Light weight gypsum-based material manufactured by expanding process with aluminum powder

Material ușor pe baza de gips fabricat printr-un proces de expandare cu pulbere de aluminiu

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Rezumat. *Lucrarea prezintă metoda de preparare a unui material ușor pe bază de gips. Principiul spumării materiei prime este aplicat la fabricarea betonului aerat autoclavizat prin eliberarea în masa materialului a hidrogenului prin reacția de coroziune a pulberii de aluminiu în soluție apoasă de $Ca(OH)_2$. Originalitatea lucrării este metoda de generare a aluminiului pulbere prin topirea cu microunde a deșeurilor metalice și atomizarea topiturii cu jeturi concentrate de azot. Produsul are densitatea între 530-600 kg/m^3 și rezistența la compresiune între 1,2-2,2 MPa, fiind similar cu betonul aerat autoclavizat, cu domenii de aplicare identice, însă costuri energetice reduse.*

Cuvinte cheie: material ușor, gips, aluminiu, soluție apoasă, microunde, spumare.

Abstract. *The paper presents the preparing method of a light weight gypsum-based material. The raw material foaming principle is applied to the manufacture of aerated autoclavized concrete by hydrogen release in the material mass by the corrosion reaction of aluminum powder in aqueous solution of $Ca(OH)_2$. The work originality is the generation method of powder aluminum by microwave melting of metal waste and melt atomization by concentrated nitrogen jets. The product has density between 530-600 kg/m^3 and compressive strength between 1.2-2.2 MPa, being similar to aerated autoclaved concrete, with identical application fields, but low energy costs.*

Key words: light weight material, gypsum, aluminum, aqueous solution, microwave, foaming.

1. Introduction

In recent decades, the trend of cellular materials made for buildings has intensified. Especially in countries with harsher climates (Sweden, Norway, Finland, Germany, Poland, etc.) this is one of the most effective materials for enveloping the building as panels, small-size blocks and monolithic buildings [1]. Calcined gypsum ($CaSO_4 \cdot 0.5H_2O$) is considered one of the binders with ecological properties suitable for buildings. Despite this quality, its use in this field is quite limited. According to [2], the achievement of light gypsum products with good thermal and acoustic insulation characteristics and lower transport costs could be a solution for the application of gypsum in buildings. A method of reducing the weight of this material is using light inorganic or organic fillers. The best known inorganic fillers are: perlite (an amorphous volcanic glass) and vermiculite (a phyllosilicate mineral). Gypsum products with inorganic fillers available on the market have a bulk density between 300-800 kg/m^3. Limits can be easy exceeded. An experiment presented in [3] shows that 5-10 % perlite added to a mixture of coal fly ash, lime and gypsum, with a low addition of silica fumes, led to a material with the bulk density of 730 kg/m^3 and the compressive strength of 2.3 MPa. The use of vermiculite together with polypropylene fibers has been tested in [4]. The decrease of the product bulk density by about 10 % and the decrease of thermal conductivity by 30 % were obtained with 20 % vermiculite. The negative effect of the test was reducing the mechanical strength by up to 30 %. Fillers of polymeric materials were also tested in gypsum-based materials. Using polystyrene beads (2 %) and polypropylene fiber (2 %), the product bulk density was decreased by half compared to pure gypsum, while the tensile strength increased by 23 % [5]. Other authors have made products with extremely low bulk density (200 kg/m^3), but the mechanical strength has been greatly affected [2, 6]. Natural fillers of vegetable (chopped straw, sawdust, etc.) or animal (chicken feather, cowhide, hoof, etc.) origin were added to the gypsum mass. The paper [7] presents a solution for the use of cork granules with the size below 12 mm. For 20 % cork filler, the product bulk density was around 800 kg/m^3 and very good compressive strength of about 5 MPa was obtained. By addition of 2 % glass fibers, the tensile strength increased nearly 2 times.

