# NONCONVENTIONAL TECHNIQUE FOR PREPARING COMPRESSION-PROOF CELLULAR GLASS

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ABSTRACT: Producing foamed bottle type with excellent heat-insulating features (denseness within the limits of 0.30-0.45 $g \cdot cm^{-3}$, heat conduction in the range of 0.064-0.093 $W \cdot m^{-1} \cdot K^{-1}$, and porousness within the limits of 78.6-85.7 %), and simultaneously high compression properties (6.3-8.2 MPa) was achieved in economical and environmentally friendly conditions using nonconventional method of preponderantly direct electromagnetic wave warming. The material mixture was composed of recycled residual coloured glass, $CaCO_3$, borax, glycerin, water glass, and water adding. The sintering/expanding temperature had values between 829-845 °C. Due to the high energy effectiveness of the warming technique designed by authors, the warming rate had excellent values (18.8-23.8 °C/min) without affecting the quality of cellular bottle specimens and the electricity consumption was reduced (0.83-1.06 kWh/kg).
KEYWORDS: cellular bottle, direct microwave warming, residual bottle, borax, glycerin, water glass.

## 1. INTRODUCTION

The long time of unprecedented industrial development at the global level characterized by huge consumption of hydrocarbons for energy purposes and excessively high emissions of greenhouse gases (mainly carbon dioxide) inevitably led to a situation of ecological and energy crisis felt throughout the world and having unwanted effects [1]. International organizations reacted by imposing harsh restrictions on $CO_2$ emissions in the atmosphere, which affected large energy-consuming industrial technologies [2, 3]. The most well-known example of a wide field of human economic activity is the construction sector. Based on cement as the essential binder of concrete used on a large scale in this sector, the construction materials industry experienced an important setback, replacing cement with other types of cheap and environmentally friendly materials becoming the only solution. Obviously, not only the construction sector was affected, but also other industrial branches (metallurgy, glass industry, plastic materials, paper and cardboard production, etc.). That is why the residual material recycling (metals, plastics, bottles, paper and cardboard) became a main concern of industrial producers in the last decades. Also, the elevated annual rate of waste generation due to the development of human civilization worldwide and especially, in highly developed countries and in developing countries, has created the danger of increasing soil and subsoil pollution, aquatic areas as well as the terrestrial atmosphere. Therefore, the role of waste recycling for the production of new materials or their reintroduction into the industrial circuit is multiple, favouring both energy and material savings as well as eliminating or reducing the danger of their storage in nature.

Residual glass recovered from post-consumer drinking glass or from building demolition is a suitable material source for making alternative insulating materials for construction. The manufacturing method already tested and applied in the industrial stage is based on sintering at high temperature (750-1150 °C) and expanding the bottle-based powder mixture by releasing a gas resulting from decomposing or oxidizing the mineral pore-forming agent. The resulting product (cellular bottle) uniquely embeds many properties that make it very attractive especially for builders: light weight, rigid, compression resistant, good thermal and acoustic insulator, non-toxic, chemically stable, resistant to fire, frost, water, vapour, resistant to insect, rodent and bacteria attack [4].

Several silicate wastes (such as fly ash resulting from coal combustion or waste incinerators, sludges from zinc hydrometallurgy, metallurgical slag, red mud, etc.) mixed with residual bottle subjected to sintering at 800-1150 °C together with a pore-forming material leads to the manufacture of glass-ceramics, predominantly polycrystalline porous materials [5]. Their mechanical abilities are higher compared to the precursor bottle and the fields of

application are generally the same as in the case of lightweight cellular bottle, with the difference that their mechanical strength is higher.

The cellular bottles were industrially produced firstly in the middle of the last century, pure glass being used as the basic material. Later, it was found that replacing the pure bottle with residual bottle is an efficient way of waste recycling without changing the quality of the product. Several types of cellular bottle produced in industry are known, of which "Technopor" (Misapor) and "Foamglas" (Pittsburgh Corning) are the main. "Technopor" products have high compression resistance (4.9-6.0 MPa), high durability, little heat conduction (0.075-0.095 $W \cdot m^{-1} \cdot K^{-1}$), relatively reduced denseness (1.21-1.40 $g \cdot cm^{-3}$), fireproof, waterproof, resistance to frost and aggression of acids, salts, insects, and other external agents [6, 7]. "Foamglas" are made as heat-insulating materials for buildings as well as large slabs broken into lumps used as porous aggregates. These products ensure good heat-insulating, high waterproof and moderate compression strength (1.6-2.75 MPa) [8].

