
POROUS MATERIAL FROM RECYCLED GLASS WASTE AS AN ALTERNATIVE TO EXISTING BUILDING MATERIALS

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ABSTRACT

Experimental results of manufacturing the cellular glass from container glass waste using an unconventional heat treatment technique (microwave energy) are presented in the paper. The cellular glass has physical, mechanical and morphological characteristics suitable for using as an insulating material in construction constituting an alternative for existing building materials. By comparison with these materials, the cellular glass has a higher resistance to mechanical stress, fire, humidity, attack of rodents, insects, bacteria, etc. The apparent density and thermal conductivity of the product are low (below 0.34 g/cm³ and 0.062 W/m·K, respectively), the compressive strength is acceptable (over 1.2 MPa) and the material structure is homogeneous with closed pores evenly distributed. The originality of the manufacturing technique of cellular glass presented in the paper is the use of microwave energy, unlike the conventional methods currently applied in the world. The advantages of the use of this unconventional technique are the low specific energy consumption (around 1 kWh/kg) and its non-polluting character.

Keywords: cellular glass; glass waste; microwave; lightweight; mechanical strength.

REZUMAT

În lucrare sunt prezentate rezultate experimentale ale fabricării sticlei celulare din deșeu de sticlă de ambalaj utilizând o tehnică neconvențională de tratament termic (energia microundelor). Sticla celulară are caracteristici fizice, mecanice și morfologice adecvate pentru utilizarea ca material izolator în construcții, constituind o alternativă pentru materialele de construcție existente. Prin comparație cu aceste materiale, sticla celulară are o rezistență superioară la solicitări mecanice, foc, umiditate, atacul rozătoarelor, insectelor, bacteriilor, etc. Densitatea aparentă și conductivitatea termică sunt mici (sub 0,34 g/cm³ și respectiv, 0,062 W/m·K), rezistența la compresiune este acceptabilă (peste 1,2 MPa), iar structura materialului este omogenă, cu pori închiși uniform distribuiți. Originalitatea tehnicii de fabricare a sticlei celulare prezentate în lucrare este dată de utilizarea energiei microundelor, spre deosebire de metodele convenționale aplicate în mod curent în lume. Avantajele utilizării acestei tehnici neconvenționale sunt consumul specific redus de energie (în jurul a 1 kWh/kg) și caracterul său nepoluant.

Cuvinte cheie: sticlă celulară; deșeu de sticlă; microunde; greutate redusă; rezistență mecanică.

1. INTRODUCTION

A large amount of waste annually generated in the world, including those of plastic, glass and metal, is represented by the post-consumer container glass (about 70%) [1]. Although the glass recycling in closed circuit for the industrial manufacture of the new glass is the most common method, the share of re-using the glass waste as a building material has significantly increased in the last decades due to the lower costs.

The manufacturing method of cellular glass from glass waste consists of its thermal

treatment at high temperature (800-1150°C) together with a foaming agent and other mineral activators if necessary. The foaming agent is so adopted as to release a gas by a decomposition or oxidation reaction. The softening point of the raw material powder mixture must be correlated with the temperature at which the gas is released, so that it meets a material with an adequate viscosity. Thus, the gas bubbles will be trapped inside the viscous mass and then by cooling they will form a porous structure [2]. Numerous types of foaming agent can be used in the manufacturing process of cellular glass:

calcium carbonate, sodium carbonate, calcium sulfate, black carbon, graphite, coal dust, silicon carbide, silicon nitride, etc. [3].

The cellular glass has a wide application field. Those with high mechanical strength (4-6 MPa) are used as aggregate or foam glass gravel for insulation fill in building foundation, lightweight fill material for landscaping, roof gardens, green roofs, road and railway construction, bridge abutments, insulating of underground pipelines and storage tanks, drainages, sport grounds, etc. [4, 5] and those with low apparent density (below 0.35 g/cm^3) and acceptable compressive strength (1.2-1.7 MPa) are used as insulating material, filter, absorber, gas sensor, etc. [3, 6, 7].

According to the information in the literature [2], the cellular glass is currently industrially manufactured. The main cellular glass assortments on the world market are "TECHNOpor", manufactured under the license of Misapor Switzerland company with facilities in Switzerland, Germany, Austria, France and "Foamglas", manufactured under the license of Pittsburgh Corning company with facilities in the United States, Europe (Belgium, United Kingdom, Czech Republic, Germany) and China.

