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MICROWAVE HEAT TREATMENT OF WASTES (CLAY, GLASS AND COAL ASH) TO MANUFACTURE A HIGH MECHANICAL STRENGTH CELLULAR AGGREGATE

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ABSTRACT: *The paper presents experimental results obtained in the manufacturing process of a lightweight aggregate using a finely ground mixture of wastes (clay, glass and coal ash) expanded with silicon carbide as a foaming agent. The originality of the work consists in the use of microwave radiation in a direct heating process, the energy efficiency being remarkable (0.58-0.85 kWh/kg), unlike all the manufacturing processes known in the world, industrial or experimental on a small scale. Given that glass alone is not suitable for foaming by direct microwave heating, but the favorable effect of adding glass to clay on the physical and mechanical characteristics of lightweight (expanded) clay aggregate is known, the experiments aimed to determine the maximum allowable proportion of glass in clay without affecting the microstructure of the material. The characteristics of the experimental samples were almost similar to those of industrially manufactured products by conventional methods (0.60-0.69 g/cm^3 the apparent density, 0.100-0.116 W/m·K the thermal conductivity, 7.8-8.3 MPa the compression strength, 9.3-17.5% the water absorption).*

KEYWORDS: direct microwave heating; lightweight aggregate; clay waste; glass waste; high energy efficiency.

1. INTRODUCTION

Microwaves represent a form of electromagnetic radiation with frequencies between 300 MHz-300 GHz (corresponding to wavelengths from 1 m to 1 mm). They have been known since the middle of the 20th century and were used at the beginning for military purposes. The microwave ovens with a frequency of 2.45 GHz became common in the household for food preparation in the late 1970s.

Very wide fields of modern technologies (microwave radio relay networks, wireless networks, radar, satellite communications, particle accelerators, spectroscopy, cancer treatment, environmental remediation through treatment, destruction and immobilization of many hazardous wastes, etc.) are beneficiaries of microwave radiation [1, 2].

The microwave heating for the chemical industry began to be tested in laboratory and was accepted in the scientific community following the papers published in 1986. Since that year, the microwave heating has become an important technique in chemical synthesis and processing, especially in the case of organic materials and solution-phase reactions. By the end of the last century, some applications of the microwave heating in chemical processing had progressed from laboratory-scale to pilot-scale or industry-scale, such as materials drying, ceramics sintering and waste vitrifying. However, the industrial application of solid state processing and synthesis techniques at high temperatures has not yet been achieved [1].

The microwave heating is radically different from the conventional heating. The electromagnetic wave field is absorbed by the material subjected to heating, which must meet the condition that at least one of its components is microwave susceptible [3]. The heating is initiated in the core of the material by converting the wave energy into heat and propagates to its peripheral areas [4], exactly the opposite of the conventional heating. Thus, the material itself is a source of energy, the process takes place volumetrically and for this reason can be very fast [1, 5]. The microwave heating has the advantage to be selective, avoiding the unnecessary heating of walls and other massive components of the oven. In this way, the energy efficiency of the process is much higher compared to the conventional heating, where a large amount of energy is initially transferred to these components [1]. All these favorable characteristics of the microwave heating are valid only in the case of the direct heating of material or a mixed heating which partially includes the direct heating.

The heating to high temperature (750-1100 ºC) for sintering and foaming some silicate waste (mainly glass) is commonly performed by conventional techniques [6, 7]. Porous products industrially manufactured by this type of techniques are known, the main world producers being Misapor, Pittsburgh Corning, Geocell, etc. There is a wide range of foamed products, generally with low densities (0.15-0.60 g/cm3), low thermal conductivities (0.04-0.1 W/m·K) and relatively high mechanical strengths (maximum 6-7 MPa) or, in the case of light weight products, at an acceptable level (1-1.2 MPa) [6]. The literature does not provide data on the specific energy consumption of manufacturing processes either in the case of industrial processes or in the case of small-scale experiments. Only a market study [8] indicates an average value of the specific consumption at the level of the entire Misapor company (0.80-0.85 kWh/kg) as well as the average total specific energy consumption of the Pittsburgh Corning company of 4.24 kWh/kg, but this consumption also embeds the material processing, including the melting of glass waste to correct its composition.

