COMPORTAREA CERAMICELOR UŞOARE CU CONŢINUT DE CENUŞĂ ZBURĂTOARE DE TERMOCENTRALĂ TRATATE ÎN CÂMP DE MICROUNDE

BEHAVIOUR OF LIGHTWEIGHT CERAMIC OBTAINED WITH COAL-FIRING POWER STATIONS FLY ASH BY CURING IN MICROWAVES FIELD

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The purpose of this work is to examine the possibility of developing a new way to use abundant ash waste from coal burning through a rapid thermal treatment of ceramic composite bodies. The formulation of ceramic bodies was realised with clay and 10-50% (by weight) fly ash (class F) originated from bituminous coal burning and followed by rapid sintering at 950°C, 1000°C and 1050°C in microwave field at varying intervals of time ranging from 5 to 20 minutes. The ceramic properties in conjunction with morphological-structural characteristics after microwave field roasting were investigated. The results show that the development of lightweight ceramic starting from clay and coal ash can be effective in appropriate heat treatment conditions in microwave field at 2.45 GHz frequency. The effectiveness of the sintering process was evaluated as a function of the sintering time and specimen composition. The mineralogical phase analysis of samples cured at 1050°C for 20 minute showed the major crystalline mineral compounds as: quartz, mullite and hematite. The ceramic properties recommend them as lightweight aggregates for application in building materials area.

Scopul acestei lucrări constă în examinarea posibilităţilor de valorificare a deşeurilor abundente de cenuşă de termocentrală rezultate din arderea cărbunilor, prin aplicarea unui tratament termic rapid al maselor compozite ceramice. Compoziţiile maselor ceramice au fost proiectate pe bază de argilă şi cenuşă zburătoare (clasa F) în pondere de 10-50% (în greutate) provenită din arderea cărbunelui bituminos, urmată de sinterizarea rapidă la 950° C, 1000° C şi 1050° C în câmp cu microunde, la intervale variabile de timp cuprinse între 5 şi 20 de minute. Au fost investigate proprietăţile ceramice în corelaţie cu caracteristicile morfologice şi structurale după tratamentul în câmp de microunde. Rezultatele arată că dezvoltarea ceramicii uşoare pornind de la argilă şi cenuşă de termocentrală poate fi eficientă în condiţii adecvate de tratament termic în câmp de microunde la frecvenţa de 2,45 GHz. Eficacitatea procesului de sinterizare a fost evaluată ca o funcţie a timpului de sinterizare şi a compoziţiei probelor. Analiza mineralogică a probelor arse la 1050 ° C timp de 20 minute a evidenţiat prezenţa principalilor compuşi cristalini ca: cuarţ, mulit şi hematit. Proprietăţile ceramice ale maselor investigate le recomandă ca agregate uşoare pentru aplicaţii în domeniul materialelor de construcţii.

Keywords: *Coal ash, Lightweight ceramic, Microwaves*

1. Introduction

The fly ash resulting after coal burning is one of the most important wastes produced in the world, including Romania and its historical dumps or currently generated has become a real challenge for researchers. Generating massive ash coal firing thermal power plant involving environmental and economic issues, as far as storage of such waste has recently become stricter because of environmental regulations and high costs related to waste disposal. The physical and chemical properties of ash obviously depend on the type of coal used and combustion conditions. The coal ash contain valuable oxides, such as SiO_2, Al_2O_3, CaO, Fe_2O_3 and other oxides having a variable composition, density, texture, porosity and water absorption capacity. At the same time, given their very fine grain size, they can be used directly in the production of lightweight ceramic without any communition treatment [1]. During technological process of coal combustion in boilers, ash and bottom ash (slag) results separately. Ash contains fine particles with diameter less than 0.25 mm (also called fly ash, as is easily driven by wind). Slag consists of particles having size of 0.25 – 1 mm or more. The diverse chemical, mineralogical and morphological properties of ash offer an opportunity to process it and recover various fractions with particular attributes. A variety of fly ash has been converted into useful ceramics and glass-ceramics (GCs) by several research groups [1-12]. Up to date, the investigation was mainly oriented towards the production of dense ceramic tiles and GC materials for use as architectural components in buildings. On the other hand, fly ash has potentially hazardous nature, mainly due to the toxic metals that it contains (e.g. Cadmium, Zinc, Lead, Mercury

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etc.). Thus, for the inertization of fly ashes, it is necessary to develop new technologies in order to immobilize their dangerous components in glass, glass-ceramic or ceramic materials. The chemical composition of fly ash is typical of a common glassy quaternary system (SiO_2–Al_2O_3–CaO-Fe_2O_3) and therefore, it is feasible to produce glass materials from coal fly ash. The production of vitreous materials can be an effective route for recycling of fly ash because the high temperature involved in the process leads to the complete destruction of the organic pollutants, Furthermore, heavy metals can be incorporated in the glassy product [1]. The fly ash was introduced into a red firing terracotta composition and the optimum fly ash addition was found to be 5 % at the maximum firing temperature of around 900^{o}C [4]. Very interesting results for mixtures of 60 wt % fly ash and 40 wt % clay, with firing temperatures between 900^{o}C and 1200^{o}C in the processing of pressed ceramic products were obtained [5]. The use of fly ash as a raw material in the ceramic field is supported by the large production of ceramics products and the amounts of coal fly ash [6-7]. In Romania only a small amount is utilized, mainly in the concrete and Portland cement production, etc. The addition of fly ash can significantly improve the workability of self compacting concrete and has a positive effect on the drying shrinkage and reduces the drying shrinkage.The drying shrinkage decreases with the increase of fly ash content at all ages of self compacting concrete [13]. Recently, concretes were obtained with unitary Portland cement or with fly ash addition and recycled aggregates resulted from old crushed concretes [14].