The "TECHNOpor" products are made from 100% glass waste. The literature does not provide information about the used foaming agent. The average heating temperature is 900°C in a conveyor furnace. The throughput time is around 30 min [8]. The main characteristics of this product are: compressive strength between 4.9 and 6.0 MPa, thermal conductivity between 0.075 and $0.095 \text{ W/m}\cdot\text{K}$, bulk density in the range $1.21\text{-}1.40 \text{ g/cm}^3$, resistance to the action of salts, acids, bacteria, insects and other external agents, resistance to fire and frost and the fact that it is non-absorbent of humidity [4]. The "Foamglas" products are made by a previous correction of glass waste by melting at 1250°C , followed by cooling, grinding and the addition of the foaming agent (black carbon). The compressive strength of the products is between 1.6 and 2.75 MPa and the permeability to water and steam is null [7].

All the world industrial processes of cellular glass manufacturing are based on conventional heating techniques (electrical resistances or burning fossil fuels).

Several results of experiments on cellular glass production from glass waste and other silicate waste performed by teams of researchers from various countries are presented in literature. Numerous silicate waste such as coal ash, dust and fly ash collected in the filters of waste incinerators, sludges from zinc hydrometallurgy, metallurgical slag, etc., mixed with glass waste, thermal treated at high temperature of over 1100°C , in the presence of a suitable foaming agent, lead to the formation of cellular glass-ceramics, partially polycrystalline material obtained by the controlled crystallization of glass [6]. A cellular glass-ceramic from a mixture of container glass waste (80 wt.%) and coal ash (20 wt.%) incorporating less than 5 wt.% silicon carbide (grain size of $5\text{-}25 \mu\text{m}$) as a foaming agent was experimentally produced by a team of British researchers [6, 9]. Sintered at about 1000°C , the fine mixture has foamed as a cellular glass-ceramic with the porosity up to 90% and the pore size between 0.2 and 1.5 mm. The compressive strength of these products was between 1.2 and 1.7 MPa.

The concerns of the researchers on the peculiarities of the cellular glass manufactured from flat glass waste or glass from the cathode ray waste (CRT) are also presented in the literature [10, 11].

As in the case of the industrial manufacturing of cellular glass, all the works carried out in the experimental stage used exclusively conventional heating techniques.

An advanced unconventional heating technique, known since the mid-20th century, but industrially applied to a very small extent so far, is the use of the microwave irradiation. The higher heating rate and the lower energy consumption are the main advantages of this technique [12]. Referring to the possibility to apply the microwave heating in the foaming process of glass waste, the work [13], achieved in 1997, considers that the microwave heating starting at the room temperature is difficult

due to the presence in the glass composition of SiO_2 and Al_2O_3 that are microwave transparent compounds. As the temperature rises, the dielectric characteristics of glass, that allow the microwave absorption, increase significantly, but the maximum efficiency is reached only around 500°C . Thus, the authors of the paper [13] concluded that two systems for heating the manufacturing furnace of the cellular glass would be needed, one conventional and the other unconventional. This theory has been accepted and taken up by other authors in their works [2, 3]. Theoretically, this is correct. However, practically, due to the presence (even in small weight proportions) in the commercial glass composition of some inherent contaminants (Fe_2O_3 , Cr_2O_3 , etc.) with microwave susceptibility, the microwave heating process can be performed with maximum intensity starting from the ambient temperature, fact experimentally proved by the authors of the paper [14].

In the last three years, the Romanian company Daily Sourcing & Research has carried out numerous experiments for producing cellular glass by microwave irradiation, the products having physical, mechanical and morphological characteristics almost similar to those of the products manufactured by conventional methods. The results were published in several international and Romanian journals [14-18].

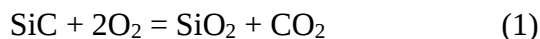
The research results on the manufacture of a cellular glass-ceramic from glass waste using the technique of microwave heating are presented below.

