EXTREMELY ATTRACTIVE NEW MATERIAL TYPE (CARBON FOAM) MADE FROM RECYCLED WOOD BIOMASS USING THE MICROWAVE IRRADIATION HEATING DURING THE CARBONIZATION STAGE

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ABSTRACT: Carbon foam, an extremely attractive new material type for important fields of application, with low density (0.28 g cm^{-3}), low thermal conductivity (0.110 W m^{-1} K^{-1}), relatively high compressive strength (3.5 MPa), high electrical conductivity (29.8 S m^{-1}), and very high electromagnetic shielding effectiveness (46 dB) was experimentally made using recycled wood biomass from industrial wood processing and aqueous solution of sucrose. The carbonization was performed by heating at 750 ºC through microwave irradiation technique, the pressed and dried material in the intermediate state being introduced into a SiC crucible with a lid, thermally protected with ceramic fiber, without inert medium. The specific energy consumption was very low (0.785 kWh/kg), unlike the high energy consumption of the usually carbonization stages. The optimal carbon foam specimen was considered suitable for the special application fields (military, aeronautics, construction, transport).
KEYWORDS: carbon foam, wood biomass, sucrose, carbonization, microwave irradiation.

1. INTRODUCTION

In the last decades, the manufacture of lightweight materials with low thermal conductivity due to foaming the raw material and at the same time with sufficiently high mechanical strength has been achieved worldwide. Glass foams from recycled residual glass are making at industrial scale in the form of different types of construction materials that compete in terms of quality with existing materials on the market [1]. Also, numerous organic wastes (agricultural, forestry, animal, etc.) constituting biomass represent cheap resources for the production of biomaterials, chemicals, etc. [2]. The tendency of manufacturers from different economy sectors for producing new materials with outstanding characteristics, low costs (materials and energy) as well as low emissions of pollutants in the atmosphere is increasingly evident.

Recently, an extremely attractive material for applications of high interest in the fields of military, aeronautics, construction, transport, etc. is the carbon foam. Representing a network of carbon atoms with a rigid, porous structure, which gives it remarkable characteristics (mechanical strength, light weight, non-flammability, resistance to high temperatures, sound and radiation absorption, electrical and thermal conductivity, low thermal expansion), the foam carbon is considered suitable for the insulation of aircraft and ships, avoiding radar detection, electrodes, fuel cells, support for catalysts, insulation for building walls, etc. Generally, the manufacturing conditions of carbon foam (high temperature and pressure) involve high costs and their reduction represents the main challenge of research in this field. American researchers are working to create lignin-based carbon foam, lignin being widely available as a by-product of the pulp and paper industry (about 70 million tons annually) through a more cost-effective process. Lignin-based foam is light, with low thermal conductivity and strong. The research team's alternative technique aims for producing the foam in open containers avoiding the high costs caused by ensuring high pressure [3].

There are two groups of carbon foam: graphitic and non-graphitic. The graphitic carbon foam has high thermal and electrical conductivity, but its mechanical strength is lower. It is produced from petroleum products, coal tar or synthetic pitches. These are easily converted into crystalline graphitic structures. The non-graphitic carbon foam is stronger, has good thermal insulation properties and lower manufacturing costs [3].

Foam made from cheaper raw materials such as coal is manufactured on a larger scale and has competitive prices in applications such as: thermal and fire protection, composite tooling, composite core, radar absorption, and electromagnetic shielding [4]. An interconnected network of open pores,

whose mechanical strength is determined by the material density and the thickness of cell walls, is required. The commercialized carbon foam produced from coal has the following properties: density between 0.27-0.40 g cm^{-3}, compressive strength over 4.8 MPa, tensile strength over 1.7 MPa, thermal conductivity in the range 0.25-25 W m^{-1} K^{-1}, electrical resistivity between 0.01-10^7 Ω cm. In the same bibliographic source it is shown that preliminary results of the pilot scale tests obtained in Touchstone Research Laboratory in Triadelphia, West Virginia, the United States indicate the achievement of superior performances of carbon foams: density between 0.048-0.192 g cm^{-3}, compressive strength of 1.4 MPa corresponding to the highest density value and up to 41 MPa for a density of 0.8 g cm^{-3}.