Also, obtaining a porous structure either by chemical reactions that release foaming gases for gypsum expansion, or by surface active substances contributes to the decrease of the weight of the gypsum-based material. The best known reaction that releases foaming gas is the reaction of a carbonate (calcium carbonate $CaCO_3$, sodium bicarbonate $NaHCO_3$ or ammonium bicarbonate NH_4HCO_3) with an acidic component (aluminum sulphate $Al_2(SO_4)_3$, sulfuric acid H_2SO_4, boric acid $B(OH)_3$), in which carbon dioxide (CO_2) is released [2]. The most usual foaming method is the release of hydrogen from the reaction of aluminum powder with the aqueous solution of $Ca(OH)_2$, used in the manufacture of aerated autoclaved concrete (AAC). In the first experiments performed with 65 % gypsum, 33 % $Ca(OH)_2$ and 1 % aluminum powder, the bulk density had relatively small values (640 kg/m^3), but the compressive strength was below 1 MPa [8]. Also, the macrostructure of the material was slightly homogeneous, containing even pores over 5 mm [2]. According to [9], the addition of light fine aggregate (e.g. perlite) homogenizes the macrostructure, and the pore size is significantly reduced.

Several types of waste can be used as substitutes for foaming agents with the ability to release CO_2. Thus, the sludge from the sugar manufacturing process, coal fly ash as a by-product of coal burning in thermal power stations or dust resulting from cutting and polishing marble or granite were used in the manufacturing process of light weight gypsum-based materials such as substituting $CaCO_3$ [2, 10]. According to [10], gypsum based-materials manufactured with dust from stone waste processing had characteristics almost similar to those of aerated autoclaved concrete (bulk density below 600 kg/m^3, thermal conductivity below 0.2 W/m·K and compressive strength around 2 MPa). They are suitable for using as thermal insulation blocks, light weight boards, light weight fire-resistant plasters or thermal insulation plasters.

The objective of the current paper is the manufacture of light weight gypsum-besed material applying an improved technical solution for foaming with aluminum powder aiming to reduce the bulk density and thermal conductivity values and to keep the compressive strength at an acceptable level.

The originality of the work consists on the one hand in applying the own method of producing aluminum powder by spraying the melted aluminum waste with concentrated nitrogen jets in a closed enclosure and on the other hand in adopting new additives compared to the known manufacturing recipes as components of the raw material mixture.

2. Methods and materials

Unlike most processes of foaming silicate raw materials based on chemical reactions to release a gas blocked in the viscous mass (thermally softened) of the mixture, which requires high temperatures (750-1150 ºC) [11], the expansion with hydrogen gas resulting from the corrosion process of aluminum powder in aqueous $Ca(OH)_2$ solution takes place at room temperature. An approximately similar process based on this chemical reaction shown below (1) was successfully tested by the authors' team in a process of foaming the glass waste powder as a raw material mixed with aluminum powder, carboxymethyl cellulose as a stabilizer of the foaming process, $Ca(OH)_2$ and distilled water [12].

$$2Al + 3Ca(OH)_2 + 6H_2O = Ca_3Al_2(OH)_{12} + 3H_2 \quad (1)$$

The cubic $Ca_3Al_2(OH)_{12}$ phase (named katoite) enters in the melting mass of gypsum and the gaseous hydrogen is released. According to the paper [13], the reaction mechanism (1) could include several successive reactions that take place at the separation zone between the outer surface of aluminum particles and the aqueous $Ca(OH)_2$ solution.

$$Al_2O_3 + H_2O = 2AlOOH \quad (2)$$

$$AlOOH + H_2O = Al(OH)_3 \quad (3)$$

$$Al(OH)_3 + OH^- = Al(OH)_4 \quad (4)$$

$$3Ca^{2+} + 2Al(OH)_4 + 3H_2O = Ca_3Al_2(OH)_{12} + 6H_2 \quad (5)$$

The sludge prepared into a ctlindrical metal mold was continously stirred with a metal propeller up to the beginning of expansion process.