2. METHODS AND MATERIALS

2.1. Methods

As mentioned above, the principle of thermal foaming of glass-based raw material powder mixture consists in the incorporation into the mixture of a foaming agent able to release a gas at a temperature similar to that of the softening point of raw material, generating bubbles in the viscous mass, then blocked by cooling and forming the specific porous

structure. The foaming agent adopted in experiments was the silicon carbide, which at temperatures of over 900°C [19, 20] reacts with oxygen from the air existing between the powder mixture particles, releasing CO_2 and CO according to the below reactions:



The presence in the mixture of coal ash contributes to the increase of the softening temperature of glass at over 950°C . Depending on the weight proportion, the foaming temperature can reach values close to 1000°C or even higher [9]. The silicon carbide, being a high microwave susceptible material, is obviously suitable for the sintering / foaming process carried out in the microwave field.

The experimental microwave equipment used in experiments was a 0.8 kW-microwave oven, currently used in the household at the food preparation, adapted to operate at high temperature (up to 1200°C). The material, previously pressed into a mold and then released, was placed freely on a metal plate deposited on the bed of ceramic fiber mattresses from the base of the oven through a metal support. The pressed material was protected against the microwave field with a ceramic tube made of silicon carbide with the thickness wall of 5 mm, provided with a ceramic lid from the same material. More ceramic fiber mattresses have protected the outer surface of the ceramic tube and lid to avoid the heat loss to the outside. The control of the process temperature was achieved with a radiation pyrometer mounted above the oven at about 400 mm, viewing the surface of the heated material through holes in the upper metal wall of the oven, the ceramic lid and the ceramic fiber mattress that protects the lid. Fig. 1 shows the constructive scheme of the experimental microwave equipment.

The thickness (5 mm) of the ceramic tube is considered optimal to reduce the destructive effect of the direct microwave heating on the internal structure of the material subjected to the heating. The microwave absorbent

character of the silicon carbide wall allows the partial penetration of the microwave field generating the direct heating of the material. On the other hand, the ceramic tube wall absorbs the rest of the microwave flow ensuring its rapid heating, which then transfers the heat by thermal radiation (indirect heating). This mixed heating system has proven to be optimal for efficient and very good quality foaming of the material and was successfully tested in numerous experiments carried out by the Daily Sourcing & Research Company.

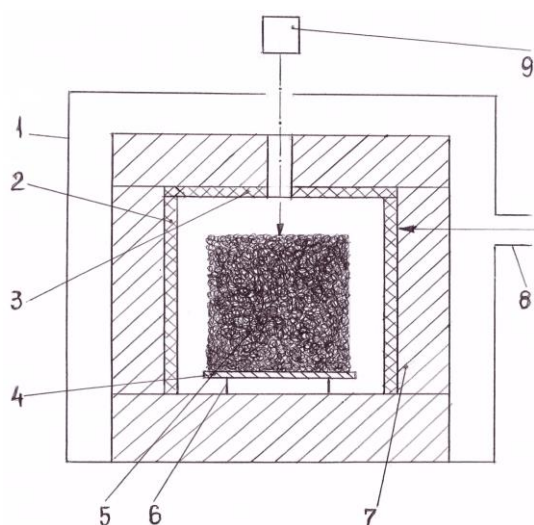


Fig. 1. Constructive scheme of the experimental equipment

1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – metal plate; 5 – pressed powder mixture; 6 – metal support; 7 – ceramic fiber; 8 – microwave generator; 9 – radiation pyrometer.

2.2. Materials

The glass-based raw material used in experiments was composed of 75 wt.% container glass waste (colorless, green and amber in the 50/ 20/ 30 weight ratio) and 25 wt.% colorless flat glass waste. The weight ratio of the container glass waste adopted when preparing the raw material is considered close to the real ratio of the recycled glass sorts in Romania [17]. The chemical composition of the container glass types [17] and the colorless flat glass [16] are shown in Table 1.

Table 1. Chemical composition of the glass waste

Component	Container glass, wt.%			Flat glass wt.%
	Colorless	Green	Amber	
SiO ₂	71.7	71.8	71.1	71.1
Al ₂ O ₃	1.9	1.9	2.0	1.3
CaO	12.0	11.8	12.1	9.3
Fe ₂ O ₃	0.05	0.01	0.25	0.2
MgO	1.0	1.2	1.1	3.9
Na ₂ O	13.3	13.1	13.3	Total
K ₂ O	-	0.1	0.1	14.2
Cr ₂ O ₃	0.05	0.09	-	-
SO ₃	-	-	0.05	-

The glass waste was thermally washed at 250 °C for removing the inherent organic contaminants. Then, it was ground in a ball mill and sieved at the grain size below 250 µm.