The manufacture of carbon foam from coal involves pyrolysis processes, which generate large amounts of carbon residues. Obvious ecological considerations lead to the need to use alternative renewable precursors in the carbon foam manufacturing process. The literature offers several variants of precursors, of which sucrose widely available in nature has begun to be studied for this objective. Sucrose contains carbon, hydrogen, and oxygen concentrated in a glucose molecule and a fructose molecule bonded together [5]. Due to the low yield of carbon, the use of only sucrose for the production of carbon foam would lead to obtaining a foamed product with low mechanical strength.

An attractive solution for improving the foam strength was the use of boric acid as a foaming agent proposed by [6]. The H^+ ion resulting from the reaction between sucrose and boric acid catalyzes OH^- in its condensation reaction, leading to the polymerization and foaming the molten sucrose. Thus, the carbon yield of the solid foam increases from 24 to 39 % with the increase in the concentration of boric acid up to 8%. The proportion of boron in the carbon foam was identified between 0.44-3.4 %. The density of the product decreased (0.1103-0.16 g cm^{-3}) with the increase of the cell size (0.67-1.17 mm) and the thermal conductivity also decreased to 0.043-0.057 W m^{-1} K^{-1} ensuring very good thermal insulation properties.

The experiment of thermal foaming of activated carbon powder into an aqueous sucrose resin followed by carbonization for the purpose of carbon foam manufacturing is shown in [7]. The condensation reactions leading to the crosslinking of sucrose polymer were delayed by the activated carbon powder. Adsorbed on the gas-liquid interface, it contributed to the stabilization of the gas bubbles, generating foaming the polymer resin. The carbon resulting from the carbonization process binds the activated carbon particles. The carbon foam had the density between 0.138-0.22 g cm^{-3}, cell size between 0.62-3 mm, and compressive strength between 0.42-3.4 MPa.

Sawdust is a batch of fine wood particles resulting as a by-product in the wood processing industry. The literature mentions the use of sawdust for the production of carbon foam [8] including birch sawdust in several stages of liquefaction, resinification, expansion, carbonization and activation. The foam had cell sizes between 100-200 μm depending on the carbonization and activation temperature. The carbon foam manufactured by liquefaction decomposed in two stages at 286 and 413 ºC. Further activation at over 800 ºC in a neutral atmosphere (nitrogen) improved the pore microstructure. It became more homogeneous by increasing the activation temperature. Through the liquefaction process, the sawdust is polymerized using phenol and formaldehyde and the foam manufacturing becomes more complicated and expensive.

In the last decade, a new orientation of research and producers in the world was exposed. Interest in renewable natural materials (sucrose, lignin, olive pit or tannin) used in the manufacture of non-graphitic carbon foam has increased significantly [9-11].

Producing method of porous carbon foam included in its recipe melted sucrose and aqueous solution of aluminum nitrate as a foaming agent [12]. The method included the following successive operations: mixing the components, their heating to 150 ºC for foaming in open mold, dehydration at 250 ºC, and carbonization in a neutral atmosphere at 900 ºC. The mixture foaming occurred due to the release of hydroxyl groups and gaseous NO_x as a result of the thermal decomposition of aluminum nitrate (between 0.5-4 wt. %). The experimental carbon foams had the density between 0.053-0.083 g cm^{-3}, cell size having values within the limits 0.83-1.55 mm. Foaming and setting process time varied between 4-24 hours decreasing with the aluminum nitrate concentration increasing between 0.5-4 wt. %.

