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LIGHT CELLULAR GYPSUM-BASED MATERIAL FOR CONSTRUCTION APPLICATIONS

BY

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Abstract. The procedure of preparing a light gypsum-based material using aluminium powder activated in aqueous $Ca(OH)_2$ solution was tested in this work. The aluminium was recycled from post-consumer soft drink packaging waste, melted under the action of microwaves and atomized into very fine particles by direct contact with concentrated nitrogen jets. Except for calcined gypsum as the main raw material, other additive materials were used: coal ash, vermiculite, polypropylene fibres, polystyrene beads, silica fume, and carboxymethyl cellulose. The process took place at room temperature. The obtained product had good thermal insulation properties (density between 527-595 $kg \cdot m^{-3}$ and heat conductivity in the range of 0.147-0.180 $W \cdot m^{-1} \cdot K^{-1}$) as well as satisfactory compression strength (between 1.2-2.0 MPa).

Keywords: gypsum, aluminium, $Ca(OH)_2$ solution, microwave, vermiculite.

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1. Introduction

Gypsum as a calcium sulfate dihydrate is a very old binder applied in construction having an age of about nine thousand years. Its ability as a fireproof material, excellent operability, and its relatively easy making have constituted the main qualities of gypsum properties used since ancient times. Only at the beginning of the 20^{th} century, the appreciation of this material as a setting retarder of Portland cement and due to its availability from the recycling of industrial waste significantly increased. Thus, the possibility of manufacturing lightweight materials based on gypsum became a certainty, the construction sector being the main application field of this material type (Vimmrova *et al.*, 2014).

In recent decades, the idea of applying cellular materials in building construction has been widely adopted. In particular, the Nordic countries (Sweden, Norway, Finland, Poland, Germany, etc.) use these materials as excellent methods of enveloping buildings through boards, low-dimension blocks, or monolithic pieces. The main product used is calcined gypsum ($CaSO_4 \cdot 0.5H_2O$) which supplementary has adequate ecological properties. However, the use of this material is quite limited (Il'ina and Rakov, 2016).

Recently, the idea of designing lightweight gypsum-based thermal insulation materials, with lower transportation costs, has gained ground in the perspective of their application for buildings (Doleželova *et al.,* 2016).

A technique to reduce the material density is the use of lightweight fillers. The most commonly used inorganic fillers are perlite (an amorphous volcanic material) and vermiculite (a silicate mineral), commercially available products with bulk density within the limits of 300-800 $kg \cdot m^{-3}$. According to (Demir and Baspinar Serhat, 2008), an addition of 5-10 % perlite in the mixture containing carbon ash, lime, gypsum, and a small amount of silica fume allowed to obtain a product with bulk density of 730 $kg \cdot m^{-3}$ and compression resistance of 2.3 MPa. The results of the experiment using vermiculite (20%) into a mix with polypropylene fibre were shown in (Gencel *et al.*, 2014), indicating the decrease of bulk density by 10% and the heat conductivity decrease by 30%. Also, the compressive strength was reduced by up to 30 %.

Polymeric fillers have also been experimentally tried in gypsum-based materials. By adopting polystyrene beads (2%) and polypropylene fibre (2%), the bulk density was decreased by 50% compared to pure gypsum and the tensile resistance increased by 23% (Sayil and Gurdal, 1999).

The aim of the research presented in (Yavas *et al.*, 2022) was to evaluate the ability of a gypsum-based self-cleaning composite material for buildings regarding both the self-cleaning capacity and the technical characteristics (physical, thermal, acoustic, and mechanical) of this composite. The results showed that nano-sized TiO_2 added to the composite structure in different proportions between 0-20% by replacing the anhydrite binder improved the thermal and acoustic properties. Also, the compression resistance improved for proportions of TiO_2 up to 2.5%.

Several polymer materials can be used as a light filler in gypsum-based materials. In the work (Gutierrez-Gonzales *et al*., 2012a), excellent results have been obtained by using industrially produced polyamide powder waste in laser sintering processes. Using a ratio of polyamide powder to calcined gypsum of 4:1, a material with the density of around 750 $kg \cdot m^{-3}$ was produced.