In the composition of the raw material also entered coal ash provided by the Paroşeni (Romania) thermal power station. The grain size of the ash was below 130 µm. The chemical composition of this waste was the following: 46.5% SiO₂; 23.7% Al₂O₃; 3.2% MgO; 7.9% CaO; 10.1% Na₂O + K₂O and 8.6% Fe₂O₃.

The silicon carbide used as a foaming agent had the grain size below 40 µm.

2.3. Characterization of the foamed samples

The cellular glass samples made by the sintering/ foaming process of glass waste were characterized in laboratory. The main determined features were: apparent density, porosity, thermal conductivity, water permeability, compressive strength, hydrolytic stability, microstructural configuration and crystallographic structure of the cellular glass samples. The apparent density was measured by the gravimetric method [21]. The porosity was calculated by the comparison method of the density of the compact material (melted and cooled) and the density of the porous material, experimentally measured [22]. The determination of the thermal conductivity was carried out by measuring the thermal flow that passes through the sample mass with a thickness of 50 mm, placed between two metal plates. One plate was heated and protected

with a thermal insulation and the other plate was cooled [23]. The water permeability of the cellular glass-ceramic was measured by the method of immersion of the sample in water (ASTM D 570). To determine the compressive strength of the porous material an own conception device was used to develop an axial compression force with a hydraulically operated piston. The sample had a cylindrical shape with the diameter of 80 mm and the height of 70 mm. The ultimate compression force applied axially to the sample before cracking was considered the compressive strength value. The microstructural configuration of the samples was observed with a smartphone digital microscope. The hydrolytic stability of the samples was determined by the standard procedure ISO 719:1985 with a 0.01M HCl solution [24, 25]. The crystallographic structure was investigated with the X-ray diffraction method (XRD), according to the standard EN 13925-2: 2003, using a X-ray diffractometer Bruker-AXS D8 Advance with CuK α radiation.

3. RESULTS AND DISCUSSION

3.1. Results

For the experimental manufacture of cellular glass-ceramic from glass waste and

coal ash using silicon carbide as a foaming agent four experimental variants were adopted. The composition of these variants is shown in Table 2.

Table 2. *Composition of the experimental variants*

Variant	Glass waste wt. %	Coal ash wt. %	Silicon carbide wt. %	Water addition wt. %
1	88.7	8.9	2.4	9.0
2	87.7	9.5	2.8	9.0
3	86.7	10.0	3.3	9.0
4	85.6	10.5	3.9	9.0

The range values of the weight proportions of coal ash (between 8.9 and 10.5%), silicon carbide (between 2.4 and 3.9%) and water addition (9%) to facilitate the raw material mixture pressing were adopted based on the own previous experience.

The main functional parameters of the manufacturing process of cellular glass-ceramic performed in the microwave oven are presented in Table 3 and the physical and mechanical characteristics of the samples are shown in Table 4.

According to the data in Table 3, the final temperature of the foaming process of raw material had high values (between 958 and 975°C), the increase of the coal ash proportion directly influencing the temperature value.

Table 3. *Main functional parameters of the manufacturing process of cellular glass-ceramic*

Variant	Dry raw material/ cellular glass amount g	Sintering/ foaming temperature °C	Heating time min	Average rate, °C/ min		Index of volume growth	Specific energy consumption kWh/ kg
				Heating	Cooling		
1	590/ 571	958	47	20.0	5.9	1.8	0.96
2	590/ 573	965	48	19.7	6.0	1.9	0.98
3	590/ 570	969	49	19.4	5.9	2.1	1.00
4	590/ 568	975	51	18.7	6.4	2.6	1.05

Table 4. *Physical and mechanical features of the samples*

Variant	Apparent density g/ cm ³	Porosity %	Thermal conductivity W/ m·K	Compressive strength MPa	Water permeability %	Pore size mm
1	0.34	84.5	0.062	1.7	0.8	0.5 – 0.9
2	0.31	85.9	0.057	1.5	1.3	0.6 – 1.6
3	0.28	87.3	0.053	1.4	1.1	0.9 – 1.8
4	0.25	88.6	0.046	1.2	1.2	1.2 – 3.5

The expansion of the material was more accentuated with the increase of the final temperature of the process, the initial volume increasing up to 2.6 times. What is remarkable and due to the microwave heating technique is the low level of the specific energy consumption (between 0.96 and 1.05 kWh / kg) close to that of a similar industrial process carried out in continuous operation (between 0.8-1 kWh / kg [2]). According to [12], by the use of high power microwave equipment, specific to the industrial sector, the energy efficiency of the process could increase by up to 25% compared to a microwave oven of the type used in experiments with the power of 0.8 kW.