Worldwide, the microwave energy application in sintering-foaming processes of silicate waste is extremely low used. The industrial manufacturers of glass foam are not interested in this unconventional technique, although the literature recognizes its advantages compared to the conventional heating methods [9]. The explanation should be sought in a work published in 1997 [10], which shows that the main components of glass (SiO_2, Al_2O_3) used as raw material in the manufacture of glass foam are not microwave susceptible, their dielectric properties improving significantly only at about 500 ºC. According to this theory, the current industrial furnaces should use two different energy sources, one conventional up to 500 ºC and another unconventional from 500 ºC up to the final foaming temperature of 750-1100 ºC, which could be unprofitable.

A team of researchers from the Romanian company Daily Sourcing & Research Bucharest did not experimentally confirmed this conclusion showing that, due to the presence in the composition of commercial glass (which is the main raw material) or in the additives composition, of some chemical compounds (Fe_2O_3, Cr_2O_3, SiC, etc.) that are microwave susceptible, the sintering-foaming process can occur with maximum energy efficiency starting from room temperature [11]. Moreover, other works in the literature have theoretically confirmed these experimental results [3, 12].

The same team of researchers mentioned above experimentally found [11, 13] that glass waste is not suitable for direct microwave heating because it causes severe destruction of the internal structure of glass at the specific temperature of sintering-foaming process. The solution adopted was a mixed heating (partially direct, partially indirect) of the glass-based mixtures by placing on the path of the waves a ceramic screen of a high microwave susceptible material, that partially absorbed the microwave field and diminished the effect of the direct impact of microwave with the material subjected to heating. Forwards, numerous experiments have been successfully performed by this type of microwave heating [14-16].

The authors of the present paper were able to experimentally manufacture a lightweight aggregate from recycled masonry rubble using silicon carbide as a foaming agent and coal ash by the method of direct microwave heating [17]. The masonry rubble composed of old clay brick, concrete and cement mortar in weight ratios 48/40/12 was crushed and ground to a grain size below 300 μm and was mixed in proportions between 85.6-90.8% with coal ash (4-9%) and SiC (3.5-5.5%) using a water addition of 18% to facilitate the raw material pressing. The pressed sample, thermally protected with thick layers of high quality ceramic fibre, was sintered in the microwave oven at 1168-1185 ºC with very high heating rate (35.3-35.9 ºC/min) and with a specific energy consumption around 1 kWh/kg resulting foamed products with an apparent density between 0.75-0.98 g/cm3, porosity between 60.8-70%, thermal conductivity in the range 0.123-0.140 W/m·K, high compressive strength (6-7.3 MPa) and water absorption between 12.9-14.6%. The physical and mechanical properties of the aggregate were almost similar to those of lightweight aggregates manufactured industrially by conventional heating techniques. The most known lightweight aggregate on the world market is LECA-light expanded clay aggregate produced at about 1200 ºC in rotary kiln. Its density varies between 0.38-0.71 g/cm3, the thermal conductivity has values between 0.09-0.101 W/m·K and the water absorption is about 18% for aggregates with dimensions below 25 mm [18].

A similar approach to the same research objective regarding the use of clay waste mixed with coal ash (in slightly variable proportions, but around the 50/50 ratio) in a process of direct microwave heating is in the process of publishing in the journal Constructii (Paunescu, B.V. and Paunescu, L. “Lightweight high mechanical strength aggregate made by microwave irradiation”). The foaming agent was silicon carbide in weight proportions between 2-3.4%. The sintering process temperature was between 1160-1180 ºC, the heating rate being at a very high level of over 35 ºC/min. Due to a slightly higher raw material load, the specific energy consumption of the process decreased to 0.83-0.89 kWh/kg. The lightweight aggregate obtained after foaming the powder mixture had physical and mechanical characteristics comparable to those of the sintering-foaming process of the mixture containing predominantly masonry rubble (85.6-90.8%) and much less coal ash (4-9%). The apparent density had values between 0.70-0.80 g/cm3, the thermal conductivity was in the range 0.129-0.148 W/m·K and the compressive strength between 6.8-8 MPa. From a morphological point of view, the aggregate had a homogeneous porous structure with pore sizes from 0.9-3.5 mm to 0.6-2.5 mm, compared to a less homogeneous structure (pore size between 1-4 mm) when using masonry rubble [17].