Foam from granulated polyurethane waste from the automotive and construction industries was tested in (Gutierrez-Gonzales *et al*., 2012b). The foam was introduced into the gypsum mass in a ratio of 4:1. The obtained material had a bulk density between 500-1300 $kg \cdot m^{-3}$, but the compression resistance corresponding to the specimen with the lowest density was very low. Using a ratio of 1:1, the sample had an acceptable strength for construction applications.

Another material tried as a filler in gypsum-based materials was ground rubber from residual tyres. The amount of rubber was 5% and technical difficulties were reported in obtaining a uniform distribution of rubber particles in gypsum plaster (Serna *et al*., 2012).

Other technical solutions applied to obtain gypsum-based materials were the addition of natural plant fillers (sawdust, chopped straw, etc.) or animal fillers (chicken feather, cowhide, hoof, etc.) to the gypsum mass (Doleželova *et al.*, 2016).

Another experiment consisting in the use of cork granules (grain size under 12 mm) was reported in (Hernandez-Olivarez *et al*., 1999). Under the conditions of adopting 20% cork filler, bulk density of the gypsum-based product was about 800 $kg \cdot m^{-3}$ and the compression resistance had acceptable values (about 5 MPa) for construction applications. With the supplementary addition of 2% glass fibres, the tensile resistance increased two times.

In general, known techniques for producing a porous material include either chemical reactions releasing gases with foaming effect or surface-active substances (surfactants), both contributing to reduce the density of gypsum-based materials. Of the chemical reactions mentioned above, the most common is that involves a carbonate ($CaCO_3$, $NaHCO_3$ or NH_4HCO_3) reacting with an acidic compound ($Al_2(SO_4)_3$, H_2SO_4, $B(OH)_3$), the effect being the release of carbon dioxide (Doleželova *et al.*, 2016).

The industrial production of autoclaved aerated concrete currently uses the hydrogen-release foaming procedure through the chemical reaction between a fine aluminum powder and an aqueous solution of $Ca(OH)_2$. It has been found that this reaction leads to low bulk densities, but at the same time, to quite weak compression resistance (under 1 MPa). According to (Vimmrova *et al.*, 2011), the addition of a lightweight aggregate (such as perlite) improves the porous structure by reducing the cell size and homogenizing it.

The work (Paunescu *et al*., 2022) performed by authors of the current paper presented a procedure for made a light weight gypsum-based material. The chosen process was borrowed from the field of autoclaved aerated concrete manufacturing, operating on the principle of hydrogen release as a foaming gas following the corrosion reaction of powder aluminum in aqueous $Ca(OH)_2$ solution. An original technique was used to produce fine aluminum powder by atomizing the metal molten with concentrated nitrogen jets. Recycled aluminum wastes from post-consumer soft drink packaging were used and their melting was performed through unconventional microwave heating. The final product had a density in the range of 530-600 $kg \cdot m^{-3}$ and a compression resistance between 1.2-2.2 MPa, with low preparing energy costs.

Another making procedure of light gypsum material expanded with stone dust and acid agent was tested and presented in (Nazmunnahar and Vimmrova, 2015). The liquid acid agent was made from aluminum sulfate ($Al_2(SO_4)_3$) and citric agent dissolved into water as a foaming agent, while residual stone dust was chosen as a $CaCO_3$ source. Calcium sulfate hemihydrate or calcined gypsum ($CaSO_4 \cdot 0.5H_2O$) was selected as a gypsum binder in ratios of 80-95%. The residual stone dust was recycled from mechanical processing (cutting and polishing) of stone and has contained 50% marble and 50% granite. The mixture in form of slurry was dried and ground at particle size under 100 µm.

Recently, a composite based on chemically foamed phospho-gypsum with hydrogen peroxide (H_2O_2) in the range of 0.5-2.5% was experimentally produced (Zhang *et al.*, 2023). Growing the proportion of H_2O_2 caused higher porosity and implicitly, decreased mechanical resistance and waterproof ability. Thermal and acoustic insulation properties were improved. An addition of polyvinyl alcohol fibres led to significant growing of mechanical strength and waterproofing. Calcium stearate (15-25%) was chosen as a foam stabilizer. The final product manufactured with 1.5% H_2O_2, 1% reinforcing fibres, and 20% calcium stearate had a density of 826 $kg \cdot m^{-3}$, compression resistance of 5.1 MPa, and heat conductivity of 0.221 $W \cdot m^{-1} \cdot K^{-1}$. The results were considered promising, the use of phospho-gypsum requiring further investigations regarding long-term durability as well as in different environmental conditions.