The purpose of this work is to examine the possibility of developing a new way to use abundant ash waste from coal burning through a rapid thermal treatment of ceramic composites bodies. Mixtures of caly and fly ash originating from a Romanian power plant was investigated as a source of secondary raw material to produce lightweight ceramic materials.

Microwave energy offers many advantages for eco-friendlier processing of materials over conventional processing. The microwave processes, with their selective and volumetric heating, can enhance the solidification efficiency and reduce the processing cycle time considerably, thereby resulting in substantial energy and cost savings [15-17]. They also provide finer microstructures leading to improved mechanical properties. These characteristics provide sufficient motivation to promote the use of microwaves in “greener” materials processing. The potential use of microwave technology as an energy-efficient alternative to conventional heating technologies employed in the processing and treatment of waste, such as used tyres, plastic waste and even sewage sludge, is currently being investigated.

The chemical composition of fly ash consists of silica, alumina, iron oxide, calcium oxide, etc. - mostly in glassy state. Morphologically, fly ash is composed of tiny solid or hollow spheres. Due to the complex composition, the melting temperature is narrow. Microwave sintering of various ceramic materials such as: glass-ceramics, nano-ceramics and bio-ceramics has been investigated [15-26]. Since the heat during the microwave sintering is generated due to microwave field – material interaction, volumetric heating can be achieved if the piece is within the penetration depth of the microwaves. Actually, microwave energy is delivered directly to the material through molecular interaction with the electromagnetic field, as microwaves can penetrate the material and supply energy. The dielectric parameters (dielectric constant and loss tangent) determine the effect of a given microwave electromagnetic field upon the material. The dielectric constant measures the ability of a material to store microwave energy. The loss tangent provides an indication of how well a material can be penetrated by the electric field and how it dissipates energy as heat. Since energy transfer occurs at a molecular level, the interaction of microwaves with dielectric material results in translational motions of free or bound charges and rotation of the dipoles. The resistance of these induced motions due to inertial, elastic, and frictional forces causes heat losses, and thereby heat can be generated throughout the volume of the material resulting in volumetric heating, thus reducing processing time and enhancing product quality. Therefore, the thermal gradient in a microwave-processed material is the reverse of what happens in a material processed by conventional heating, where slow heating rates are normally selected to reduce an abrupt thermal gradient possibly leading to process-induced stresses. Moreover, rapid heating that is possible upon microwave sintering usually favors densification while limiting excessive grain coarsening.

2.Materials and methods

2.1. Batch preparation

Five composition of mixtures of clay and fly ash as given in **Table 1** were uniaxially cold pressed in a stainless steel die using a hydraulic press to form a series of 15 mm diameter green compacts (pellets). A lubricant was applied on the die wall. All compacts obtained at 10 MPa had a reasonable green strength for the subsequent manufacturing.

The coal fly ash used in these experiments result from the combustion of bituminous coal in a thermal power plant was added to a clay occurring from Romania, too. The chemical composition is presented in **Table 2.**

Table 1

Composition of fly ash and clay pellets / *Compoziţia peletelor realizate pe bază de cenuşă zburătoare şi argilă*

% by weight	FAK10	FAK20	FAK30	FAK40	FAK50
Fly ash	10	20	30	40	50
Clay	90	80	70	60	50

Table 2

Chemical composition of fly ash and clay / *Compoziţia chimică a cenuşii zburătoare şi a argilei utilizate*

%	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	Na_2O	K_2O	SO_3	LOI*
Fly ash	53.73	24.75	3.44	1.66	11.6	0.57	2.18	0.26	1.81
Clay	56.09	25.31	2.85	1.98	1.56	2.32	2.64	0.4	6.85

*Loss of ignition

2.2. Microwave curing

With microwave processes, it is possible to achieve rapid and uniform heating of thick materials. Microwave irradiation at frequency of 2.45 GHz was used in our trials heating. Microwave sintering of the green specimens was carried out at at 950°C, 1000°C and 1050°C in microwave field at varying intervals of time, namely at 5, 10, 15 and 20 minutes in a special purpose microwave oven with installed power of 1000 W designed for laboratory rapid heating of materials. Finally, the sintered specimens were gradually cooled to ambient temperature in the furnace. The effectiveness of the sintering process was evaluated as a function of the sintering time and specimen composition.

2.3. Physical-chemical analysis

The elementary samples of raw materials were mixed in order to obtain cumulative average samples of 1 kg prepared for laboratory analyses according to SR ISO 5069-1and 2:1994. General physical-chemical characterization of cumulative average samples was focused to establish the limits variation of the basic parameters, as: dimensional distribution of particles (according to SR ISO 1953: 1999-Coals, Grain size analysis), moisture content (according to SR ISO 331:1994-Coals, Total moisture content), bulk density (STAS 5630-73-Coke, Determination of bulk density).

Laboratory tests have been carried out in order to establish the elemntal chemical composition of the supplied ash samples. For the determination the X-Ray Fluorescence (XRF) method according to ASTM E 1621-05 and SR EN ISO 12677:2004 was used. The XRF analysis was performed using an Axios-Panalytical device and the corresponding (IQ+) soft, allowing qualitative and semiquantitative evaluation of the chemical composition. An amount of 6 gram of ash has been melted in a flux of lithium tetraborate, in a Pt crucible using an induction furnace at temperature 1200°C, Finally, a 32 mm diameter homogeneous lens was obtained and analyzed by using a sequential X-ray spectrometer.