The technique of cementing sawdust particles with sucrose ($C_{12}H_{22}O_{11}$) by filter-pressing and carbonization was tested in [13]. Sucrose, containing 42,1 wt. % carbon, 6.5 wt. % hydrogen, and 51.4 wt. % oxygen, was used in aqueous solution with the concentration between 100-700 g L^{-1} and the sawdust particles came from artocarpus hirsutus

wood, a tropical tree native to India. The density of the carbon foam had values between 0.17-0.35 g cm^{-3}, thermal conductivity was between 0.12-0.20 W m^{-1} K^{-1}, compressive strength between 0.24-3.2 MPa, electrical conductivity between 18.2-33.8 S cm^{-1}, and electromagnetic shielding effectiveness in the range 25-53 dB.

The current work represents the contribution of authors on the making process of carbon foam. The main element of this paper originality is the carbonization mode of raw material mixture after removing it from the mold in which it was pressed and dried. Due to the adoption of the nonconventional fast and economic microwave heating technique with low energy losses outside the heating zone in the oven, the optimal temperature of the process was limited to 750 ºC. The raw materials chosen for this experiment were the aqueous solution of sucrose in which an industrial by-product of wood processing (acacia sawdust) was dissolved. Next, the peculiarities of the mentioned process are presented.

2. METHODS AND MATERIALS

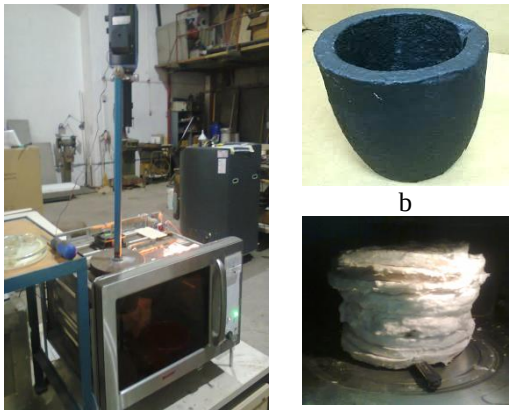
2.1 Methods

The method adopted by the authors for the manufacture of carbon foam uses two available natural materials: one (acacia sawdust) is a by-product of wood processing and the other (sucrose) is found naturally in fruits and vegetables (sugar cane, sugar beet, apple, orange, carrot, etc.) and is the raw material for the industrial production of sugar intended for human consumption. The two types of materials ensure the ecological character of the adopted method.

In technological terms, the aqueous sucrose solution has the ability for binding the finely ground sawdust (under 200 μm) dissolved in the sucrose solution and the resulting slurry is consolidated by pressing in a mold and is then air-dried at over 100 ºC. The following technological operation has an original character compared to similar techniques used in the world. The carbonization process at about 750 C is preceded by the introduction of the pressed and dry mixture removed from the mold into a cylindrical crucible made of silicon carbide (SiC) provided with a lid made of the same material. The thickness of the crucible wall is 15 mm, as is the thickness of the lid. The crucible containing the raw material subjected to carbonization, as well as the lid is effectively thermally protected with high temperature resistant ceramic fiber mats (up to 1200 ºC) and they are placed into a microwave oven of the type used in the household, but constructively adapted for operation in conditions of much higher thermal stress. Unlike the usual methods for carbonization, in this case a neutral atmosphere is not created because the material is closed relatively tightly in the crucible with the lid and the free internal volume is sufficiently dimensionally limited, thus avoiding keeping a dangerous amount of atmospheric air for the self-ignition of carbonaceous material. The oven is powered by a single 800 W-magnetron, the waveguide being placed in one of its side walls. SiC is known as an excellent microwave susceptible material. It heats up quickly and efficiently under the influence of the electromagnetic waves emitted by the magnetron and transfers the thermal energy to the carbonaceous material. Determining the temperature of the material subjected to carbonization in the microwave oven was indirectly performed by measuring the temperature on the surface of the SiC lid (on an area without ceramic fiber coating) and applying the pre-established correlation between this value and that of the material inside the crucible. The temperature on the lid was measured with the Pyrovar type-radiation pyrometer through the 30 mm-hole in the upper wall of the oven.