Table 4 highlights the main features of the cellular glass-ceramic. The porosity of the material had high values, reaching 88.6% in the case of the sample corresponding to variant 4. Consequently, the thermal conductivity was low with values between 0.046 and 0.062 W/m·K. The material was practically waterproof, absorbing below 1.3% water. The compressive strength reached the maximum value (1.7 MPa) in the case of variant 1, decreasing up to an acceptable value for using as an insulating material (1.2 MPa) in the case of variant 4.

According to the data in Table 4, the pore size of sample 1 is between 0.5 and 0.9 mm. The dimensions are larger in the case of samples 2 and 3 (0.6-1.6 mm and 0.9-1.8 mm, respectively) and grow much in the case of sample 4 reaching 1.2-3.5 mm. The pore distribution is homogeneous in the section of the four samples, as shown in Fig. 2.

The same conclusion on the homogeneous distribution of the pores also resulted by analyzing the microstructural configuration of the samples (Fig. 3).

Considering the physical, mechanical and microstructural features of the samples, it can be concluded that all these embed the qualities required for an insulating material usable in construction. However, the samples 2 and 3 were selected as optimal for this application.

The four cellular glass-ceramic samples were subjected to the XRD analysis. The diagrams from Fig. 4 have shown that the

main crystalline phase identified after the thermal treatment was wollastonite-2M (CaSiO_3) and traces of silicon carbide (SiC). Cristobalite was not detected in the cellular glass-ceramic samples, although silica (SiO_2) was found in high proportion in the raw material composition.

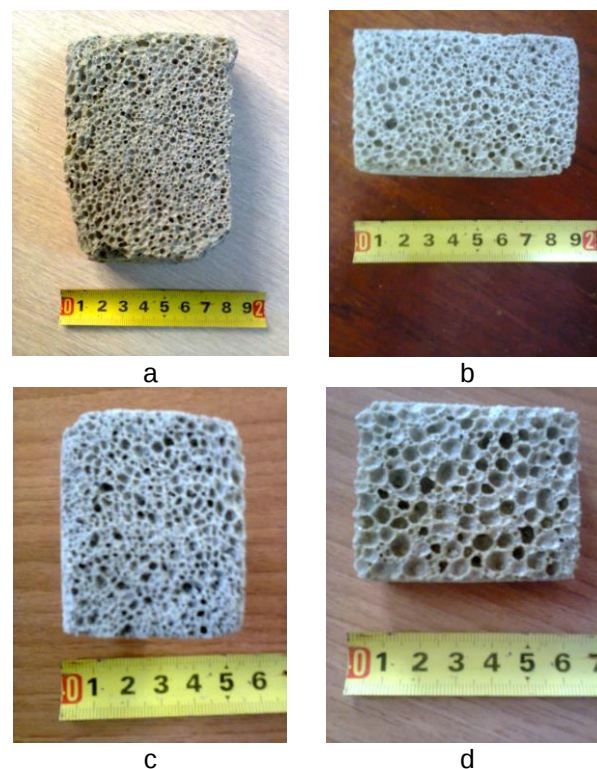


Fig. 2. Pictures of longitudinal section of the cellular glass samples
a – sample 1; b – sample 2; c – sample 3;
d – sample 4

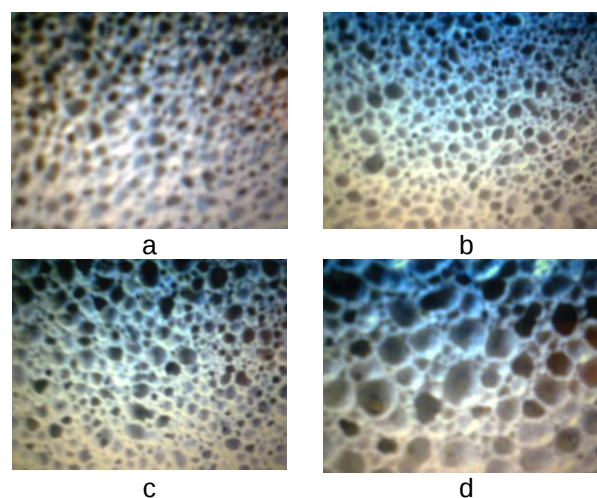


Fig. 3. Pictures of the microstructural configuration of samples
a – sample 1; b – sample 2; c – sample 3;
d – sample 4