2.4. Mineralogical analysis

Mineralogical phase analyzes were performed by X-ray diffraction method with parallel beam - scanning axis 2θ / θ, the bulk samples. A Shimadzu XRD 6000 diffractometer with the radiation generator tube power of 1200 W, respectively using Cu-Kα characteristic radiation (λ = 1,541874 Å) was employed. Scanning range (2θ) of goniometry was located between 5° and 80°, with 5°/minute angular speed and 0,02° step.

2.5. TG/DTA analysis

TG/DTA analysis was performed on a Mettler Toledo 851 equipment in the 25 – 1300°C temperature range, under normal atmosphere (air) and a heating rate of 10°C/min. For making the determination the ash samples were placed in crucibles of high purity aluminium oxide.

3. Results and discussion

3.1. Mineralogical and morphological characteristics

Mineralogical phase analysis carried out by X-ray diffraction method highlighted a non crystalline pattern as shown in Figure 1**.** The fly ash is constituted of an amorphous phase of aluminosilicate glass indicated by a broad pattern recorded on the diffraction spectrum.

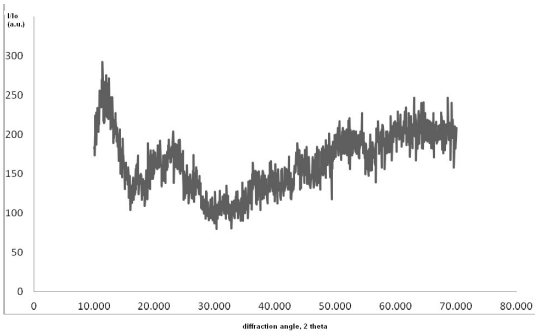


Fig.1 - XRD pattern of F class fly ash / *Difractograma cenuşii de termocentrală (clasa F).*

3.2. Complex thermal analysis

TG/DTA diagram for fly ash (on average sample) is given in Figure 2a. It might be noticed the structural exothermal effect at 473°C due to the oxidation of unburned coal and the endothermic effect at 1114°C - 1205°C due to the melting of ash, accompanied by mass loss probably due to the decomposition of some carbonated forms.

The TG/DTA diagram for clay (on average sample) is given in Figure 2b. On DTG curve on can remark its structural changes at 55 °C, 113°C, 115°C, 144°C, 292°C due to the gradually loss of physically and chemically bonded water respectively. The endothermic effect at 513 °C corresponds to dehydroxilation of clay by water

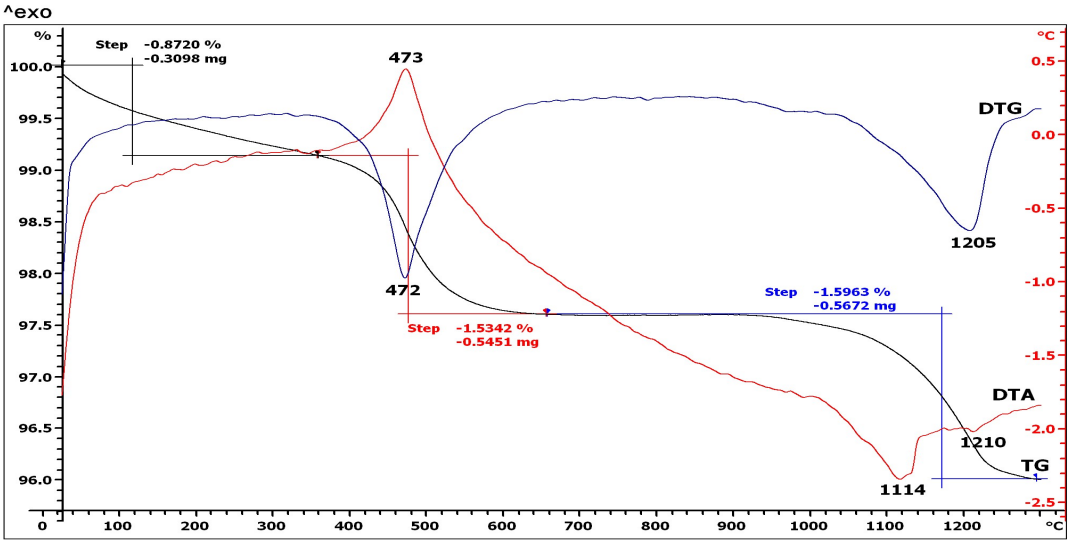


Fig. 2a - TG/DTA diagram for class F fly ash / Diagrama TG / DTA a cenuşii de termocentrală (clasa F).

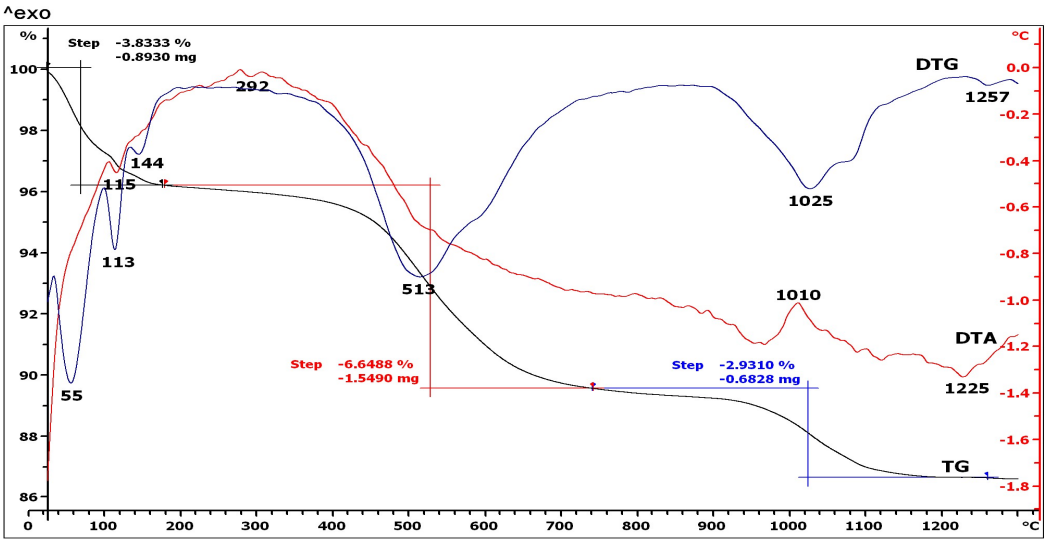
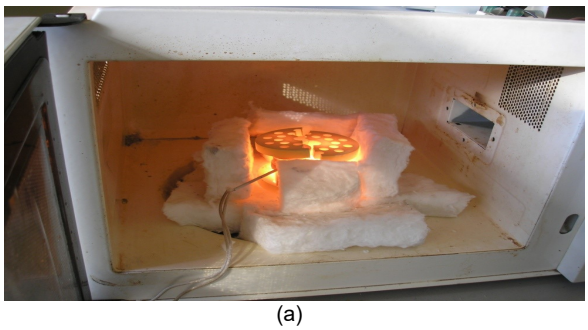