The literature mentions the use of unconventional microwave heating in some pyrolysis and hydrothermal carbonization processes for making high density-solid biofuels using biomass as raw material [14]. The critical analysis notes the superior efficiency of this heating type by comparison with conventional heating methods, but the comparison of techniques mentioned in [14] with that presented in the current work indicates their inferior energy-technological performances.

The main components of the microwave equipment made for the carbonization process are shown in Figure 1.



a b c

Figure 1. Microwave equipment for the carbonization process
a – overall image of the equipment; b – SiC crucible; c – thermal protection of crucible and lid.

2.2 Materials

Acacia wood is strong, durable, and stable being often used for construction, furniture, and other applications. The wood powder (sawdust) was provided by a furniture manufacturing workshop. Its subsequent processing was carried out by grinding in a ball mill, the grain size being reduced below 200 μm.

The natural sucrose ($C_{12}H_{22}O_{11}$) was purchased from the market as a crystalline powder. Sucrose was dissolved in water in three experimental variants of its concentration: 700, 750, and 800 g L^{-1}.

The acacia sawdust was dissolved in the aqueous sucrose solution and the material in form of slurry was then poured and pressed into the mold in order to performer the foam carbonization.

2.3 Methods for characterization the carbon foam specimens

The density was measured using the gravimetric method [15]. Determining the thermal conductivity (at 30 ºC) was made with HFM 446 Lambda apparatus based on the heat-flow method (SR EN 1946-3:2004). The compressive strength was measured using 100 kN-hydraulic axial press machine (EN 826:2013). The microstructural characteristics of specimens were investigated with Biological Microscope MT5000 model with captured image, 1000 x magnification. Electrical conductivity was determined by adopting an experimental method proposed by the research team from Belgium [16] using an original laboratory device. The electromagnetic shielding performance of carbon foam specimens was measured using the nested reverberation chamber method in the frequency band 1–4 GHz [17].

3. RESULTS AND DISCUSSION

3.1 Results

Three experimental variants for making carbon foam specimens were chosen. The total amount of dry solids, including sucrose and acacia sawdust, was adopted at the value of 350 g and was kept constant for all tested variants. The compositional differences were the concentration of sucrose in water (between 700-800 g L^{-1}) and the weight ratio between sucrose and sawdust which was varied between 56.9/43.1 and 59.0/41.0.

The composition of experimental variants is shown in Table 1.

Table 1. Composition of experimental variants

Material	Variant		
	1	2	3
Sucrose - amount (g)	199.2	202.3	206.5
- dry material weight ratio (wt. %)	56.9	57.8	59.0
Acacia sawdust - amount (g)	150.8	147.7	143.5
- dry material weight ratio (wt. %)	43.1	42.2	41.0
Total dry solid amount (g)	350	350	350
Distilled water amount (g)	284.5	269.7	258.1

The carbonization step of carbonaceous material removed from the mold after pressing and drying has a major importance for the microporous structure of the carbon foam. According to the literature [18], generally, increasing the carbonization temperature of wood in the absence of air between 400-1000 ºC or even higher increases the volume of micropores and the yield of carbon by comparison with wood carbonized at low temperature. The absence of oxygen from the atmosphere in which the carbonization takes place is essential to avoid the ignition and burning the carbon in composition of material subjected to this process. In the current experiment, the optimal carbonization temperature was pre-determined at 750 ºC and this value was used in all three tested variants. The nonconventional microwave heating process into SiC crucible allowed a low time process (about 24 min), the heating rate being extremely high (around 30 ºC/min) compared to the specific parameters of conventional heating processes.