The material used in this experiment were: calcined gypsum ($CaSO_4 \cdot 0.5H_2O$), hydrated lime ($Ca(OH)_2$), coal fly ash, perlite, silica fume, carboxymethyl cellulose and aluminum. The oxide composition of coal fly ash, perlite and silica fume are presented in Table 1 [14-16].

Table 1

Oxide composition of coal fly ash, perlite and silica fume

Material	SiO_2 wt. %	Al_2O_3 wt. %	Na_2O wt. %	K_2O wt. %	Fe_2O_3 wt. %	MgO wt. %	CaO wt. %	Other oxides wt. %
Coal fly ash	46.5	23.7	10.1		8.6	3.2	7.9	-
Perlite	70-75	12-15	3-4	3-5	0.5-2	0.2-0.7	0.5-1	-
Silica fume	85-98	< 2.0	< 1.8	< 1.1	< 1.8	< 1.9	< 2.5	< 4.0

Commercially, the calcined gypsum is available ground below 1 mm. Fine grinding and sieving were required for the experimental use, with optimal granulation below 100 μm. The coal fly ash was purchased from the Romanian Paroseni-thermal power station initially with a grain size below 250 μm and after grinding in a ball mill the grain size was reduced to values less than 80 μm. Perlite available on the market is in a granular state being obtained by expanding the ore. Its fine grinding in a ball mill was performed repeatedly until the grain size was reduced below 25 μm, values suitable for its use in the experiment. Silica fume with extremely fineness (below 10 μm) and high silica content was used as such being am effective pozzolanic material, that increases the compressive strength of the material in whose composition it enters. Carboxymethyl cellulose was added in the mixture of solid starting materials as a binder and foam stabilizer. Its solubility in water is very high. For this reason, the fine powder should not be introduced into the water, but the water should be poured over the powder mixture [17].

The aluminum powder with a very fine granulation (below 10 μm) was made by the research team from the Romanian company Daily Sourcing & Research on a molten aluminum waste atomization plant. The waste was melted in a ceramic crucible using an unconventional microwave heating technique and was discharged through a central nozzle. The molten aluminum jet was atomized by the direct contact with several concentrated jets of nitrogen gas distributed at high rate. As a result, a very fine aluminum powder was directed and accumulated to the water-cooled base of the plant. Figure 1 shows a detail of the atomization area of molten aluminum (a) and a batch of fine aluminum granules (b).

a

b

Fig. 1. Details on the atomization of molten aluminum
a – the atomization area of the plant; b – batch of aluminum granules
(Reproduced by permission of Junkoeko SRL Slobozia, Romania).

In principle, the basic manufacturing recipe of gypsum-based material includes gypsum as the main raw material (between 70.7-78.8 %). Coal fly ash (between 3.4-5.1 %), usually a partial replacement of the binder, allowed a slight reduction of the gypsum ratio. Significant proportions of hydrated lime (between 9.5-10.4 %) were adopted to facilitate the corrosion reaction of aluminum in the aqueous solution of $Ca(OH)_2$ and the release of hydrogen as a foaming gas. Silica fume which is an effective pozzolanic material was used in low proportions between 0.7-1.4 %. Perlite with a filler role, which contributes to increasing the bulk density, has been used in a wide range (between 0-10 %). A material that easily forms an aqueous solution and acts as a foam stabilizer was carboxymethyl cellulose used in a low proportion (2 %) kept constant in all four tested variants. Aluminum (3 %) kept constant is the material that practically ensures the foaming process of gypsum by its corrosion reaction in the aqueous medium of $Ca(OH)_2$. The distilled water ratios added supplementary to the mixture of solids varied between 25-35 %.

The proportion of materials used in experiment coresponding to the four variants adopted for manufacturing the gypsum-based materials are presented in Table 2.