The current work aims to manufacture a cellular aggregate with high mechanical strength of clay waste from recycling bricks of the building demolition mixed with different proportions of glass waste, coal ash and silicon carbide as a foaming agent in a direct microwave heating.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in the experiments were: red clay waste of the recycling of old clay bricks from the building demolition, container soda-lime glass waste, coal ash and silicon carbide.

According to the literature, the ingredients of the clay brick are: SiO_2 (55%), Al_2O_3 (30%), Fe_2O_3 (8%), MgO (5%), CaO (1%) and organic matter (1%) [19]. The recycled clay brick was crushed and ground in a ball mill at a grain size below 100 µm.

The glass waste was a mixture of colorless, green and amber glass with an average chemical composition which included SiO_2 (73.19%), Al_2O_3 (1.44%), Na_2O (12.85%), K_2O (0.75%), CaO (10.20%), MgO (1.34%), Fe_2O_3 (0.06%), SO_3 (0.19%), TiO_2 (0.04%) and BaO (0.03%) [20]. The glass waste was crushed and ground in a ball mill at a grain size below 63 µm.

The coal ash is an industrial waste produced by thermal power stations, the amount generated worldwide reaching 2 billion tons in 2020 [21]. The waste used in experiments was provided by Paroseni thermal power station having the following chemical composition: SiO_2 (46.5%), Al_2O_3 (23.7%), CaO (7.9%), MgO (3.2%), Na_2O (6.0%), K_2O (4.1%) and Fe_2O_3 (8.6%), determined by X-ray fluorescence. The grain size of coal ash was below 32 µm.

The silicon carbide was used at a grain size below 32 µm as purchased from the market.

According to the literature [12, 22], some components (Na_2O, K_2O) of materials subjected to microwave heating facilitate the absorption of these waves. Thus, the soda-lime glass waste contains 13.6% (Na_2O + K_2O) and coal ash has in its composition 10.1%. On the other hand, Fe_2O_3 contents (8% in clay, 8.6% in coal ash and 0.06% in glass), to which is added the presence of SiC (5%) in the powder mixture, which are microwave susceptible compounds to room temperature, allow an efficient heating starting from this temperature [3], although the raw material contains significant proportions of SiO_2 and Al_2O_3, that are microwave transparent at low temperature.

2.2 Methods

As mentioned above, the maximum energy efficiency of sintering-foaming processes can be reached by the direct microwave heating of the material, being much higher compared to that of processes that use conventional heating techniques. It has also been shown that glass waste is not suitable for direct microwave heating according to previous tests. The research that was the basis of the current paper aimed the possibility of mixing (and in what proportion) the clay waste with soda-lime glass waste in the conditions of processing the mixture in the microwave field without negatively affecting the microstructure of the material.

It is known from the literature that the addition of soda-lime glass waste between 20-50 wt.% contributes to the improvement of some physical and mechanical characteristics of the clay aggregate by reducing the bulk density, reducing the water absorption and increasing the compressive strength by a conventional heating process. Also, by using the addition of glass it is possible to decrease the softening point of the mixture, reducing the foaming temperature and implicitly, decreasing the specific energy consumption [23].

Given the results of preliminary tests, the authors adopted four experimental variants of material mixtures, using red clay waste and container soda-lime glass waste as raw material, silicon carbide as a foaming agent and coal ash as a mineral additive. The weight proportions of the three categories of materials were kept constant: 86% raw material, 5% SiC and 9% coal ash. The variables were the weight proportions of clay and glass, whose ratio had values between 1/32.1 and 1/4.55. It was also necessary to add additional water (between 20-28 wt.%) to facilitate the raw material pressing, the highest value corresponding to the maximum proportion of clay. The composition of the four experimental variants is shown in Table 1.

Table 1. Composition of the experimental variants

Var.	Clay waste wt.%	Glass waste wt.%	Coal ash wt.%	SiC wt.%	Water addition wt.%
1	83.4	2.6	9.0	5.0	28.0
2	80.0	6.0	9.0	5.0	25.0
3	76.0	10.0	9.0	5.0	22.0
4	70.5	15.5	9.0	5.0	20.0

The experimental microwave equipment is a 0.8 kW-domestic microwave oven adapted for operation at high temperature (below 1200 ºC), in which the pressed powder mixture is freely deposited on a metal plate placed by means of a metal support on a thermal insulation bed from fibres ceramic mattresses. The pressed mixture is coated with thick layers of ceramic fibre, including its upper area. The oven is equipped with only a microwave generator whose waveguide is placed on one of its side walls. The process temperature control is performed with a radiation pyrometer mounted above the oven at about 400 mm. The upper wall of the oven has a hole with a diameter of 30 mm, through which the upper surface of the material can be viewed by the pyrometer. An overall image of the experimental microwave equipment is shown in Figure 1.