The current work presented below aimed to improve the properties of cellular gypsum-based material by comparing with previous experimental results. One of the important technical solutions adopted in this experiment was the choice of a light inorganic filler consisting of vermiculite together with polypropylene fibres as well as polystyrene beads. This solution, which replaces perlite used in the own experiment of 2022 (Paunescu *et al.*, 2022), allows a sharp decrease of the density of gypsum-based material by over 40% compared to the pure gypsum density (around 1000 $kg \cdot m^{-3}$), according to (Ciemnika *et al.*, 2019). On the other hand, the original method of processing aluminum powder from recycled packaging waste of this metal, melted by unconventional microwave heating technique and atomized with fast nitrogen jets, was also adopted in this experiment.

2. Materials and Methods

The list of materials that make up the starting mixture includes calcined gypsum ($CaSO_4 \cdot 0.5H_2O$), hydrated lime ($Ca(OH)_2$), coal ash, vermiculite, polypropylene fibres, polystyrene beads, silica fume, carboxymethyl cellulose, and aluminum powder.

Calcined gypsum as the principal raw material is the result of dehydrating gypsum into plaster through the heating process of gypsum for evaporating the crystalline water (Ridge and Boell, 1962).

Hydrated lime ($Ca(OH)_2$) in form of aqueous solution is used as active medium for developing the corrosion process of aluminum powder with releasing the hydrogen gas.

Coal ash was procured as a by-product of the energy producing industry from the Paroseni thermal power plant (Romania) containing 46.5% SiO_2, 23.8% Al_2O_3, 10.2% (Na_2O + K_2O), 3.2% MgO, 3.6% CaO, 8.5% Fe_2O_3. The initial ash granulation was under 250 µm, requiring ball milling to reduce its particle size below 80 µm.

Vermiculite is a hydrated silicate mineral, with the chemical formula $(Mg, Fe, Al)_3((Al, Si)_4 O_{10}) (OH)_2 \cdot 4H_2O$ and the composition containing 36.3 SiO_2, 14.6% MgO, 14.8% Al_2O_3, 4.8% K_2O, 12.4% Fe_2O_3, 2.7% TiO_2, 4.2% CaO, 0.3% Na_2O, 9.1% LOI. In its natural state, vermiculite is a flat and shiny mineral roughly similar to mica. It is available in mines in several world countries such as the United States, Russia, South Africa, China, and Brazil and is commercially capitalized. The interest exhibited on vermiculite is due to its ability as an insulating material as well as a coating material. By heating, this mineral expands enough to create a network of air voids, resulting a light, fireproof and chemical resistant product (Vermiculite, 2023). In the current experiment, vermiculite originating from China is mechanically processed through fine grinding at the particle size less than 50 µm.

Polypropylene fibres with lengths of 12 mm originated in United Kingdom, produced through chain polymerization of propene monomer, were utilized in this experiment. The fibres are stable, heat resistant, hard, and have a long lifespan. This fibre type is produced in large quantities in the world, being the fourth most widely known artificial fibre after polyesters, nylon, and acrylics. Its field of application is exclusively industrial (automobile industry, construction as reinforcement fibres, textiles, packaging, sanitary products, sports, etc.) (Menyhard *et al.*, 2020).

Polystyrene beads are commonly used as light aggregates in making process of cellular gypsum-based materials. This method reduces the product density and improves its thermal and acoustic insulation abilities (Doleželova *et al.*, 2016). For this purpose, low weight proportions (about 2%) of commercially available polystyrene beads were adopted to complete the preparing material mixture.

A product from the micro-material category (silica fume) with extremely fine grain size (below 1 μm) and high SiO_2 content (85-99%) as well as other oxides in very low proportions (Al_2O_3, CaO, Fe_2O_3, Na_2O, K_2O) in amorphous and non-crystalline state, originated in Poland, was adopted to increase the compression strength of the porous product. Silica fume is known as an effective material with pozzolanic properties, resulted as a by-product during the silicon industrial making (Silica fume, 2024).

Carboxymethyl cellulose $[C_6H_7O_2(OH)_x(OCH_2COONa)_y]_n$, that easily turns into aqueous solution and acts as a froth stabilizer, was chosen as an additive material in low proportions (around 2%) within this experiment. The chemical formula shown above represents a long chain of glucose units with carboxymethyl groups attached to each hydroxyl group, "n" indicating the number of similar glucose units repeated in the chain (Ergun *et al*., 2016; Zhivkov, 2013).

