100°C
Valori medii ale coeficientului de dilatare termică în domeniul de temperatură 200 - 1100°C

Characteristic	Composition code		
	A0	A10-1	A45-1
α x 10^{-6} / K^{-1}	11.227±0.758	12.407±1.235	12.970±1.116

3.2.4.Thermal characteristics

Thermal expansion coefficient was determined in order to supply information about dimensional stability of ceramic object in operation period from very low temperatures until the highest ones.

The determination of the coefficient of thermal dilatation was performed on samples of aluminous ceramics A0, A10-1 and A45-1, in the following conditions: temperature range 20 ... 1200°C, in atmosphere of static air, at a heating speed of 5^0C/min.

The values of the coefficient of thermal dilatation depending on temperature and average values of this in temperature range 200 – 1100°C are presented in the Tables 4, and 5 respectively.

The ceramic samples with aluminum hydroxide have a similar behavior to that of the standard, the sample with aluminum hydroxide below 10 microns being the best.

In the Figure 7, the diagram of the curves $\Delta L/L_0$ and the thermal expansion coefficient (α) depending on temperature for the sample A45-1 are represented. It is remarked that on temperature range 200 – 1100°C the sample present a narrow interval of variation of the thermal expansion coefficient.

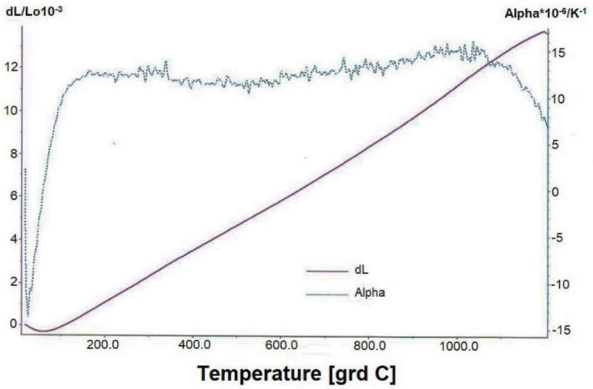

Fig. 7 - Diagram of the curves $\Delta L/L_0$ and the thermal expansion coefficient (α) depending on temperature for the sample A45-1 / *Diagrama curbelor $\Delta L/L_0$ și coeficientului de dilatare termică (α) în funcție de temperatură pentru proba A45-1.*

4.Conclusions

The using of aluminum hydroxide in aluminous ceramics had led to the obtaining of some physical characteristics (porosity and density) superior to the standard ceramic performed from calcined alumina. The dosing of aluminum hydroxide (AH 10), with minimum 95% particles below 10 μm, in proportion

of 50% lead to obtaining of the best compressive strength, increasing in comparison with standard ceramics being of 123%.

The use of 25% of aluminum hydroxide below 45 microns in the mixture allows to obtain a ceramic with better porosity and mechanical characteristics compared to the mixture in which the same percentage of finer powder of aluminum hydroxide (below 10 microns) was used. Increasing the proportion of coarser aluminum hydroxide to 50% worsens the mechanical properties, but the values obtained are close to those resulting from the use of finer aluminum hydroxide in the proportion of 25%.

The ceramics obtained through combination of calcined alumina with aluminum hydroxide present good apparent porosity and thermal stability, but optimizations of obtaining technology are necessary in order to increase mechanical strength in order to be used at performing of filtering elements.

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