STEEL SLAG-BASED CELLULAR CERAMIC WITH HIGH-STRENGTH AND EXTREMELY FINE POROSITY AS A BUILDING MATERIAL MADE BY NONCONVENTIONAL HEATING TECHNIQUE

Lucian Paunescu[1], Sorin-Mircea Axinte[2,3] and Bogdan-Valentin Paunescu[4]
[1] Cosfel Actual SRL Bucharest, Romania, lucianpaunescu16@gmail.com
[2] Daily Sourcing & Research SRL, Bucharest, Romania, sorinaxinte@yahoo.com
[3] University POLITEHNICA of Bucharest, Faculty of Applied Science and Materials Science Bucharest, Romania, sorinaxinte@yahoo.com
[4] Consitrans SA Bucharest, Romania, pnscbogdan@yahoo.com

ABSTRACT: Ceramic foam prepared from steel slag particles in colloidal suspension was stabilized with propyl gallate and after drying was sintered by direct electromagnetic wave radiation at successive thermal regimes within the limits of 980-1200 ºC. The microwave heat treatment is the originality of the work. The heating rate reached very high values of up to 40 ºC/min without affecting the microstructural configuration of the foam. The peculiarities of ceramic foam include reduced heat conductivity (in the range of 0.060-0.085 $W\cdot m^{-1}\cdot K^{-1}$), high compression resistance (about 9.8 MPa) and extremely low pore size (between 20-160 μm).
KEYWORDS: ceramic foam, direct microwave irradiation, colloidal suspension, steel slag, propyl gallate.

1. INTRODUCTION

In the last time, recovery of residual materials (glasses, plastics, metals, paper, textiles, etc.) and secondary products of industry (coal fly ash, slag, red mud, lubricating oil, etc.) [1] became imperative for developed societies and developing countries. The annually generation rate of wastes is growing and their storing is no longer an adequate resolution. Under these conditions, the re-use of waste and secondary products in the production processes of new final materials of similar type is no sufficient solving in ecological terms, new valorisation variants being required.

Steel slag is a secondary product of the iron and steel industry used to remove unwanted chemical compounds in molten steel. The slag is discharged from the steel furnace and transported to the landfill. Extremely low amounts of valuable metal components can be accidentally found in landfills and some recovery methods are applied. Unlike the blast furnace slag which is granulated and expanded on the technological fluxes of metallurgical plants being used then as a construction material, the steel slag has fewer known applications. In the construction sector, it is adequate for using as a cement raw meal and as filler in the basement of buildings. It can be also used to produce fertilizers or soil improvers for agriculture [2]. Its landfilling is an important source of environmental pollution. The chemical composition of steel slag includes mainly total iron, CaO, and SiO_2 (above 80 %), but also very low amounts of heavy metal elements (cadmium, chromium, copper, manganese, lead, zinc) [3].

In the paper [4] the chemical composition of EAF steel slag from ArcelorMittal Galati (Romania) is presented. The composition was determined by measurements performed by a team of University of Galati and contains: 42-44 % CaO, 5-7 % MgO, 30-35 % SiO_2, 2.5-3.7 % Al_2O_3, 15-20 % Fe_2O_3, 2.5-5.0 % FeO, 3-6 % MnO, and other oxides (Cr and V) below 0.5 %.

The method of manufacturing cellular ceramic using colloidal suspension foaming is a method experienced in recent years. In thermodynamic terms, the instability of wet foamed materials, that could cause unwanted bubbles over time, can be significantly modified by the use of surfactant powders as foaming stabilizers [5-7]. The fine powders have the ability to irreversibly adsorb at the contact surface between water and air. According to [8], a relatively recent method of manufacturing particle-stabilized foams is based on "in situ" hydrophobization of the hydrophilic powders allowing their adsorbing on the air bubbles outward face. Hydrophobization is achieved by adsorption of short-chain amphiphiles on the particle surface. Propyl gallate ($C_{10}H_{12}O_5$) was used. It is an antioxidant providing protection against oxidation with hydrogen peroxide (H_2O_2) and oxygen free radicals [9]. Several antioxidants, including propyl gallate, have been investigated [10] to evaluate their resistance to thermal oxidation. Propyl gallate has higher resistance and its decomposition processes begin at higher temperatures (above 200-220 ºC as high as 300 °C) compared to other antioxidants. Propyl gallate decomposes into propyl alcohol named also n-propanol (C_3H_8O) in a liquid state, identified (from the TG-FTIR coupled techniques) as a volatile compound with the boiling point of 97 ºC [11]. It is possible that different carbon-based compounds result from the decomposition process as well as hydroxyl compounds.