As mentioned in the work (Paunescu *et al*., 2022), aluminum powder with very fine grain size (under 10 μm) was made under experimental conditions by a research team from Daily Sourcing & Research company (Romania), some of whom are authors of the current paper. The adopted procedure used aluminum waste recovered from post-consumer soft drink packaging. The metal melting was performed through the original method of microwave heating and the melt atomization was carried out by its contact with concentric fast jets of nitrogen.

Among the methods mentioned above in this work aiming at the formation of a cellular structure in the gypsum-based material, the technique of releasing hydrogen gas through the corrosion process of aluminum powder in the aqueous $Ca(OH)_2$ solution at ambient temperature was adopted.

The chemical reaction that characterizes the mentioned hydrogen release process is shown below:

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

The $Ca_3Al_2(OH)_{12}$ phase known as katoide penetrates the molten mass of gypsum, while hydrogen gas is released. According to (Paunescu *et al*., 2022; Kaneshira *et al*., 2013), the general reaction (1) mechanism contains several successive steps, which occur at the separation area between the outer surface of aluminum particles and the aqueous activating solution.

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

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

$$Al(OH)_3 + OH^- \rightarrow [Al(OH)_4]^- \quad (4)$$

$$3Ca^{2+} + 2[Al(OH)_4]^- + 4OH^- \rightarrow Ca_3Al_2(OH)_{12} \quad (5)$$

The slurry processed into a cylindrical metal mould was continuously mechanical stirred up to the slurry expansion begins to appear.

Investigation methods of gypsum-based specimen characteristics were the following. Archimedes' principle was applied to determine the apparent density of cellular gypsum-based specimens. Utilizing the ASTM C642-97 standard, the apparent porosity was calculated by dividing the difference between wet and dry weight by the difference between wet weight and suspended weight of the sample. Heat conductivity was determined at room temperature using the HFM448 Lambda heat-flow-meter (SR EN 1946-3:2004). The compression strength measuring was made utilizing a universal testing machine. The specimens were compressed at 1.3 $mm \cdot min^{-1}$ (ASTM D695) and the compression resistance was determined at 10% compression (ASTM D1621-16). The microstructural aspect of froths could be examined with ASONA 100X Zoom Smartphone Microscope. Water uptake was measured through maintaining cellular gypsum-based specimens in humidity chamber at 85% humidity for 30 days according to ASTM C272/C272M-18.

3. Results and Discussion

The adopted experimental variants including the above-mentioned materials and distilled water added to the mixture are shown in Table 1.

Table 1
Material composition of experimental variants

Composition (wt. %	Variant 1	Variant 2	Variant 3	Variant 4
Calcined gypsum	71.4	69.9	68.2	66.6
Hydrated lime	9.7	10.0	10.3	10.6
Coal ash	3.5	4.0	4.5	5.0
Vermiculite	5.5	6.0	6.5	7.0
Polypropylene fibres	2.5	2.5	2.5	2.5
Polystyrene beads	2.0	2.0	2.0	2.0
Silica fume	0.6	0.6	0.8	1.0
Carboxymethyl cellulose	1.9	2.0	2.1	2.1
Aluminum powder	2.9	3.0	3.1	3.2
Water addition	24.0	26.0	28.0	30.0

The composition of variants in Table 1 was slightly modified mainly aiming at improving the insulating properties of the gypsum-based material. Thus, the proportion of hydrated lime ($Ca(OH)_2$) was increased from 9.7 to 10.6%. An increase in the proportion of vermiculite from 5.5 to 7.0% took into account the insulating ability of this material. Carboxymethyl cellulose had slightly increasing values (between 1.9-2.1%) due to its beneficial properties as a foam stabilizer. Silica fume was used in increasing proportions (from 0.6 to

1.0%) due to its ability to improve the material strength. Aluminum powder was also increased (from 2.9 to 3.2%), having a major role in the expansion process of the gypsum-based material.

Images of the physical aspect of the four experimentally made specimens are exposed in Fig. 1.