Figure 1. Overall image of the experimental microwave equipment

The mechanism of the foaming process is well known and is based on the incorporation of a foaming agent into the powder mixture of the raw material. By a thermal process at high temperatures, the foaming agent releases a gas in the softened mass of the material, most often through a decomposition or oxidation reaction. There should be a good correlation between the softening point and the temperature range in which the reaction that releases the gas occurs as well as an adequate value of the material viscosity, so that the gas is captured in the form of bubbles in its viscous mass. At the end of the process, by cooling, a structure is formed with numerous pores, generally evenly distributed in the mass of material, which acquires the appearance of a foam [6].

The silicon carbide (SiC) used in experiments is considered a very effective foaming agent capable of producing a foamed material with a very homogeneous cellular structure with controllable dimensions. Generally, the temperature range in which SiC is active is 950-1150 ºC. It reacts with oxygen in the oxidizing atmosphere of the furnace, releasing CO2 and CO according to the reactions below. The residual silicon oxide resulting from the reactions is incorporated into the mass of the material [6].

$$SiC + 2O_2 = SiO_2 + CO_2 \tag{1}$$

$$SiC + O_2 = SiO + CO \tag{2}$$

Using various silicate waste such as coal ash, metallurgical slag, filter dump and fly ash from waste incinerators, mud from metal hydrometallurgy, sludge as well as glass waste, the characteristics of the foaming process can be significant modified both in terms of the temperature range at which it occurs (increasing compared to the foaming process only of glass waste) and by the tendency of crystallization of the foamed material. Thus, a cellular glass-ceramic is formed by the controlled crystallization of silicate, a partially crystalline material with a fine microstructure, which also contains the vitreous phase [7].

2.3 Characterization of the samples

The cellular aggregate samples experimentally obtained by the sintering-foaming process of clay waste, glass waste and coal ash were characterized by traditional analysis methods. The main physical, mechanical and morphological characteristics were: apparent density, porosity, thermal conductivity, compressive strength, water absorption, crystallographic structure and microstructural configuration of the samples. The apparent density was measured by the gravimetric method [24]. The porosity was calculated by the comparison method between the porous sample density and the density of the same material type in compact state [25]. The thermal conductivity was determined by measuring the thermal flow that passes through a sample placed between two metal plates (one heated and the other cooled) [26]. The compressive strength was determined using an uniaxial press and the water absorption of the samples was measured by the traditional method of their water immersion (ASTM D 570). The X-ray diffraction (XRD) was used according to the standard EN 13925-2:2003 to determine the crystallographic structure of the samples. A X-ray diffractometer Bruker AXS D8 Advance with CuKα radiation was used. The porous microstructure was identified with a Smartphone Digital Microscope.

3. RESULTS AND DISCUSSION

3.1 Results

Generally, the lightweight aggregate is manufactured in the form of granules by a previous granulation process before sintering in the rotary kiln. Due to the low aggregate amount experimentally manufactured, the authors did not used granulated materials. The aggregates were broken at the end of the sintering-foaming process to can examined the products microstructure.

The main functional parameters of the foaming process of the powder mixtures, according to the four experimental variants in Table 1, are presented in Table 2.

Table 3 presents the physical and mechanical characteristics of samples.