Fig. 2b - TG/DTA diagram for clay / *Diagrama TG / DTA a argilei.*



(a)

(b)

Fig.3 - Inside view of microwave oven (a) and pellets aspect after microwaves curing (b) / *Interiorul cuptorului cu microunde (a) şi aspectul peleţilor după arderea in câmp de microunde (b).*

loss followed by meta-structure formation. The exothermal effects in the range 1010 -1025°C can be assigned to the formation of mullite nuclei and thus having a low crystalinity degree.

Inside view of the microwave oven used for experimental works and the aspect of the pellets obtained after microwaves curing are given in Figure 3. The sintered pellets at 950 °C were yellowish-brown and those heated at 1000 °C and 1050 °C were darker reddish-brown and harder (Fig. 3b), probably due to the formation of liquid phase with increased content of iron oxide at higher temperature. The samples made at 1100°C were deformed due to a large amount of liquid phase formed. For the materials of fair dielectric loss, high heating rates, short processing time and

uniform structure are common features in microvawes field heating. It is expected that the presence of silica, alumina and iron oxide in glassy state to allow the absorbpition of microwave energy very well, as efficient microwave absorption of silica and alumina in amorphous state has been reported [26].

Sintered ceramics in microwaves at 950°C, 1000°C and 1050°C were characterized by bulk density, open porosity and water absorption as function of fly ash content.

In Figure 4 (a,b,c,d,e) is given the evolution of bulk density of sintered pellets as function of curing duration and temperature for the samples: a) FAK10; b) FAK20;c) FAK30; d) FAK40 and e) FAK50. The bulk densities of the lightweight pellets are between 1.02 and 1.21 g/cm^3. The lowest values close to 1.02 - 1.05 g/cm^3 were measured on samples FAK30 fired at 950 °C for 5 and 10 minutes, respectively. The highest value,1.21 g/cm^3, was observed for the sample FAK50 at the temperature of 1050 °C for 20 minutes. The density slightly increased as increasing temperature and curing duration. In Figure 5 (a,b,c,d,e) is shown the evolution of open porosity for the sintered pellets as function of curing duration and temperature for the samples: a) FAK10; b) FAK20;c) FAK30; d) FAK40 and e) FAK50. The lowest porosity, 52-53 %, was on pellets obtained for the sample FAK50 fired at 1050 °C for 20 minutes. The highest porosities 59-60 % were obtained for the samples FAK10, FAK 20 and FAK30 fired at the temperature of 950 °C for 5 -10 minutes. In Figure 6 (a,b,c,d,e) is given the evolution of water absoption of sintered pellets as function of curing duration and temperature for the samples: a) FAK10; b) FAK20;c) FAK30; d) FAK40 and e) FAK50. The water absorption