Table 2

Weight ratios of materials that compose the experimental variants

Variant	Calcined gypsum wt. %	$Ca(OH)_2$ wt. %	Coal fly ash wt. %	Perlite wt. %	Silica fume wt. %	Carboxy-methyl cellulose wt. %	Aluminum wt. %	Water addition wt. %
1	78.8	10.4	5.1	-	0.7	2.0	3.0	35
2	77.0	10.2	4.8	2	1.0	2.0	3.0	32
3	74.1	10.1	4.6	5	1.2	2.0	3.0	29
4	70.7	9.5	3.4	10	1.4	2.0	3.0	25

Common methods have been applied to characterize the experimentally manufactured gypsum-based materials. The bulk density was determined by the method of dividing the mass of foamed samples loaded into a cylindrical vessel at its volume [18]. To calculate the porosity, the comparison method [19] between the density of the porous sample and the density in compact state (true density) of the same material was applied. Using an analyzer TA.XTplus Texture type, the compressive strength was measured and the Guarded-Comparative-Longitudinal Heat Flow method (ASTM E1225-04) allowed to determine the thermal conductivity value. By the water immersion method (ASTM D570) it was determined the water absorption coefficient of samples. The microstructural characteristics of samples were identified with an ASONA 100X Zoom Smartphone Digital Microscope.

3. Results and discussion

Table 3 presents a synthese of manufacturing process parameters of gypsum-based material. The mass of materials entered in process (dry and wet), the mass of final product, the temperature and duration of foaming process and the index of volume increasing compared to the initial volume are highlighted.

Table 3

Main functional parameters of the expanded process of gypsum

Variant	Dry/wet starting material amount (g)	Process temperature (ºC)	Process duration (min)	Index of volume increasing	Expanded gypsum amount (g)
1	230/310.5	26	11	2.50	218.6
2	230/303.6	26	9	2.20	218.2
3	230/296.7	26	8	2.00	218.9
4	230/287.5	26	6.5	1.70	218.5

The macrostructural apearance of the cross section of samples corresponding to the four experimental variants is shown in Figure 2.

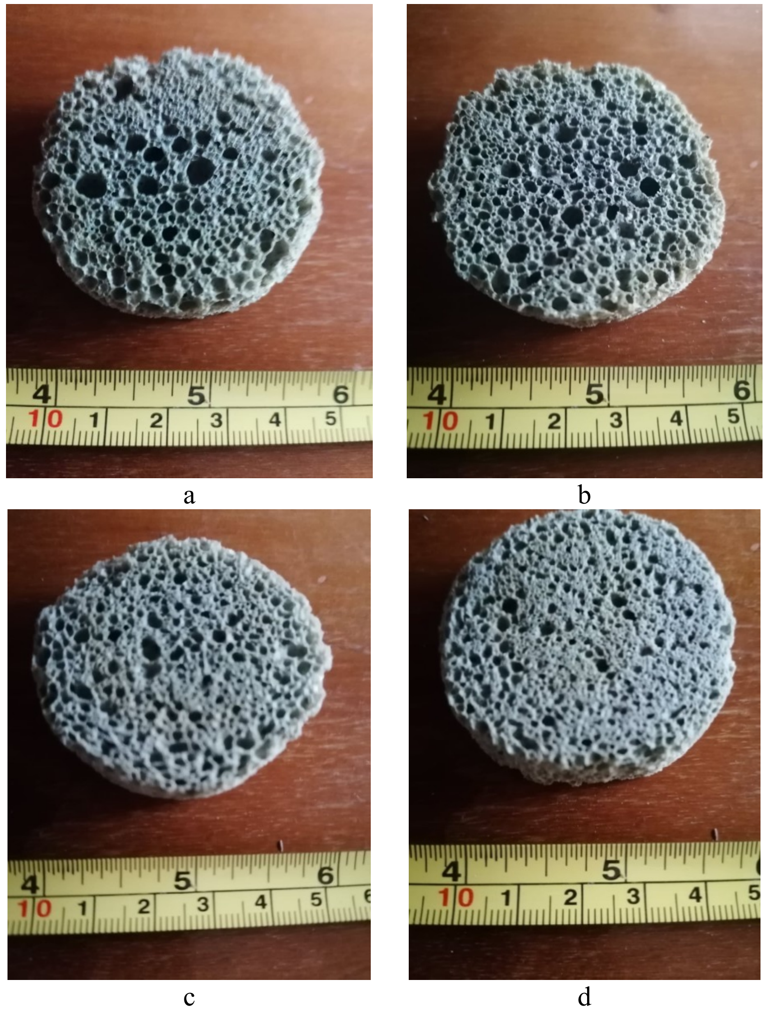


Fig. 2. Macrostructural apprearance of the cross section of samples
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