Table 2. Main functional parameters of the foaming process

Variant	Dry/wet raw material g	Process temperature ºC	Heating time min	Average rate, ºC/min		Expansion of material volume %	Lightweight aggregate amount g	Specific energy consumption kWh/kg
				Heating	Cooling			
1	500/640	1150	32	35.3	6.3	150	484	0.85
2	500/625	1120	30	36.7	6.1	170	480	0.73
3	500/610	1085	27	39.4	6.4	190	483	0.65
4	500/600	1055	24	43.1	6.3	220	480	0.58

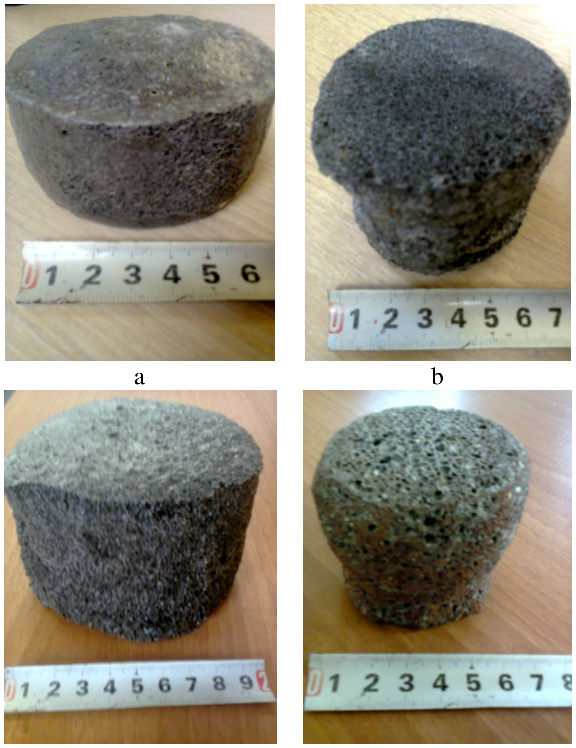
Table 3. Physical and mechanical characteristics of samples

Variant	Apparent density g/cm^3	Porosity %	Compressive strength MPa	Thermal conductivity W/m·K	Water absorption %	Pore size mm
1	0.69	68.6	7.8	0.116	17.5	0.1 – 0.3
2	0.65	70.5	8.3	0.109	15.0	0.3 – 0.5
3	0.63	71.4	8.2	0.104	11.6	0.4 – 0.6
4	0.60	72.7	8.0	0.100	9.3	0.4 – 0.7

Table 2 reveals that the replacement of a part of the clay waste with soda-lime glass waste reduces the final temperature of the foaming process from 1150 ºC (in the 2.6% glass variant) to 1055 ºC (in the 15.5% glass variant) due to decreasing the softening point of the raw material. Implicitly, the process duration is reduced from 32 to 24 min and also the value of the specific energy consumption reaches an excellent minimum level of 0.58 kWh/kg (in the variant with 15.5% glass). This specific consumption is very low being even below the level of consumptions achieved in industrial processes. The direct microwave heating of the mixture of clay waste, glass waste, coal ash and SiC allowed to reach very high heating rates (35.3-43.1 ºC/min), which in the conditions of conventional heating as well as indirect microwave heating would not be possible. On the other hand, it should be noted that the proportion of 15.5% glass waste in the powder mixture (i.e. 18% in the clay-glass mixture) was experimentally determined that the maximum permissible value at which the microstructure of the foam product was not affected. Above this limit, the destruction of the internal microstructure of the material was observed, being similar to that suffered by the glass waste used alone in direct microwave heating.

A significant improvement of the physical and mechanical characteristics of the samples manufactured with the addition of glass waste by direct microwave heating results from Table 3. Thus, the influence of glass waste on the values of the apparent density is obvious by their decrease below 0.69 g/cm^3. Implicitly, the porosity of the samples increased with increasing the proportion of glass (from 68.6 to 72.7%). Also, the thermal conductivity of the aggregate samples was considerably improved decreasing in the range 0.100-0.116 W/m·K. The compressive strength increased for all samples compared to those manufactured without the addition of glass having values between 7.8-8.3 MPa. The water absorption was significantly reduced from 17.5 up to 9.3% due to the use of glass waste as a partial replacement of clay waste.

Pictures of the lightweight aggregate samples are shown in Figure 2.