The industrial processes were based on recycled consumed drinking bottle and window bottle (flat bottle) from demolitions as basic material as well as carbon black, SiC, glycerin, and $CaCO_3$ as pore-supplying agents.

Except for cellular bottle industrially manufactured in Europe (Switzerland, Belgium, Austria, Germany, Italy, etc.), the United States, and China, numerous small-scale manufacturing recipes and techniques have been tested in the world.

A mixture composed of residual bottle in a very high proportion (99 wt. %), 1 wt. % $CaCO_3$ as a pore-forming agent, and 8 wt. % as a binder sintered at 850 °C allowed to obtain a cellular bottle with reduced heat conduction (0.031 $W \cdot m^{-1} \cdot K^{-1}$) and acceptable compression strength (0.7-1.6 MPa) [9].

$CaCO_3$ and residual alkaline battery was a very effective combination used in [10] for intensifying the expansion process of residual glass. The foamed product reached a very high compression strength (18.7 MPa).

An almost similar performance was achieved by the authors of the paper [11], who used the aqueous solution of $Na_2SiO_3$ (less than 40 wt. %) combined with fine powder of residual bottle and 1 wt. % $CaCO_3$ as a pore-supplying material. The sinterization at 850 °C led to making a cellular bottle with compression resistance of 17.5 MPa as well as heat conduction (0.030 $W \cdot m^{-1} \cdot K^{-1}$).

Recycled cathode-ray-tube (CRT) bottle in mixture with C and $MnO_2$ as pore-forming materials facilitated obtaining a cellular product with excellent thermal insulating properties (heat conduction of 0.042 $W \cdot m^{-1} \cdot K^{-1}$) [12].

A very efficient pore-supplying agent (SiC) in weight proportion of 4 % and an addition (between 0.4-1.2 wt. %) of oxidizing agents ($Fe_2O_3$ and $Co_3O_4$) suppliers of gaseous oxygen, together with residual bottle (96 %), water and polyvinyl alcohol as binders constituted a mix subjected to sintering at 850 °C. The optimal cellular bottle specimen had a reduced denseness of 0.25 $g \cdot cm^{-3}$. Flexural strength reached 0.75 MPa [13].

Other tests aiming at reducing the price of the manufacturing process have replaced the usual types of mineral pore-forming agents with agents of vegetable origin. Thus, a Brazilian research team [14] used dry and ground banana leaves in a weight proportion equal to that of residual bottle (50/50). The sintering temperature reached in the process was 850 °C. The denseness of the product was little (0.52 $g \cdot cm^{-3}$) and the heat conduction value decreased to 0.060 $W \cdot m^{-1} \cdot K^{-1}$. The compression resistance had acceptable values of over 1.5 MPa.

Also, a vegetable material that abundantly exists in warm climate areas of Brazil, Argentina, and Paraguay (yerba mate) [15] was used as a pore-supplying agent in the cellular bottle manufacturing process. This finely ground CaO-rich plant was mixed in proportions between 10-30 % together with water (10 %) and residual post-consumer drinking bottle. The compacted mix was warmed to 850-900 °C for 60 min. The samples thus prepared had compression resistance between 1.5-15 MPa and heat conduction within the limits of 0.040-0.600 $W \cdot m^{-1} \cdot K^{-1}$.

All manufacturing methods (industrial and experimental) of cellular glass presented above are characterized as conventional heating techniques. Unlike these, the utilize of electromagnetic waves for the nonconventional heating of solids has been experimentally proven to be a more efficient procedure. Despite this recognition by some specialists [16] and although microwaves have been discovered for over 7 decades, their use has found its application in communications and radars and very little in high temperature heating processes. Microwave heating in drying processes as well as in heating processes at low temperatures is applied, but reaching high temperatures required by many technologies, including the production of cellular glass, is still in different testing periods. Mechanism features of the nonconventional microwave warming

process of mixtures have been investigated and presented in several works published in the literature [17-19].