In terms of energy, it should be mentioned that previous measurements carried out on the 800 W-microwave oven showed that the effectively dissipated microwave power inside the oven is approx. 625 W (i.e. 78.1 %). On the other hand, the measurement of the amount of carbon foam specimens produced in this experiment indicated the following values: 318.5 g (variant 1), 325.5 g (variant 2), and 332.5 g (variant 3), respectively. So, the weight loss during the thermal carbonization process varied depending on the sucrose concentration (between 700-800 g L^{-1}), decreasing with the increase of its value from 9 % (variant 1) to 5 % (variant 3). The specific consumption of energy measured and calculated for the three variants had the following values, according to the data in Table 2.

Table 2. Calculation of specific energy consumption

Date	Variant		
	1	2	3
Sucrose concentration (g L^{-1})	700	750	800
Carbon foam amount (g)	318.5	325.5	332.5
Weight loss (wt. %)	9	7	5
Process time (min)	24	24	24
Specific energy consumption (kWh/kg)	0.785	0.768	0.752

According to the data in Table 2, the values of specific energy consumption were very low (0.752-0.785 kWh/kg) being influenced by the weight loss of material. However, the comparison with energy consumption values in similar processes in the world is not possible because this efficiency parameter is not indicated in the literature.

Applying the advanced technique of carbonization by electromagnetic wave heating led to obtaining the following physical, thermal, mechanical, and microstructural characteristics of experimentally made carbon foam in the three variants (Table 3).

Table 3. Main features of carbon foam

Feature	Variant		
	1	2	3
Density (g cm^{-3})	0.28	0.25	0.20
Thermal conductivity (W m^{-1} K^{-1})	0.110	0.104	0.097
Compressive strength (MPa)	3.5	3.3	2.7
Electrical conductivity (S m^{-1})	29.8	27.9	25.2
Electromagnetic shielding effectiveness (dB)	46	40	31
Interconnected cell size (μm)	180-260	210-480	250-520

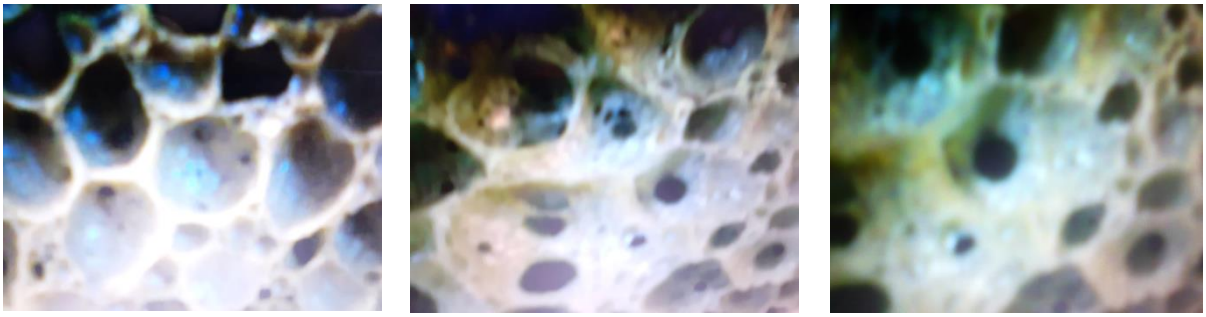
The characteristics of carbon foam specimens in Table 3 show that values of their density were low (between 0.20-0.28 g cm^{-3}), suggesting, however, that their structure is not of the reticular type, which due to the completely open cells allows reaching extremely low density values (below 0.10 g cm^{-3}). The carbon foam produced during the experiment described above achieved an adequate correlation between the thermal conductivity that determines the heat-insulating properties of the material and the compressive strength that must have acceptable values for the application fields mentioned in this paper. Thus, the thermal conductivity was in the range of 0.097-0.110 W m^{-1} K^{-1} and the compressive strength was within the limits 2.7-3.5 MPa. The density of the foam also influenced the electrical conductivity and the electromagnetic shielding effectiveness, their maximum values (29.8 S m^{-1} and 46 dB, respectively) being obtained at the highest density value of 0.28 g cm^{-3} corresponding to variant 1. Especially, the high value of the electromagnetic shielding effectiveness is remarkable.