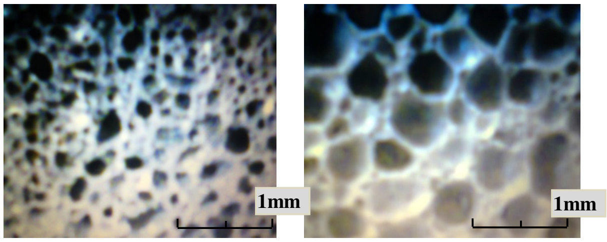


a b

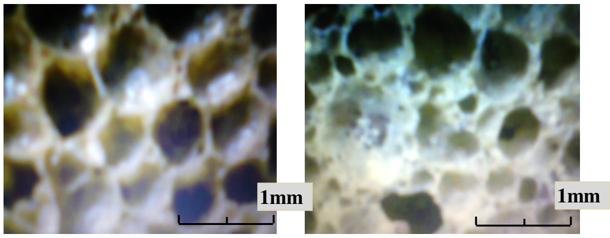
c d

Figure 2. Pictures of the lightweight aggregate samples
a – sample 1, heated at 1150 ºC; b – sample 2, heated at 1120 ºC; c – sample 3, heated at 1085 ºC; d – sample 4, heated at 1055 ºC.

Images of the samples microstructure are presented in Figure 3.



a b



c d

Figure 3. Images of the samples microstructure
a – sample 1, heated at 1150 ºC; b – sample 2, heated at 1120 ºC; c – sample 3, heated at 1085 ºC; d – sample 4, heated at 1055 ºC.

According to the pictures, sample 1 had a very fine microstructure with a pore size between 0.1-0.3 mm. Samples 2 and 3 had slightly larger pore sizes (0.3-0.5 mm and 0.4-0.6 mm, respectively) with a homogeneous distribution of them. Sample 4 with pore size between 0.4-0.7 mm also had a homogeneous distribution, but less remarkable compared to samples 2 and 3. The pore sizes corresponding to the four aggregate samples are centralized in Table 3.

The XRD analysis to identify the crystalline phases in the structure of the cellular aggregate samples after the heat treatment in the range 1055-1150 ºC revealed an only main phase represented by quartz and traces of cristobalite, feldspar and hematite.

3.2 Discussion

Information from the literature that the partial replacement of clay waste with glass waste allows to improve the characteristics of the foamed material as well as the own experimental finding that glass waste is not suitable for the direct microwave heating were the starting points of the research that was the basis of the current work.

The experiments started from very low proportions of glass waste (2.6 wt.%) introduced in the powder mixture, following mainly the microstructural integrity of the foamed material. The proportion of glass could be increased in stages, being performed the physical, mechanical and morphological characterization of each sample of lightweight aggregate. In this way, the maximum limit (15.5%) was determined until which the glass waste could be used in a mixture with clay and coal ash without negative microstructural effects.

The physical and mechanical effects were favorable: decreasing the apparent density, increasing the porosity, decreasing the thermal conductivity, increasing the compressive strength and decreasing the water absorption, i.e. all the characteristics of the lightweight aggregate mentioned above were improved.

The main achievement of the research was the significant reduction of the specific energy consumption for the experimental manufacture of the product (up to 0.58 kWh/kg), which, despite the experimental working conditions, was below the estimated level of consumption of industrial manufacturing processes (0.80- 0.85 kWh/kg) [8].

The originality of the research results consists in the use of microwave radiation as an unconventional, fast, "clean" and economical energy source, unlike the use of conventional energy forms practiced by both industrial producers and research teams in small-scale experiments.

4. CONCLUSION

The direct microwave heating, a high energy efficiency technique, was experimentally applied in the manufacturing process of a lightweight aggregate from clay waste, glass waste as a partial replacer of clay and coal ash.

The weight proportion of glass waste was successively increased from 2.6 up to 15.5% considering that the glass is not a suitable material for its direct microwave heating, according to previous tests.

The experimental results showed that the glass waste could be used in the sintering-foaming process without negative effects on the microstructure of the foamed material up to 15.5% and the presence of glass mixed with clay contributed to significant improvement of the product characteristics.

The apparent density had relatively low values (0.60-0.69 g/cm^3) as did the thermal conductivity (0.100-0.116 W/m·K). The compressive strength was high with values up to 8.3 MPa. The water absorption was reduced with the increase of glass ratio up to 9.3%. The pore size of the foamed products was very low for all the tested variants (below 0.7 mm), the microstructure being homogeneous. The characteristics of the experimentally manufactured lightweight aggregate are almost similar to those of the industrial products.

The main advantage of the aggregate manufacturing by direct microwave heating is the very low level of the specific energy consumption (minimum 0.58 kWh/kg), economical compared to the average consumption of industrial processes based on conventional heating techniques.

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