An important consequence of the principle presented by Knox and Copley in 1997 [20] regarding the using conditions of microwave warming for the industrial manufacture of cellular bottle practically discouraged the trend of modernizing this process. According to [20], the traditional bottle composition (soda-lime-silica bottle) used as the main basic material is not suitable for efficient heating with electromagnetic waves, because silica ($SiO_2$) and alumina ($Al_2O_3$) as the main components of the glass are not wave-susceptible under 500 ℃, but only above this value by increasing the electrical conductivity. This conclusion was also taken over by Scarinci in 2005 [4] and Hurley in 2003 [21] without experimental verification of the information. Tests performed by some authors of the current paper [22] disproved Knox and Copley's theory by experimentally proving that commercial bottle can be effectiveness warmed starting at the beginning process due to the content (even in low proportions) of $Fe_2O_3$ and $Cr_2O_3$ that have the ability to be microwave susceptible at ambient temperature [19].

After 2016, the societies Daily Sourcing & Research SRL and Cosfel Actual SRL (Romania) started to test producing cellular bottle by microwave heating the basic material in the common experimental base. Among the works appeared in the literature, the manufacture of light cellular bottle by sintering at 820-851 ℃ is mentioned, having denseness between 0.60-0.90 $g \cdot cm^{-3}$, heat conduction between 0.081-0.105 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance between 2.5-6.2 MPa. The mixture included recycled consumed drinking bottle (90-94 %), borax (5 %) as a flux agent, $CaCO_3$ (1.5-5 %) as a pore-supplier agent, and water (8.5 %). The method of preponderantly direct microwave warming was tested using a susceptible ceramic tube with 3.5 mm-wall thickness [23].

Another work based on preponderantly direct microwave heating was presented in [24]. The method of using aluminum nitride (AlN) together with manganese dioxide ($MnO_2$) was adopted for expanding the material mixture based on recycled residual glass. The sintering/expanding temperature was within the limits of 820-850 ℃. The foamed products had the following features: denseness within the limits of 0.35-0.43 $g \cdot cm^{-3}$, heat conduction in the range of 0.080-0.094 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance in the range of 4.0-6.7 MPa. The energy effectiveness of the procedure had reduced values (0.80-0.95 kWh/kg) due to the influence of applying the electromagnetic wave warming method.

A foamed product with high mechanical resistance was prepared under the influence of wave radiation [25]. Residual flat bottle recovered from building demolition (86-95 %) was mixed together with 1 % $CaCO_3$ as an expanding material and aqueous solution of sodium silicate ($Na_2SiO_3$) between 4-13 %. Cellular bottle specimens had very good heat-insulating features (denseness in the range of 0.40-0.45 $g \cdot cm^{-3}$ and heat conduction within the limits of 0.076-0.081 $W \cdot m^{-1} \cdot K^{-1}$) as well as high compression resistance values in the range of 4.9-6.2 MPa (highest resistance value corresponding to the highest value of sodium silicate).

Keeping the same nonconventional technique of heating the mixture based on powder glass mentioned above, the current work aimed at obtaining compression-resistant cellular bottle by adopting the simultaneous combination of two known preparing systems for expanding the residual bottle, one in solid medium with $CaCO_3$ and borax and one in liquid medium with glycerin and water glass ($Na_2SiO_3$) solution. The first system has the ability to increase the strength and durability of material and the second one is known for favouring its fine porosity.

## 2. METHODS AND MATERIALS

### 2.1 Methods

As stated above, two simultaneous cellular glass preparation systems were chosen to achieve the qualitative objectives of the work.

The solid medium preparation method is based on the thermal decomposition of $CaCO_3$ (mixed with glass powder) at temperatures above 750 ℃ [26]. In the case of commercial glass, the expansion process has optimal conditions for occurring between 800-900 ℃.

$$CaCO_3 = CaO + CO_2 \quad (1)$$

The thermal decomposition of glycerin starts at about 300 ℃, increasing in intensity around 750 ℃. The process releases many types of gases (CO, $CO_2$, $H_2$, $CH_4$, $C_2H_4$, $C_2H_6$, etc.) up to pure carbon [27]. For avoiding early oxidation of carbon, it was used water glass in association with glycerin, which envelopes the fine particles of pure carbon.

The gaseous products formed at the optimal temperature spread in the heat softened mass of the mix, being trapped as gas bubbles. By the increase of temperature, the pressure inside the bubbles also increases and their volume grows and at the same

time the material expands. Through subsequent cooling, the specific porous structure is formed.