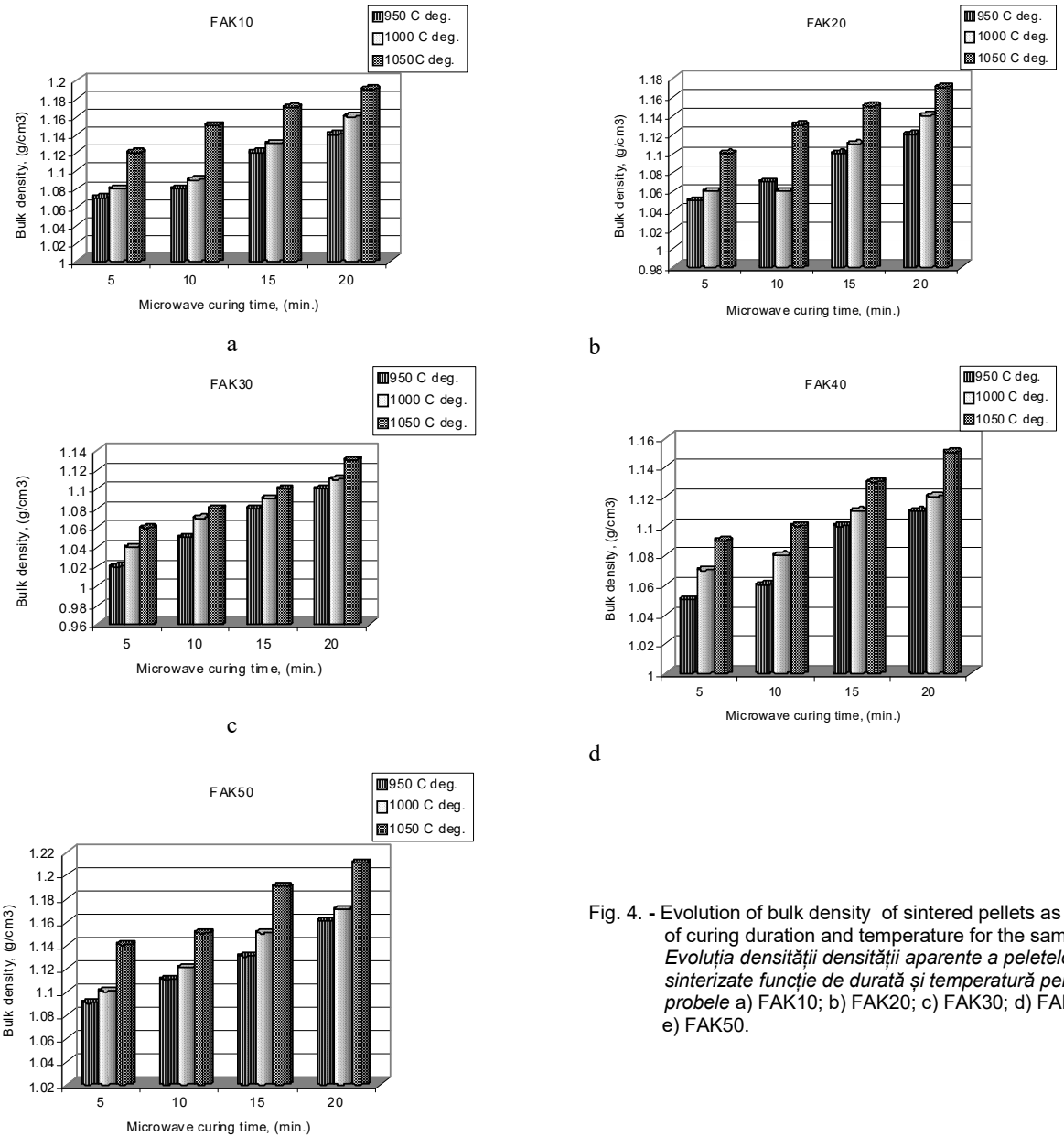
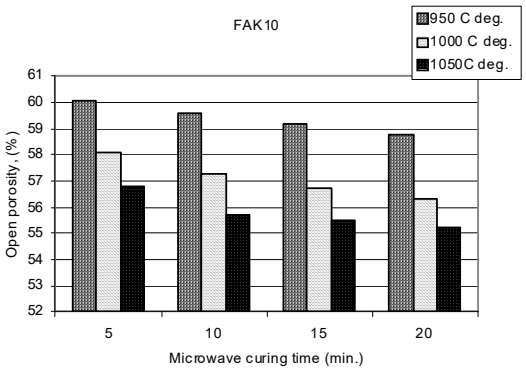


Fig. 4. - Evolution of bulk density of sintered pellets as function of curing duration and temperature for the samples: *Evoluția densității densității aparente a peletelor sinterizate funcție de durată și temperatură pentru probele* a) FAK10; b) FAK20; c) FAK30; d) FAK40; e) FAK50.



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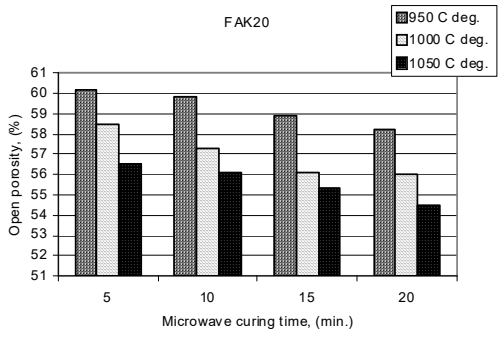

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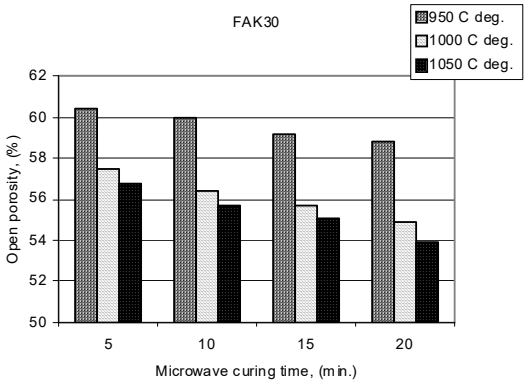

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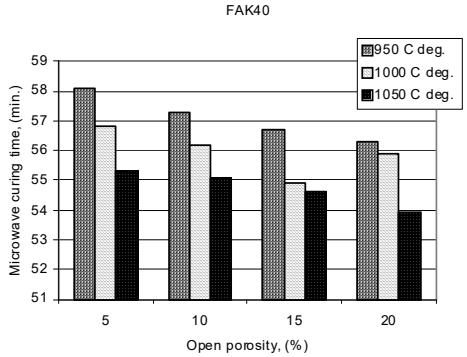