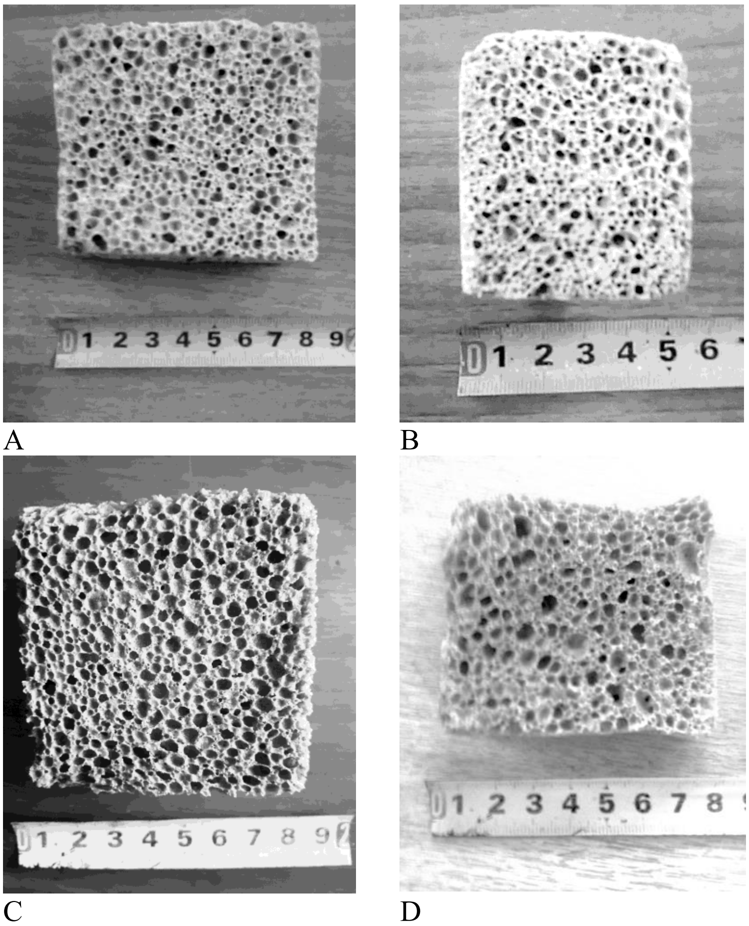

Fig. 1 – Aspect of experimentally made specimens
A – variant 1; B – variant 2; C – variant 3; D – variant 4.

The dry material mixture was determined by weighing at 235 g kept constant in all experimental variants, while the wet mixture due to the addition of distilled water varied between 259-265 g. The process duration was short, between 7-12 min in ascending order from sample A to sample D.

Determining the physio-mechanical, heat, and morphological characteristics of samples, presented in Table 2, was performed in accordance with investigation methods specified above.

Table 2
Physio-mechanical, heat and morphological features of samples

Feature	Variant 1	Variant 2	Variant 3	Variant 4
Apparent density (kg·m^{-3})	595	581	550	527
Apparent porosity (%)	71.6	72.5	73.9	74.9
Heat conductivity (W·m^{-1}·K^{-1})	0.180	0.170	0.159	0.147
Compression strength (MPa)	2.0	1.8	1.5	1.2
Water uptake (vol. %)	3.3	3.0	3.1	2.9
Cell size (mm)	0.3-0.8	0.6-1.0	0.7-1.8	0.8-2.0

Analyzing the data exposed in Table 2, good performances in terms of insulation are observed, with the apparent density values ranging between 527-597 kg·m^{-3}, decreasing from sample A to sample D, and in a similar trend also in the case of heat conductivity. The conductivity values are within the limits of 0.147-0.180 W·m^{-1}·K^{-1}. The apparent porosity is sufficiently high with increasing values in the range of 71.6-74.9%. The compression strength follows a normal decreasing trend from 2.0 MPa corresponding to specimen A, to 1.2 MPa in the case of specimen D. Regarding the water uptake, the results fall within normal limits for this type of cellular materials (2.9-3.3 vol. %).