The temperature level necessary for the expanding process of the mix was reached using the microwave radiation. The wave is not a heat resource, but a very efficient potential heat carrier. Contact with a microwave susceptible material is necessary, whether it is the material being heated or a crucible containing the product for heating made of a material with the mentioned property. Through direct contact, the wave power is turned into heat. The warming is thus started in the central area of the irradiated sample where the maximum temperature point quickly develops. Heat propagation occurs volumetrically in the entire mass of the sample, being oriented from the interior to the exterior, i.e. completely inverse to traditional warming [17-19]. The hot sample represents the heating source of the system and for this reason it is necessary its thermal protection (with ceramic fiber for 1200 ℃) to avoid the loss of heat out of the system.

The originality of the equipment adopted by the authors is the use of a cylindrical tube from SiC and $Si_3N_4$ (with high wave susceptibility) procured from China, with the wall thickness of 2.5 mm, which proved to be very efficient to ensure mixed microwave warming (predominantly direct warming and partially indirect warming by heat radiation). This mixed warming method is necessary in the case of the mixture based on glass powder because the structural destruction of the product that receives direct microwave heating is avoided. The ceramic tube has the role of a protective screen that tempers the power intensity of the microwave field. The oven used in the experiments since 2017 is an 800 W-wave oven traditionally utilized in domestic activities, but with important constructive and operational transformations that allow its application at high temperatures above 1100 ℃. Pictures of the furnace (a), the tube (b), and its heat protection with ceramic fiber (c) are presented in Figure 1.

a b c

**Figure 1.** Pictures of the wave oven and its components used in experiment
a – 800 W-wave oven; b – ceramic tube; c – heat protection of the tube and its lid.

## 2.2 Materials

The components of the mix for the production of cellular bottle were the following: residual bottle, $CaCO_3$, borax, glycerin, and water glass.

The residual bottle was constituted from recycled post-consumer green and amber drinking bottle. The chemical composition of the two bottle types is presented in Table 1.

**Table 1.** Chemical composition of bottle

| Composition | Green bottle (wt. %) | Amber bottle (wt. %) |
|---|---|---|
| $SiO_2$ | 71.6 | 71.3 |
| $Al_2O_3$ | 1.9 | 2.0 |
| CaO | 11.9 | 12.1 |
| $Fe_2O_3$ | 0.1 | 0.2 |
| MgO | 1.2 | 1.1 |
| $Na_2O$ | 13.1 | 13.0 |
| $K_2O$ | 0.1 | 0.1 |
| $Cr_2O_3$ | 0.1 | 0.1 |
| $SO_3$ | - | 0.1 |

The bottle was selected by colour, washed, crushed, ground in a ball mill, and sieved, particle size under 90 μm being allowed.

$CaCO_3$ is a cheap material frequently used as a pore-supplying agent in the production technique of cellular bottle [4]. It is commercially available as a fine powder (less than 20 μm).

Sodium tetraborate (borax) in powder form is a mineral from the borate class. Theoretically, borax contains 30.8 % sodium oxide ($Na_2O$) as a very important fluxing material and 69.2 % boric oxide

($B_2O_3$), where boron favourably influenced mechanical properties.

Glycerin ($C_3H_8O_3$) is a liquid organic carbonic pore-forming material. Due to its physical state, glycerin has the capacity to penetrate between the fine bottle particles, improving the fineness of the expanded material porousness. Under the oxidizing conditions of the oven, glycerin breaks down into a very wide range of gaseous products that participate in the expanding process.

Water glass ($Na_2SiO_3$) is a compound containing $Na_2O$ and $SiO_2$ that forms a glassy solid soluble in water. In this experiment, commercially available water glass as a slightly viscous liquid was used.

The processing of the materials mentioned above was carried out separately for the solid and liquid components, respectively. Together with the liquid materials (glycerin and water glass) distilled water was also added (in a constant proportion of 9 wt. %).

Four experimental manufacturing recipes were adopted in this paper, their composition being shown in Table 2.