The paper [12] presents a method of foaming with colloidal suspension using glass waste as raw material. The foam stabilization was obtained by in situ hydrophobization of glass waste particles with propyl gallate as a surface modifier. The optimal parameters of the foaming process were obtained by determining the pH value of suspension, the weight proportion of propyl gallate and the grinding time of the glass waste in the ball mill. The foam products had very low pore sizes compared to glass foams made by current methods. Closed-pore structures, but also open-pore structures were obtained by adapting the solid loading of the colloidal suspension. The closed-pore structures are applicable as thermal insulation materials in building construction and the open-pore structures can be used in catalyst loading, filtration and separation domains.

Other porous ceramics with controllable porosity and pore size were manufactured by colloidal processing of ceramic particles [13]. Polymeric spheres as templates and very fine ceramic powders as construction blocks were used for forming porous macrostructures. Different chemical compounds such as alumina, titanium, and zirconia with uniform porosity were obtained by this procedure.

The direct foaming method is a convenient way to manufacture high porous structure ceramics with closed and open pores allowing the formation of suspensions. Granulated stabilized foam offers several advantages, especially since it is no longer necessary for adding inorganic or organic binders because the ceramic particles ensure a good stability of the gas-liquid interface [14].

For particle-stabilized foam, the pH indicating its acidic or basic character is essential for determining the chemical form of the powder surface [15]. Not only does it determine the adsorption improvement of propyl gallate on the powder surface, but it also makes the foam suspension stability. The stable foam is made with pH within the limits of 5.0-10.0. Regardless of the amount of propyl gallate added, the wet glass waste foam cannot maintain long-term stability outside the pH range. The stability of the wet foam depends mainly on the hydrophobization of the glass waste particles, which is mainly affected by the adsorption of the modifier on the surface of the glass particles.

A recent paper published in the literature [16] refers to experimental results obtained in the process of manufacturing ceramic foam from steel slag (a metallurgical by-product) using propyl gallate as a surface modifier. The used method is the foaming with colloidal suspension being made particle-stabilized foam. The grinding of the steel slag was performed in a ball mill, the average grain size being 1.62 μm. Propyl gallate was added to the suspension. It was stirred at high speed to get wet foam. Dried at room temperature, the foam was then sintered in a conventional oven at temperatures between 1150-1250 ºC by heating at a rate of 3 ºC/min and a holding time of 2 hours. The stabilization of the ceramic foam was obtained by controlling the pH value and the weight proportion of propyl gallate. The porosity and compression resistance were verified and controlled by modifying the solid amount in suspension and the sintering temperature. According to [16], the porosity varied between 62.53-85.6 %, the compressive strength had values in the range 1.74-10.42 MPa, and the heat conductivity registered a minimum value of 0.067 $W \cdot m^{-1} \cdot K^{-1}$, confirming excellent thermal insulation properties.

The present paper followed the making of high-strength ceramic foam with extremely fine porosity as a thermal insulation building material using steel slag by the colloidal suspension foaming method recently tested worldwide by conventional techniques. The paper originality was using the nonconventional electromagnetic wave direct heating technique. Unlike the use of residual glass as raw material [12], its replacement with steel slag as an industrial alumino-silicate by-product allowed the direct electromagnetic wave heating with remarkable heating speed without affecting the microstructure of the expanded product and had the effect of significantly reducing the energy consumption of making process.

2. METHODS AND MATERIALS

2.1 Methods

Several technologies use wet foams as final or intermediate products. These structures have the disadvantage of thermodynamic instability which in time causes the appearance of unwanted bubbles. Cellular material stability can be significantly perfectible by using surfactant powder as foam stabilizers. The powder had the role of adsorbing at the air-water contact surface [8, 17].