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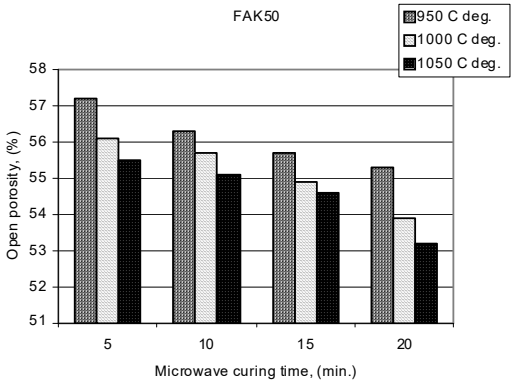
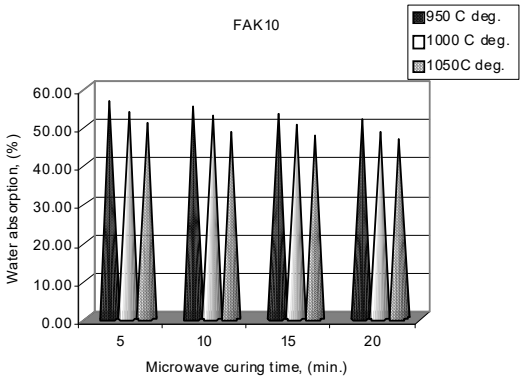


e

Fig. 5 - Evolution of open porosity of sintered pellets as function of curing duration and temperature for the samples / *Evoluţia porozităţii aparente a peletelor sinterizate funcţie de durata şi temperatura pentru probele: a)* FAK10; b) FAK20; c) FAK30; d) FAK40; e) FAK50.



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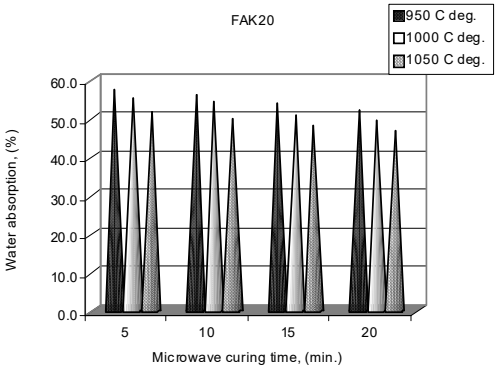

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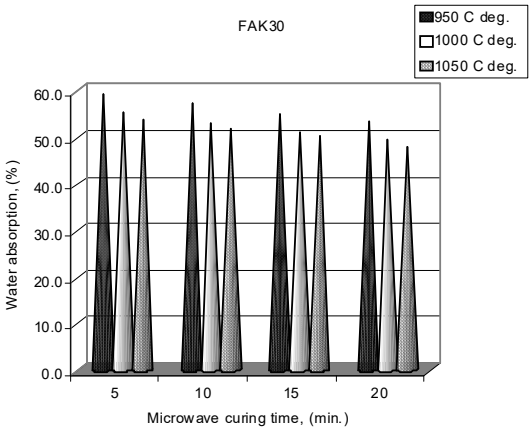
Fig. 6 continues on next page



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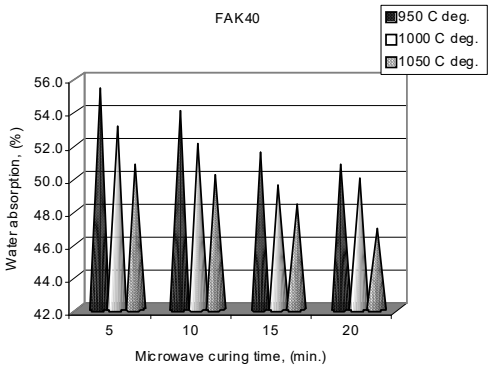

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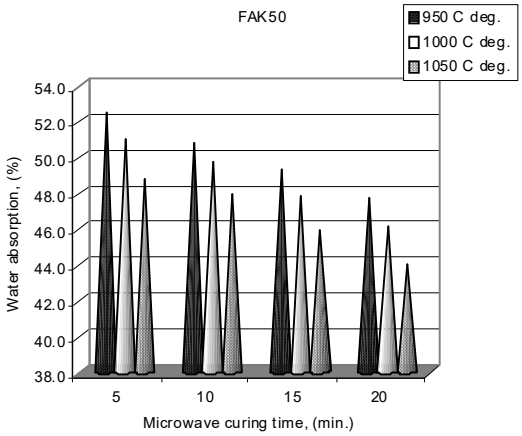

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Fig. 6. - Evolution of water absorption of sintered pellets as function of curing duration and temperature for the samples / *Evoluţia capacităţii de absorbţie a apei a peletelor sinterizate funcţie de durata şi temperatura pentru probele:* a) FAK10; b) FAK20; c) FAK30; d) FAK40; e) FAK50.

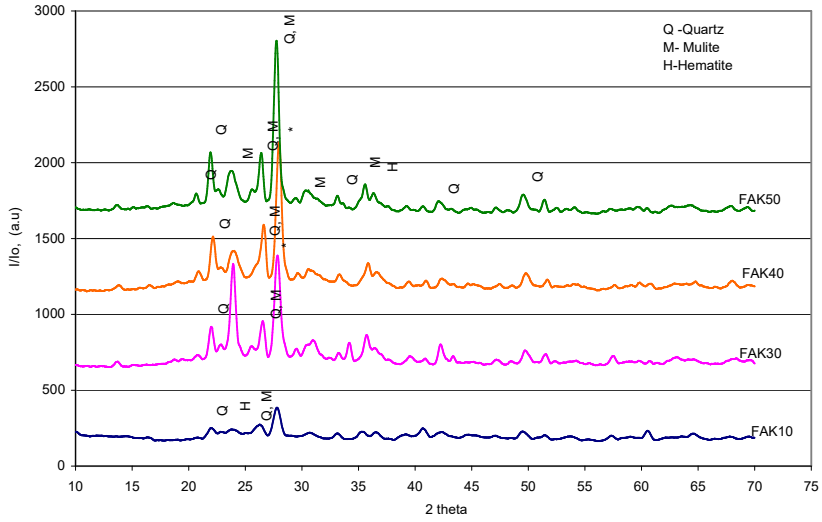


Fig.7 - XRD spectra pattern of lightweight ceramics FAK10, FAK30, FAK40 and FAK50 sintered at 1050 °C for 20 minutes in microwaves / *Difractograma ceramicilor uşoare FAK10, FAK30, FAK40 şi FAK50 sinterizate la 1050 °C timp de 20 minute în câmp de microunde*