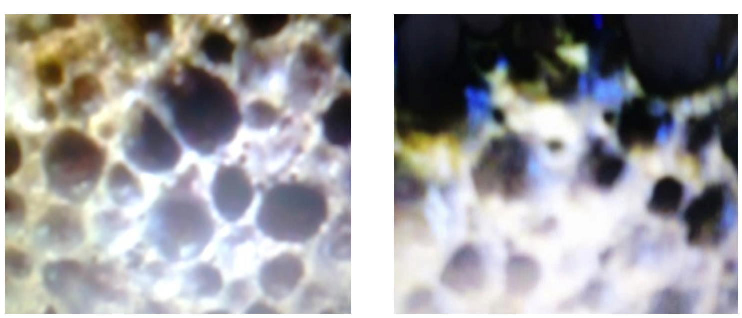
According to the data in Table 3, the dry starting material was estabilished at 230 g and, due to the variation of water addition proportion, its wet mass was between 287.5-310.5 g. The ambient temperature at which the manufacturing process took place was 26 ºC kept constant during the experiment. The foaming process was completed in a variable time period between 6.5-11 min, the increase in volume of the starting material being 2.50 in the case of variant 1 and only 1.70 in the case of variant 4. Examining the pictures in Figure 2, the change of the macrostructural appearance of the samples can be observed. If in the case of variant 1 the structural inhomogeneity is characteristic, in the case of variant 4 its macrostructure is significantly improved. The improvement of this property has an increasing trend from variant 1 to variant 4. Practically, the highest coal fly ash proportion (5.1 %), the lowest silica fume proportion (0.7 %) and the total absence of perlite were the decisive factors that determined the maximum expansion of gypsum-based material in variant 1. In contrast, the reduction of coal fly ash to 3.4 %, the increase of silica fume to 1.4 % and the large increase in perlite up to 10 % created an obviously denser product with much improved macrostructural homogeneity by decreasing the pore size and uniformizing their distribution (variant 4). The main physical, thermal, mechanical and microstructural characteristics of the gypsum-based material samples are presented in Table 4.

Table 4

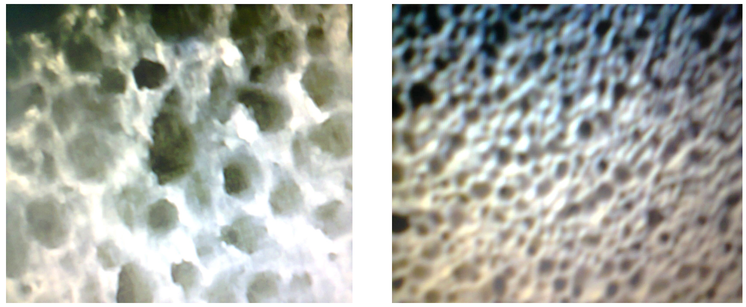
Main physical, thermal, mechanical and microstructural characteristics of samples

Variant	Bulk density (kg/m^3)	Porosity (%)	Thermal conductivity (W/m·K)	Compressive strength (MPa)	Water absorption (vol. %)	Pore size (mm)
1	530	74.7	0.177	1.2	3.7	0.4-4.5
2	552	73.7	0.161	1.6	3.5	0.7-2.6
3	586	72.1	0.129	1.9	3.7	0.6-2.2
4	600	71.4	0.184	2.2	3.9	0.4-0.8