The new method of making slag foam as a raw material is based on the colloidal suspension foaming method. It has been shown that the stabilization of the foam by in-situ hydrophobization of finely ground particles of slag is achievable, obtaining at the end of the process foams with extremely low pore size compared to the size of the most ceramic foams known in the literature. The literature has showed that, in order to increase the stability of suspensions, the slag powders should be coated at the air-water contact surface. Propyl gallate ($C_{10}H_{12}O_5$) is an antioxidant (crystalline powder) that has the ability to protect particles against oxidizing with hydrogen peroxide and oxygen free radicals [9].

According to [18], a higher solid loading in the suspension leads to an increase of its viscosity, i.e. the incorporation of lower amounts of air in the ceramic suspension. Therefore, by increasing the proportion of the solid loading in suspension, the foam porosity decreases and the compressive strength increases.

In principle, the production of steel slag cellular ceramic by colloidal suspension foaming method took place following the following steps:

Very finely crushed and ground steel slag (below 20 µm) was mixed with distilled water in a removable cylindrical mold forming a colloidal suspension. The antioxidant propyl gallate was then added as a water-soluble crystalline powder [19] and $HCl/NH_3 \cdot H_2O$ for pH adjustment (recommended 5-10 [16]). The wet material mixture was further stirred to obtain wet foam using a commonly household mixer. The drying process was carried out at room temperature for 48 hours. After removal from the mold, the ceramic material was freely placed in a 800 W-electromagnetic wave oven, commonly used in the household, but adapted for operating at high thermal regimes (up to 1200 ºC). The final foamed product was obtained by thermal sintering the material as a result of the direct microwave irradiation.

The building and operating scheme of the testing equipment is presented in Figure 1.

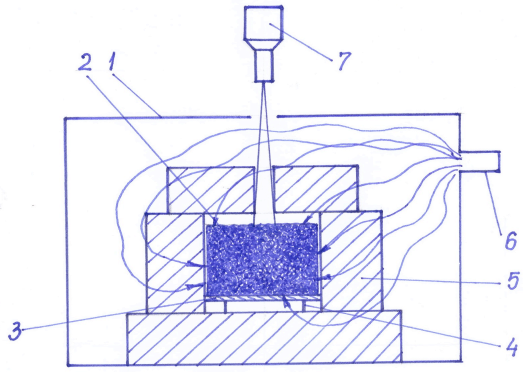


Figure 1. Building and operating testing equipment
1 – electromagnetic wave oven; 2 – irradiated material; 3 – metal plate; 4 – metal support; 5 – thermal insulation protection; 6 – waveguide; 7 – radiation pyrometer.

The peculiarities of the unconventional heating process by microwave direct irradiation, which differentiates it from conventional heating processes, have been taken into account. According to [20, 21], the beginning of warming takes place in the middle of the mixture subjected to heat, where the power of the electromagnetic wave field is turned into heat. Thus, the interior zone of the material becomes the high heated area and the heat volumetrically propagates in its entire mass from inside to outside, i.e. inversely to the heat transfer in the ordinary thermal process. Also, the selectivity of the warming is another important feature of the nonconventional process, which allows heating only the material targeted (susceptible to microwaves) and no different masonry corps into the furnace (vault, walls, hearth) in which the heating takes place. Therefore, the mixture prepared in this experiment was very well heat secured with thick ceramic fiber beds (with thermal resistance up to 1200 ºC) both at the base of the oven and on the side and upper surfaces. In this way, the heat generated inside the material and homogenized in its entire volume was very well preserved in this area and used only for sintering, so that the thin walls of furnace did not need additional protection. The maximum temperature measured on the outer surface of the tin wall during the heat treatment did not exceed 65 ºC. To control the temperature of the irradiated material, a radiation pyrometer placed overhead the furnace was used. This visualized its very warm surface through given holes in the metal ceiling of the oven and the superior layer of ceramic fiber.

2.2 Materials

The solids that compose the loading for manufacturing cellular ceramic were steel slag and propyl gallate. The steel slag was provided by ArcelorMittal Galati and had the following oxide components: 42-44 % CaO, 5-7 % MgO, 30-35 %

SiO_2, 2.5-3.7 % Al_2O_3, 15-20 % Fe_2O_3, 2.5-5.0 % FeO, 3-6 % MnO, and other oxides (Cr and V) below 0.5 % [4]. The slag lumps were broken, repeatedly subjected to grinding, and granulometrically separated by sieving. Slag particles with the grain size below 20 µm were selected.