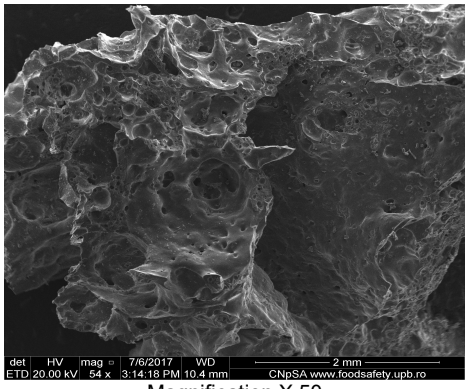
capacity of the pellets produced from clay and fly ash were found between 44% and 59 %. The lowest value was developed by the pellets obtained with 50 % fly ash fired at 1050 °C. Higher water absorption value 59% was registered for the pellets with 30 % fly ash fired at the temperature of 950 °C for 5 minutes and respectively 57% was measured for the pellets with 30 % fly ash fired at the temperature of 950 °C for 10 minutes.

XRD spectra patterns of lightweight ceramics FAK10, FAK30, FAK40 and FAK50 sintered at 1050 °C for 20 minutes in microwaves are given in Figure 7. The X-ray diffraction (XRD) analysis revealed the crystalline phase evolution in conjunction with each heat curing procedure.

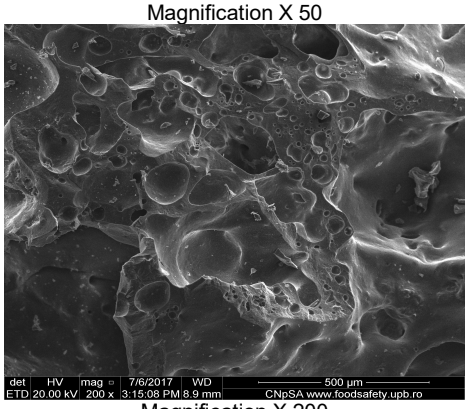
As shown in Figure 7, the major crystalline mineral compounds identified are quartz, mullite and hematite. The quartz, a hard and resistant

mineral, the most unreactive component of the ash, is frequently found on the ash particle surfaces, probably in relatively unchanged conditions, as found before combustion. The hematite presence is due to the ferrous compounds that occur in the coal. There was not significant change in crystallinity and no new phase appeared, either in the samples sintered at 1050 °C for 15 minutes. However, the relative diffraction intensity of the peak o 27.9° (2 theta) increased as increasing fly ash content from 10% up to 50% due to probably overlaping of diffraction lines of quartz and mullite.

Microstructure investigation of sintered samples was carried out by using scanning electron microscopy. In Figure 8 is given the fracture surface microstructures of the samples FAK10 sintered at 1050 °C for 20 minutes, corresponding to 10% insertion of fly ash in the ceramic formulation. The images at various magnification show a porous structure of the



Magnification X 50



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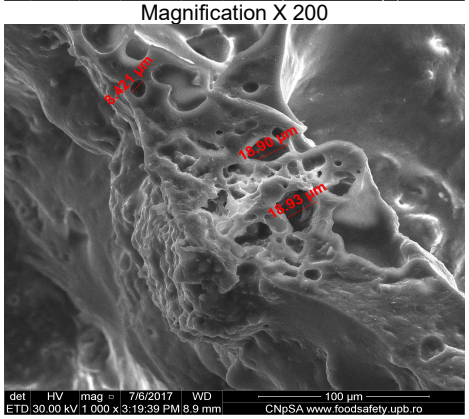

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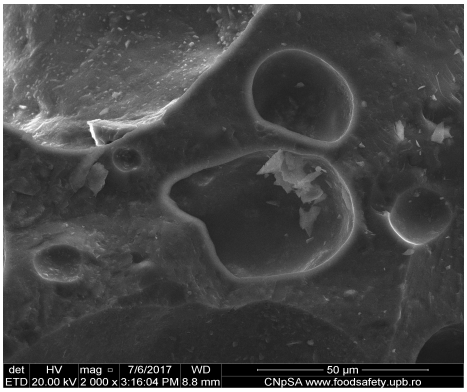


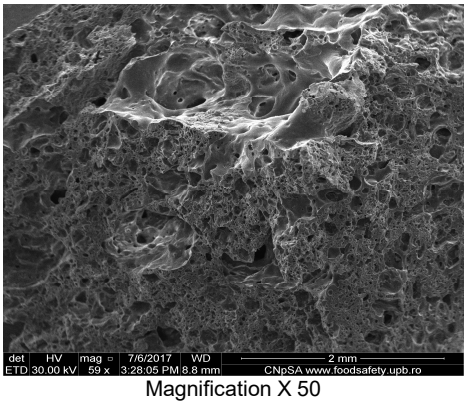
Magnification X 2000

Fig. 8 -SEM micrographs of specimen FAK10, microwave sintered at 1050 °C for 20 minutes / *Micrografiile SEM ale probei FAK10, sinterizate la 1050 °C timp de 20 minute în câmp de microunde.*

ceramic with pore size ranging between 8 -18 µm as well as particles with rounded edges due to liquid phase formation (e.g. Fig. 8 with x 2000 magnification).

In Figure 9 is given the fracture surface microstructures of the samples FAK30 sintered at 1050 °C for 20 minutes, corresponding to 30% insertion of fly ash in the ceramic formulation. The images at various magnification show a porous structure of the ceramic with pore size ranging between 11 - 85 µm determined by a more enhanced the coalescence of the pores.