**Table 2.** Composition of experimental recipes

| Composition | Experimental recipe (wt. %) | | | |
|---|---|---|---|---|
| | R1 | R2 | R3 | R4 |
| Residual bottle | 90.9 | 87.7 | 84.4 | 81.2 |
| $CaCO_3$ | 0.6 | 0.8 | 1.0 | 1.2 |
| Borax | 1.5 | 3.0 | 4.5 | 6.0 |
| Glycerin | 1.0 | 1.0 | 1.1 | 1.1 |
| Water glass | 6.0 | 7.5 | 9.0 | 10.5 |
| Water added | 9.0 | 9.0 | 9.0 | 9.0 |

### 2.3 Method of investigating specimen features

Denseness and porousness were measured by applying the Archimedes' method according to ASTM D792-20 [28]. Determining the heat conduction was performed by heat-flow method (ASTM E1225-04) [29]. The measure of compression resistance was carried out with TA.XTplus Texture analyzer. The well known immersing method of the sample under water was utilized to determine the water-absorption ability (ASTM D570). Microstructural aspects of samples were examined with ASONA 100X Zoom Smartphone Microscope.

## 3. RESULTS AND DISCUSSION

### 3.1 Results

In the conditions where the wet quantity of mixture was kept unmodified at 490 g, the operational parameters of cellular bottle making technology were experimentally identified and shown in Table 3.

**Table 3.** Operational parameters

| Parameter | R1 | R2 | R3 | R4 |
|---|---|---|---|---|
| Wet basic material/cellular bottle quantity (g) | 490/ 429 | 490/ 431 | 490/ 430 | 490/ 431 |
| Process temperature (°C) | 829 | 834 | 839 | 845 |
| Warming duration (min) | 34 | 36 | 41 | 44 |
| Warming rate (°C/min) | 23.8 | 22.6 | 20.0 | 18.8 |
| Cooling rate (°C/min) | 7.0 | 7.0 | 7.0 | 7.0 |
| Specific energy consumption (kWh/kg) | 0.83 | 0.87 | 0.99 | 1.06 |

The data contained in Table 3 indicate the performance parameters of the warming process using the own method of preponderantly direct wave warming. The high warming rate, the short process duration, and the little specific energy consumption compared to the usual parameters of heat bottle expansion by conventional methods represent proof of the energy efficiency of applying the technique designed by authors.

Pictures of the four cellular bottle specimens experimentally obtained applying making recipes shown in Table 2 are presented in Figure 2.

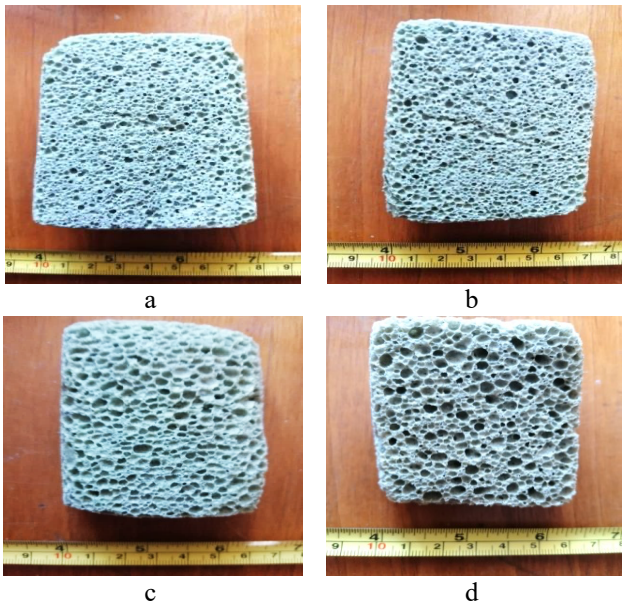



**Figure 2.** Pictures of cellular bottle specimens
a – recipe R1; b – recipe R2; c – recipe R3; d – recipe R4.

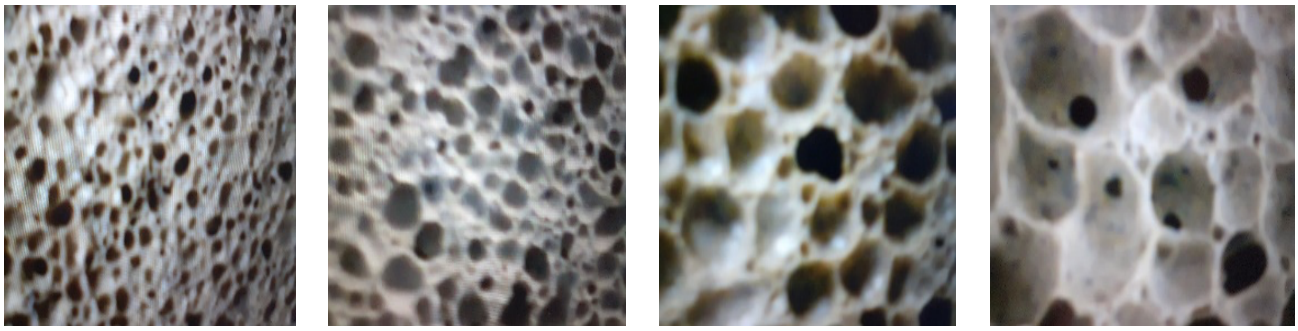
Using the investigation methods presented above, the main physical, mechanical, thermal, and morphological characteristics were determined and are shown in Table 4.