Propyl gallate is an antioxidant organic material (ester). It has been used in the food industry since the mid-20th century to prevent the oxidation of foods containing oils and fats [22]. The role of propyl gallate in this experiment was to substantially improve the stability of steel slag foam by adsorbing the slag grains at the water-air contact surface. According to the literature [8], short-chain of propyl gallate could be suitable for “in-situ” hydrophobizing the face of slag grains. Propyl gallate as an antioxidant in powder form below 50 µm has been purchased from the market. The material is soluble in water [19].

2.3 Methods of foam characterization

The method of measuring the apparent density was the gravimetric methodology [23] and the procedure of comparing the "true" and apparent density [24] was chosen for calculation the specimen porosity. Thermal conductivity was measured by the heat flow technique according to ASTM E1225-04 standard, while the compression resistance was determined with TA.XTplus Texture Analyzer. Measuring the H_2O absorption by specimens was made through immersing the sample under water for 1 day (ASTM C1585-20). The microstructural peculiarities of specimens were analyzed based on ASONA 100X Zoom-Smartphone Digital Microscope Type. The measurements mentioned above were performed in the Romanian companies Cosfel Actual SRL and Daily Sourcing & Research SRL, and also in University “Politehnica” and Metallurgical Research Institute of Bucharest.

3. RESULTS AND DISCUSSION

3.1 Results

Four testing making recipes were adopted including steel slag (43.0-45.2 %), propyl gallate (1.8-4.0 %), and distilled water (53.0 %). The dosage of materials is indicated in Table 1.

Table 1. Testing versions of porous glass specimens

Compound	Version			
	1	2	3	4
Steel slag (wt, %)	43.0	43.7	44.5	45.2
Distilled water (wt. %)	35.0	35.0	35.0	35.0
Propyl gallate (wt. %)	4.0	3.3	2.5	1.8
Total colloidal suspension (g)	450	450	450	450

The principal operating frameworks of the making procedure are shown in Table 2.

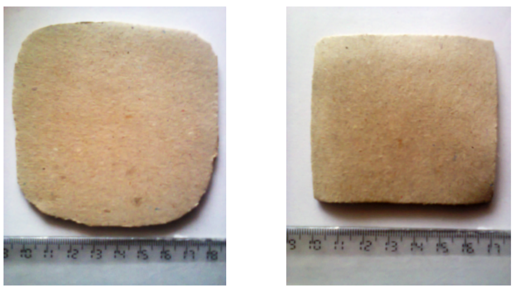
Table 2. Operating frameworks of the procedure

Framework	Version			
	1	2	3	4
Sintering temperature (ºC)	980	1120	1160	1200
Heating time (min)	26	29	36	45
Mean speed (ºC/min) - warm up - cool down	40.0 5.2	37.9 5.5	31.7 5.4	26.2 5.4
Cellular product amount (g)	217	216	218	217
Energy consuming (kWh/kg)	1.15	1.40	1.72	2.16

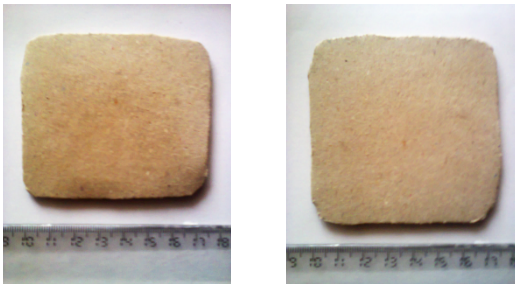
According to Table 2 data, the increase in the temperature of sintering process of colloidal suspension based on steel slag from 980 to 1200 ºC influenced the process-time, which increased from 26 to 45 min as well as the specific energy consuming of the process that increased from 1.15 to 2.16 kWh/kg. Also, the increase of sintering temperature led to reducing the mean warm up speed from 40.0 to 26.2 ºC/min.

Among the values of operating frameworks, the very high level reached by the mean warm up rate (40 ºC/min up to 980 ºC, 37.9 ºC/min up to 1120 ºC and 31.7 ºC/min up to 1160 ºC) is especially remarkable demonstrating the efficiency in energy terms of the direct electromagnetic wave warm up by comparison with the traditional warm up performance. Also, the specific energy consuming was obviously influenced by this nonconventional warm up mode.