The main objective of the experiment was to manufacture a light weight gypsum-based material with a relatively low bulk density for this material type. From the data of Table 4, a range of this physical feature values between 530-600 kg/m^3 represents the achievement of the objective. The material porosity had normal values between 71.4-74.7 %. The structural inhomogeneity of the samples made in variants 1 and 2 had a negative influence on the thermal conductivity, which at the low density values should have reached its lowest values. However, the thermal conductivity of variants 1 and 2 was higher than theoretically (0.177 and 0.161 W/m·K, respectively). According to the authors' forecast, the compressive strength of variant 4 (the densest from a structural point of view) had a maximum value (2.2 MPa), the other variants following a decreasing trend up to variant 1 (1.2 MPa). The water absorption was identified to be within a normal range (between 3.5-3.9 vol. %). The characteristics of gypsum-based material are close to the characteristics of aerated autoclaved concrete (AAC) [20], as also observed by the authors [2], this material being suitable for light weight boards and blocks, fire-resistant plasters and thermal insulation plasters. The investigation of the microstructural characteristics of the samples was performed based on the images presented in Figure 3.



a b



c d 5 mm

Fig. 3. Microstructural characteristics of the gypsum-based material samples
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

The images in Figure 3 allowed to determine the pore size of the four analyzed samples. The results, also included in Table 4, are: 0.4-4.5 mm (variant 1), 0.7-2.6 mm (variant 2), 0.6-2.2 mm (variant 3) and 0.4-0.8 mm (variant 4). Obviously, the most homogeneous pore distribution belongs to variant 4 and the most inhomogeneous belongs to variant 1.

The aerated autoclaved concrete (AAC) considered as a reference for the gypsum-based material is a light weight concrete type, which does not contain aggregates like conventional concretes. It consists of sand (as a supplier of silica) and coal fly ash as raw materials and aluminum powder as a hydrogen supplier for foaming. Despite the energy consumption in autoclave, the total energy used in the manufacturing process is 50% lower than the energy consumption of Portland cement production [20] required in the common concrete. So, like AAC, the gypsum-based material is an energy-efficient product. Unlike the conventional concrete, AAC can't be used as a finish. Being much more porous (pore size between 50-500 μm, compared to a maximum of 5 μm in conventional concrete), an exterior cladding is required, so that the moisture is not absorbed. The method of producing fine aluminum powder adopted by the authors in the current work by microwave melting of aluminum waste with nitrogen is also an energy-efficient method.

4. Conclusions

A light weight material, whose use in building construction is similar to that of aerated autoclaved concrete, but whose cumulative energy consumption is significantly lower, was experimentally small scale-manufactured in the Romanian company Daily Sourcing & Research. This material, based on gypsum, was performed of calcined gypsum ($CaSO_4 \cdot 0.5H_2O$) available on the market, finely ground (above 70 %) together with coal fly ash (3.4-5.1 %), an industrial by-product of thermal power stations. The raw material expansion occurs at the ambient temperature and is based on the corrosion reaction of a fine powder aluminum in aqueous solution of $Ca(OH)_2$, that releases hydrogen gas. The hydrogen forms bubbles with controllable size through the perlite addition (up to 10 %). The aqueous solution of carboxymethyl cellulose (2 %) acts as a foam stabilizer. The gypsum-based material produced in this experiment had the following characteristics: Bulk density between 530-600 kg/m^3, porosity between 71.4-74.7 %, thermal conductivity in the range 0.129-0.184 W/m·K, compressive strength between 1.2-2.2 MPa and water absorption below 3.9 vol. %. The pore size had the lowest values (between 0.4-0.8 mm) in the case of variant 4 (with the highest perlite addition of 10 %) and reached the highest values (up to 4.5 mm) in the case of variant 1 (without perlite addition). The originality of the paper is mainly applying the own technique of preparing aluminum powder by spraying the melted aluminum waste with concentrated nitrogen jets in a closed enclosure and also in adopting new additives in the manufacturing recipe of the final product.

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