In Figure 10 is given the fracture surface microstructures of the samples FAK50 sintered at 1050 °C for 20 minutes, corresponding to 50% insertion of fly ash in the ceramic formulation. The images at various magnification show a porous structure of the ceramic with pore size ranging between 5 - 43 µm. On can be noticed a coalescent structure of pores and a more coarser morphology of them.



Magnification X 50

Fig. 9 continues on next page

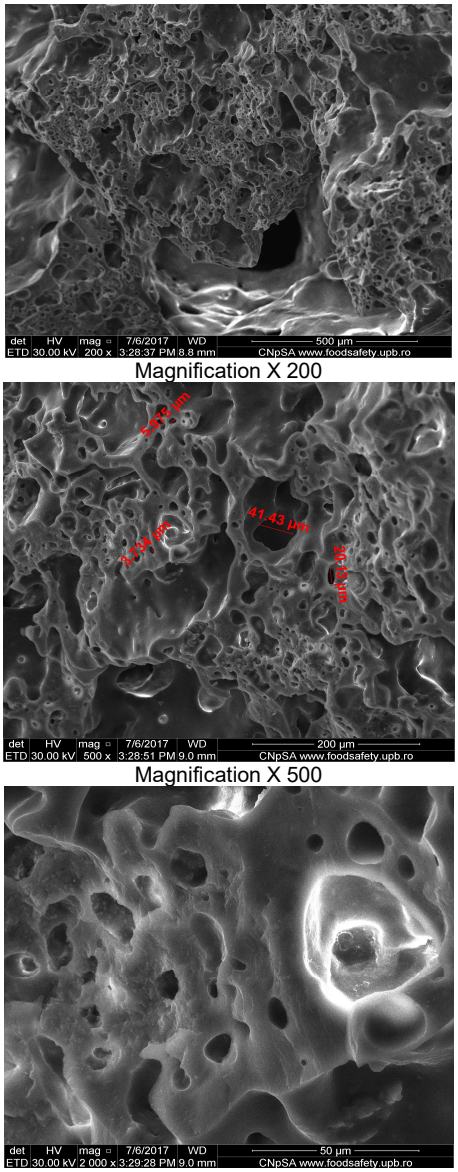


Fig. 9 - SEM micrographs of specimen FAK30, microwave sintered at 1050 °C for 20 minutes / *Micrografiile SEM ale probei FAK30, sinterizate la 1050 °C timp de 20 minute în câmp de microunde.*

The SEM microraphs are consistent with the results of ceramic properties determinations, namely with observed variations of density, open porosity and water absorption.

Microstructural physical and chemical properties of the ceramic samples developed from clay and fly ash in different amounts give an optimistic forecast in incorporating a waste material such is F class fly ash in the ceramic composite materials.

4.Conclusion

Chemical properties of ash samples reveal adequate characteristics in comparison with those of the ceramic raw materials usually used in the ceramic industry. In terms of basic physical

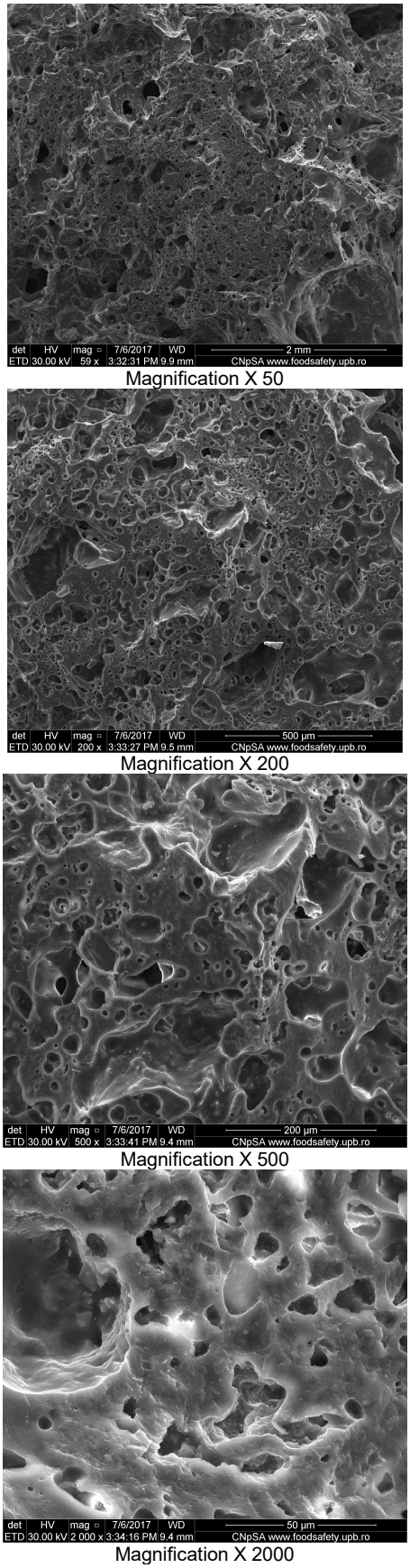


Fig. 10 - SEM micrographs of specimen FAK50, microwave sintered at 1050 °C for 20 minutes / *Micrografiile SEM ale probei FAK50, sinterizate la 1050 °C timp de 20 minute în câmp de microunde.*

characteristics, the ash can be assimilated to a natural sand aggregate of granular type, except for bulk density characterized by lower values. The results show that lightweight ceramic produced with coal-firing power stations ash and clay can by curing in microwaves field to be effective and provide conditions to obtain products with desirable properties suitable for construction sector. In the medium term, the use of lightweight aggregates in the construction industry is expected to grow taking into account their technical and economic advantages: raw materials at low cost, rapid and environmentally friendly processing.

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