Appearance of cellular ceramic specimens experimentally manufactured by the method described above is preented in Figure 2.



a b



c d

Figure 2. Transection of the foam samples
a – specimen 1; b – specimen 2; c – specimen 3;
d – specimen 4.

According to the images in Figure 2, the aspect of specimens indicates extremely fine porosity, designed before the experiment was performed.

The use of the characterization methods of specimens mentioned above allowed the identification of their physical-mechanical, thermal, and morphological features. The results of the measurements and determinations are indicated in Table 3.

Table 3. Physical-mechanical, thermal, and morphological features of foam specimens

Version	Apparent density ($g \cdot cm^{-3}$)	Porosity (%)	Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Compression resistance (MPa)	Absorbing the water (vol. %)	Pore dimension (mm)
1	0.29	86.2	0.060	2.4	2.2	0.10-0.16
2	0.42	80.0	0.069	4.0	2.5	0.07-0.10
3	0.59	71.9	0.078	6.3	2.4	0.05-0.08
4	0.77	63.3	0.085	9.8	2.4	0.02-0.05

Examining the data in Table 3 indicates that the manufacturing recipes in which the amount of solid steel slag particles in suspension increases and at the same time the thermal level of the sintering process is also increasing lead to a significant increase of the apparent density foamed material from 0.29 to 0.77 $g \cdot cm^{-3}$. By default, the porosity of the specimens decreases from 86.2 to 63.3 %. The explanation is that a lower solid loading leads to a lower viscosity of the suspension having as effect the incorporation of a higher amount of air into the ceramic suspension during the stirring. The decrease of porosity influences the compressive strength, whose value significantly increases from 2.4 to 9.8 MPa. The thermal conductivity of the material has low values in all tested variants within the limits of 0.060-0.085 $W \cdot m^{-1} \cdot K^{-1}$ indicating its adequate heat insulation properties. The special peculiarity of the final products is the fineness of their porosity. The pore size decreased in case of version 4 (with the highest amount of slag particles in suspension and the highest temperature) to 20-50 μm. By decreasing the sintering temperature, the pore size had the slight tendency to increase, but in extremely low limits. At lower temperatures increasing the pore size is explicable by the insufficient sintering densification. At high temperatures the pore structure is stable being adequate for high-performance thermal insulations.

The water absorption was in normal limits for a thermal insulation material (under 2.5 vol. %).

The investigation of the microstructure appearance of experimentally manufactured specimens was carried out with a Smartphone Digital Microscope. The pictures of the four specimens are exposed in Figure 3.

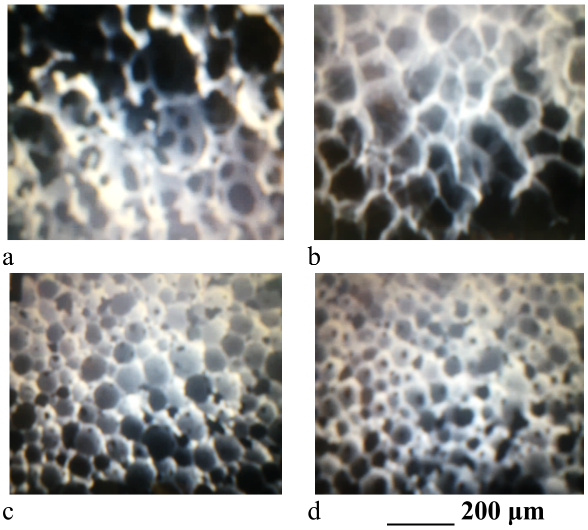


Figure 3. Microstructural configuration of foam specimens
a – specimen 1; b – specimen 2; c – specimen 3;
d – specimen 4.

Examining the images in Figure 3, it is observed that specimen 1 made at 980 ºC contains a semi-open microstructure with an intercommunication between neighboring cells. This microstructure type facilitates the low value of the apparent density, but at the same time influences the relatively low level of compression resistance.