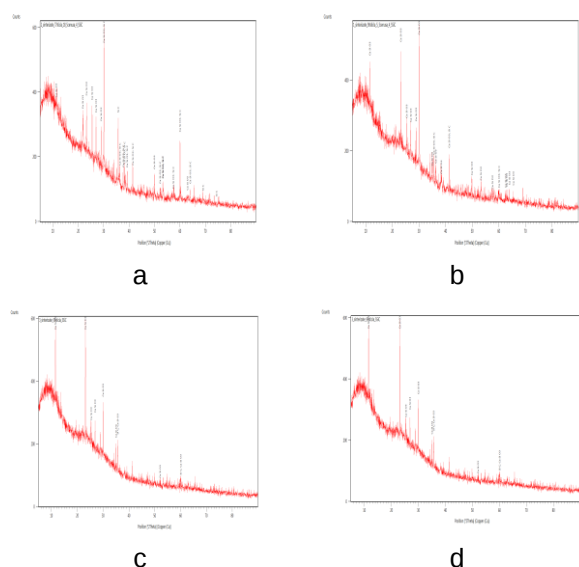


Fig. 4. Pictures of the crystallographic structure of samples

a – sample 1; b – sample 2; c – sample 3;
d – sample 4

The tests to determine the hydrolytic stability of the samples showed that the stability joins in the second hydrolytic class.

3.2. Discussion

The direct microwave heating of a material or mixture containing at least one microwave susceptible material has proven to be a very efficient energy process. Experiments previously carried out by Daily Sourcing and Research involving aluminosilicate materials and a foaming agent allowed obtaining cellular glasses with suitable characteristics and with very economical specific energy consumption despite the high sintering / foaming temperatures of over 1100°C. Unfortunately, this energy advantage cannot be applied in the case of silicates including the glass. The direct microwave heating tested in sintering / foaming processes of glass waste, whatever the nature of the foaming agent, led to the rapid destruction of the internal structure of the material being considered inadequate for this type of waste. The indirect heating of a powder glass by using a silicon carbide-based ceramic crucible or tube with the wall thickness of 15-20 mm allows producing

cellular glass with homogeneous porous structure, but lower energy efficiency. More recent research of the Daily Sourcing & Research company concluded that a mixed heating (simultaneously direct and indirect) in a silicon carbide-based ceramic crucible or tube with the wall thickness of only 3.5-5 mm constitutes the optimal solution for manufacturing with maximum energy efficiency a cellular glass qualitatively similar to those made by conventional methods. This technical solution was applied by the Romanian company in all experiments performed in the last years in the case of foaming glass waste in microwave field including also those presented in the current paper.

4. CONCLUSION

A cellular glass-ceramic was experimentally manufactured in an adapted 0.8 kW-microwave oven using container and flat glass waste as raw material and silicon carbide as a foaming agent at high temperature (958-975 °C).

Unlike the commonly conventional heating methods, the microwave heating was adopted to make a high energy efficiency cellular glass-ceramic with similar features to those manufactured by conventional techniques.

The samples 2 and 3 obtained from glass waste (86.7-87.7%), coal ash (9.5-10%) and silicon carbide (2.8-3.3%), with apparent density between 0.28-0.31 g/cm³, thermal conductivity between 0.053-0.057 W/m·K and compressive strength between 1.4-1.5 MPa were selected as optimal to be used as an insulating material in construction.

The specific energy consumption for the production by microwave irradiation of the cellular glass-ceramic samples had very low values (0.98-1 kWh/kg) close to the specific energy consumption of similar industrial processes using conventional heating techniques. Considering that a high power industrial microwave equipment can achieve a higher energy efficiency of up to 25%, compared to a 0.8 kW-microwave oven used

in experiments, it turns out that the unconventional heating method presented in the paper is superior to the conventional methods currently applied in the world.

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