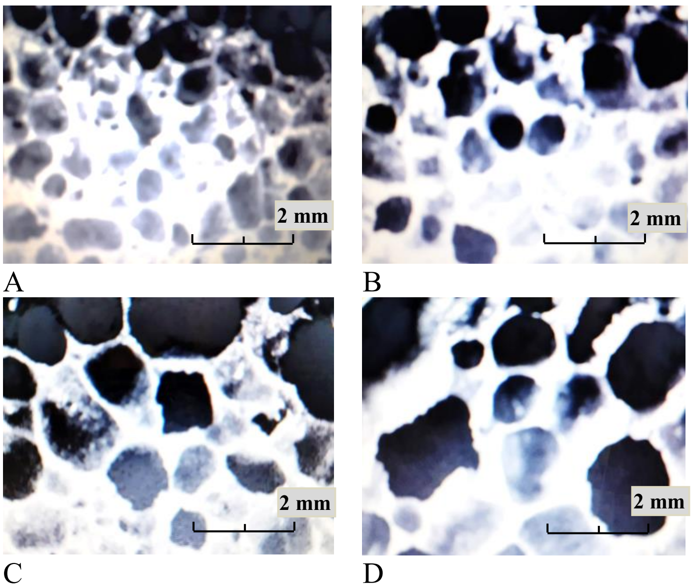

Fig. 2 – Microstructural appearance of specimens
A – variant 1; B – variant 2; C – variant 3; D – variant 4.

Investigating microstructure of the four specimens of cellular gypsum-based materials allowed the evolution identification of its appearance due to the modification of each sample composition. The microstructural appearance of specimens is shown in Fig. 2.

The size of cells composing each microstructure progressively increased from 0.3-0.8 mm corresponding to specimen A, to 0.8-2.0 mm in the case of specimen D. The dimensional ranges of the cells in all microstructures shown in Fig. 2 are indicated in Table 2.

The comparison of results obtained in this experiment with results of other similar researches reported in the literature confirmed the correctness of the adopted technical solution. Thus, the good correlation between the low values of density and heat conductivity as the main insulating properties of the material, on the one hand, and the satisfactory values of compression strength, on the other hand, certifies the process based on the corrosion of aluminum powder in the aqueous medium of $Ca(OH)_2$.

Compared to the manufacturing recipe adopted by the same research team performing the current work, perlite as filler was substituted with vermiculite, polypropylene fibres, and polystyrene beads, mainly favoring the thermal insulation properties. In both experiments, aluminum powder produced from recycled commercial waste by melting under the influence of microwaves and then atomized with nitrogen jets, was maintained as an agent providing pores in the gypsum mass.

The experimental results were generally similar, with a small qualitative advantage in favour of the current experiment, characterized by slightly better insulating properties.

4. Conclusions

The work aimed at producing a light cellular gypsum-based material for the construction sector, made using a method borrowed from the aerated autoclaved concrete manufacture, but with much lower energy consumption. The gypsum expansion agent chosen was aluminum powder placed in contact with an active aqueous medium of hydrated lime-$Ca(OH)_2$. The materials that composed the starting material mixture were calcined gypsum, hydrated lime, coal ash, vermiculite, polypropylene fibres, polystyrene beads, silica fume, carboxymethyl cellulose, and aluminum powder in various weight proportions. The making process occurred at ambient temperature, avoiding the high energy consumption required to reach the heating temperatures specific to conventional foaming processes through sintering. The technical characteristics of gypsum-based products showed very good thermal insulation properties (low density and thermal conductivity values) as well as acceptable compression resistance values, suitable for applications in the construction sector.

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MATERIAL CELULAR UȘOR PE BAZĂ DE GIPS PENTRU APLICAȚII ÎN CONSTRUCȚII

(Rezumat)

Procedeul pregătirii unui material ușor pe bază de ghips utilizând pulbere de aluminiu activată în soluție apoasă de $Ca(OH)_2$ a fost testat în această lucrare. Aluminiul a fost reciclat din deșeuri de ambalaje după consum de băuturi răcoritoare, topit sub

acțiunea microundelor şi atomizat în particule foarte fine prin contactul direct cu jeturi concentrate de azot. Exceptând ghipsul calcinat ca principală materie primă, alte materiale de adaos au fost utilizate: cenușă de cărbune, vermiculit, fibre de polipropilenă, mărgele de polistiren, fum de silice și carboximetil-celuloză. Procesul a avut loc la temperatura camerei. Produsul obținut a avut bune proprietăți termoizolante (densitate între 527-595 $kg\cdot m^{-3}$ și conductivitate termică în intervalul 0,147-0,180 $W\cdot m^{-1}\cdot K^{-1}$), precum și o satisfăcătoare rezistență la compresiune (între 1,2-2,0 MPa).