3.2 Discussion

Ceramic foam prepared from steel slag particles in colloidal suspension is part of the wet foams category. Thermodynamically, wet cellular products are not stable due to their large facet between air and water as well as the high total free energy. The facet of the ceramic particles can be transformed adding water-soluble organic materials. The molecules of these materials are irreversibly adsorbed on the facet of the oxide powder to diminish the powder affinity for water molecules. Experimentally, it has been found that propyl gallate has the ability to convert the steel slag powder into a hydrophobic material, allowing slag particles to attach to air-water interfaces. This new technique for preparing high-volume stable foams by "in-situ" hydrophobization of particles was tested in the current paper adopting propyl gallate as an antioxidant material.

The work originality was applying de warm up technique by direct electromagnetic wave irradiation, which allowed reaching very high warm up rates (up to 40 ºC/min) without disturbing the organization at the microstructural level of the stabilized foam. The high ratio of iron oxides (15-20 % Fe_2O_3 and 2.5-5 % FeO) in steel slag powder composition with room temperature microwave susceptibility allowed efficiency microwave heating since the low temperatures [20], although the main components of slag (42-44 % CaO and 30-35 % SiO_2) are microwave transparent materials.

Extremely small pore size, low thermal conductivity, and high compressive strength of closed porosity foams recommend this type of ceramic foam as an excellent thermal insulation material for buildings.

4. CONCLUSION

In this work, ceramic foam was experimentally manufactured using steel slag powder as a raw material, distilled water to form a colloidal suspension, and propyl gallate as a foam stabilizer. The choice of the heating technique by direct microwave irradiation was the originality of the work. Wet foam obtained by stirring the suspension was dried for 48 hours and sintered in the microwave oven at successive temperatures between 980-1200 ºC. The characteristics of ceramic foam specimens were: density within the limits of 0.29-0.77 $g\cdot cm^{-3}$, porosity between 63.3-86.2 %, heat conductivity within the limits of 0.060-0.085 $W\cdot m^{-1}\cdot K^{-1}$, compression resistance in the range of 2.4-9.8 MPa as well as extremely low cell dimension in the range of 20-160 µm. The specimen heated at 980 ºC had semi-open microstructure, the other specimens having closed microstructure suitable as thermal insulation material in buildings.

REFERENCES

1. *Recycling and Reuse: Industrial By-Products*, European Union Communication, May (2007). https://www.archive.epa.gov/oswer/international/web/html/byproducts-guidance-053007.html
2. Yi, H., Xu, G., Cheng, H., Wang, J., An overview of utilization of steel slag, *Procedia Environmental Sciences,* Vol. 16, No. 6, pp. 791-801, (2012). https://doi.org/10.1016/j.proenv.2012.10.108
3. Teo, P.T., Seman, A.A., Basu, P., Shariff, N.M., Characterization of EAF steel slag waste: The potential green resource for ceramic tile production, *5th International Conference on Recent Advances in Materials, Minerals and Environment (RAMM) & 2nd International Postgraduate Conference on Materials, Mineral and Polymer (MAMIP),* University Sains, Malaysia, August 4-6, 2015, *Procedia Chemistry,* Vol. 19, pp. 842-846, (2016).
4. Cioroi, M., Nistor-Cristea, L., Recycling possibilities of metallurgical slag, *The Annals of "Dunarea de Jos" University of Galati, Fascicle IX: Metallurgy and Materials Science,* Vol. 1, pp.78-82, (2007), ISSN 1453-083X.
5. Memon, M.K., Shuker, M.T., Elraies, K.A., Study of blended surfactants to generate stable foam in presence of crude oil for gas mobility control, *Journal of Petroleum Exploration and Production Technology,* Vol. 7, No. 1, pp. 77-85, (2017). https://doi.org/10.1007/s13202-016-0243-9
6. Khristov, K., Exerowa, D., Foam stabilizing properties of surfactants determined at constant and variable pressure in the foam liquid phase, *Journal of Dispersion Science and Technology*, Vol. 18, No. 6-7, pp. 561-575, (1997). https://doi.org/10.1080/01932699708943759
7. Petkova, B., Tcholakova, S., Chenkova, M., Golemanov, K., Denkov, N., Thorley, D., Stoianov, S., Foamability of aqueous solutions: Role of surfactant type and concentration, *Advances in Colloid and Interface Science,* Vol. 276, February (2020). https://doi.org/10.1016/j,cis.2019
8. Gonzenbach, U.T., Studart, A.R., Tervoort, E., Gauckler, L.J., Stabilization of foams with inorganic colloidal particles, *Langmuir,* Vol. 22, No. 26, pp. 10983 -10988, November (2006). https://doi.org/10.1021/la061825a
9. *Propyl gallate,* National Center for Biotechnological Information, (2016). https://www.pubchem.ncbi.nlm.nih.gov/compound/Propyl-gallate
10. Reda, S.Y., Evaluation of antioxidants stability by thermal analysis and its protective effect in heated edible vegetable oil, *Cléancia e*