**Table 4.** Main features of cellular bottle samples

| Feature | R1 | R2 | R3 | R4 |
|---|---|---|---|---|
| Denseness ($g \cdot cm^{-3}$) | 0.45 | 0.39 | 0.34 | 0.30 |
| Porousness (%) | 78.6 | 81.4 | 83.8 | 85.7 |
| Heat conduction ($W \cdot m^{-1} \cdot K^{-1}$) | 0.093 | 0.080 | 0.070 | 0.064 |
| Compression resistance (MPa) | 8.2 | 7.5 | 6.8 | 6.3 |
| Water-absorption (vol. %) | 0.9 | 1.1 | 1.0 | 1.3 |
| Pore size (mm) | 0.1-0.4 | 0.2-0.7 | 0.4-0.8 | 0.7-1.3 |

Both the heat-insulating features (denseness below 0.45 $g \cdot cm^{-3}$, heat conduction below 0.093 $W \cdot m^{-1} \cdot K^{-1}$, and porousness above 78.6 %), and especially the compression resistance properties (6.3-8.2 MPa) were remarkable. Water-absorption capacity was at a very low level (around 1 vol. %) and pore sizes were uniformly distributed and small.

Pictures of the microstructural aspect of the four specimens corresponding to tested manufacturing recipes (R1-R4) are shown in Figure 3.



a b c d |----------| **2 mm**

**Figure 3.** Microstructural aspect of cellular bottle specimens
a – recipe R1; b – recipe R2; c – recipe R3; d – recipe R4.

Examining the pictures containing the microstructural aspect of cellular glass specimens in Figure 3 reveals the good homogeneity of their porous structure. Except for the picture corresponding to recipe R4 with the highest contents of water glass and $CaCO_3$ in which, with the increase of the pore size (0.7-1.3 mm), a slight tendency to intercommunication between cells is observed. This microstructural modification contributes to the decrease of the material density. However, the high content of borax (6 %) keeps a high level of compression resistance (6.3 MPa).

3.2 Discussion

The testing outcomes were significantly influenced by the combination of the pore-supplying agents ($CaCO_3$ and glycerin) and the additive materials. Thus, although borax with a role in increasing the compression resistance of material and water glass with a role in increasing its porousness, had increasing values between R1 and R4, the denseness as well as heat conduction were clearly decreasing. However, the level of the compression resistance values was very high (6.3-8.2 MPa), even in the case of recipe R4, whose application led to a structure with relatively large pores.

Under these conditions, the correlation of physical, mechanical, and heat properties of cellular bottle has become important. Analyzing the interference of these property effects corresponding to the four making recipes, it was concluded that recipe R3 can be chosen as the best option. Its heat-insulating properties are excellent (denseness of 0.34 $g \cdot cm^{-3}$, heat conduction of 0.070 $W \cdot m^{-1} \cdot K^{-1}$, and porousness of 83.8 %) and at the same time compression resistance has reached an ideal value for construction applications.

## 4. CONCLUSION

The work aimed at making the cellular glass through the efficient technique of preponderantly direct wave warming. The material components of the mixture prepared for this purpose were coloured residual bottle, $CaCO_3$, borax, glycerin, and water glass solution. A good correlation of the effects on cellular bottle characteristics was obtained by combining solid (with $CaCO_3$ and borax) and liquid (with glycerin and water glass) systems for expanding the residual bottle. Products had excellent properties (denseness of 0.34 $g \cdot cm^{-3}$, heat conduction of 0.070 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance of 6.8 MPa). The making recipe using bottle (84.4 %), $CaCO_3$ (1

%), borax (4.5 %), glycerin (1.1 %), water glass (9 %), and water added (9 %), sintered at 839 °C, was chosen as the best option.

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