Tecnologia de Alimentos, Vol. 31, No. 2, pp. 475-480, June (2011).
11. Gálico, D.A., Nova, C.V., Guerra, R.B., Bannach, G., Thermal and spectroscopic studies of the antioxidant food additive propyl gallate, *Food Chemistry,* March (2015). https://doi.org/10.1016/j.foodchem.2015.02.129
12. Huo, W., Yan, S., Wu, J.M., Liu, J., Chen, Y., Qu, Y., Tang, X., Yang, J., A novel fabrication method for glass foams with small pore size and controllable pore structure, *Journal of the American Ceramic Society,* Vol. 100, No. 12, pp. 5502-5511, July (2017). https://doi.org/10.1111/jace.15089
13. Tang, F., Fudouzi, H., Uchikoshi, T., Sakka, Y., Preparation of porous materials with controlled pore size and porosity, *Journal of the European Ceramic Society,* Vol. 24, No. 2, pp. 341-344, (2004). https://doi.org/10.1016/S0955-2219(03)00223-1
14. Gonzenbach, U.T., Studart, A.R., Tervoort, E., Gauckler, L.J., Macroporous ceramics for particle-stabilized wet foams, *Journal of the American Ceramic Society,* Vol. 90, No. 1, pp. 16-22, (2007).
15. *St. John's pH adjustment,* (2018). https://www.pH-adjustment.pdf
16. Zhang, Y., Luo, X., Yang, J., Huo, W., Kang, C., A novel approach to fabricate foam ceramics from steel slag, *Advances in Materials Science and Engineering,* Vol. 2020, March (2020). https://doi.org/10.1155/2020/4649082
17. Kaptay, G., Interfacial criteria for stabilization of liquid foams by solid particles, *Colloids and Surfaces A: Physicochemical and Engineering Aspects,* Vol. 230, No. 1-3, pp. 67-80, (2003). https://doi.org/10.1016/j.colsurfa.2003.09.016
18. Gibson, L.J., Ashby, M.F., *Cellular Solids: Structure and Properties*, Cambridge University Press, 2nd edition, Cambridge, UK, (1997).
19. Yalkowsky, S.M., He Y,, Jain, P., *Handbook of Aqueous Solubility Data,* 2nd edition, CRC Press, Taylor & Francis Group, Boca Raton, USA, (2010).
20. Jones, D.A., Lelyveld, T.P., Mavrofidis, S.D., Kingman, S.W., Miles, N.J., Microwave heating applications in environmental engineering-A review, *Resources, Conservation and Recycling,* Vol. 34, No. 2, pp. 75-90, (2002). https://doi.org/10.1016/S0921-3449(01)00088-X
21. Kitchen, H.J., Vallance, S.R., Kennedy, J.L., Tapia-Ruiz, N., Carassiti, L., Modern microwave methods in solid-state inorganic materials chemistry: From fundamentals to manufacturing, *Chemical Reviews,* Vol. 114, No. 2, pp. 1170-1206, (2014). https://doi.org/10.1021/cr4002353
22. Final report on the amended safety assessment of propyl gallate, *International Journal of Toxicology,* Vol. 26, pp. 89-118, (2007), ISSN 1091-5818. https://doi.org/10.1080/10915810701663176
23. *Manual of weighing applications,* Part 1-Density, (1999). https:// www.docplayer.net/21731890-Manual-of-weighing-applications-part-1-density_html
24. Anovitz, L.M., Cole, D.R., Characterization and analysis of porosity and pore structures, *Reviews in Mineralogy and Geochemistry,* Vol. 80, No. 1, pp. 61-164, (2015). https://doi.org/10.2138/rmg.2015.80.04