Appearance of the three variants of carbon foam specimens is presented in Figure 2.

a b c

Figure 2. Appearance of carbon foam specimens
a – variant 1; b – variant 2; c – variant 3.

To identify the microstructural differences of the three carbon foam specimens, they were examined under the microscope. The obtained images are shown in Figure 3.



a b c

Figure 3. The microstructural appearance of carbon foam specimens
a – variant 1; b – variant 2; c – variant 3.

The type of carbon foam made in this experiment is characterized by interconnected cells communicating with each other through the wall that separates them, but the thickness of the wall and existing struts between the cells contribute to achieving a network with acceptable compressive strength. The experiment was focused towards obtaining semi-open cellular structures, in which the cell keeps the identifiable geometric aspect of the closed cell, with the difference that the degree of interconnection of neighboring cells is high.

Comparing the images representing the microstructure of the three specimens, it can be seen that image (a) corresponding to variant 1 has a more uniform distribution of cells compared to the other variants, the cell size is lower and the size variation range is very low (180-260 μm). Also, the free spaces in the cell walls are much lower and therefore the communication with neighboring cells is less developed. The images (b) and (c) are characterized by larger cellular structures with higher cell size and free spaces in the wall much larger allowing the more active inter-communication between cells. The range of cell size is 210-480 μm (variant 2) and 250-520 μm (variant 3).

3.2 Discussion

Carbon foam is an extremely attractive new material for very important fields of application (military, aeronautics, construction, transport, etc.). However, the manufacturing process of this very expensive product due to the conditions required for production (high pressure and temperature, neutral atmosphere) decisively influenced the low level of spreading the carbon foam production in the world. That's why the recently exposed tendency to reduce material and energy costs as well as pollutant emissions represents the main challenge of research.

According to other experiments described in the literature, methods of dissolving powders in aqueous solutions with the role of catalyst and generating slurries have proven to be suitable for the carbon foam production. Therefore, a similar method was applied in this experiment. Among the three experimental variants tested, variant 1 in which the sucrose concentration in the aqueous solution was 700 g L^{-1} was chosen as the optimal variant after comparing the physical, thermal, mechanical, and microstructural characteristics with the other variants. Recycled acacia wood as a by-product of an industrial wood processing process was used in 43.1 wt. % ratio as a biomass, its production not being pollutant for the atmosphere.

The making process of carbon foam has an especial peculiarity compared to the foam manufacture of silicate or aluminosilicate waste [1, 19]. The high carbon content of the raw material, be it is coal or other carbonaceous materials, implies the carbonization of the intermediate product (obtained after pressing and drying) in the absence of air. Usually, one opts for creating an inert environment by blowing nitrogen into the oven. The solution adopted in this experiment was a particular one, in which the intermediate product was closed almost hermetically in a crucible with a lid, thermally insulated with ceramic fiber mattresses and heated in the oven by microwave irradiation. The method had the expected results, i.e. the carbonization of the carbonaceous material could be carried out without ignition and burning the carbon.

The carbon foam obtained after the slow cooling of the final product had specific characteristics of this type of foam made by applying the usual protection methods during carbonization. In addition, by using the microwave irradiation heating technique, the making process was very economic (0.785 kWh/kg), thus solving one of the causes of high cost of technological process.

4. CONCLUSION

Very attractive new material for special applications (military, aeronautics, construction, transport) as carbon foam was experimentally made from recycled acacia sawdust (biomass) as an industrial by-product and aqueous sucrose solution. Carbonization of the slurry resulting from this mixture, pressed and dried, was original being economically and quickly done at 750 ºC by microwave irradiation in a closed SiC crucible without an inert medium. The optimal carbon foam had density of 0.28 g cm^{-3}, thermal conductivity of 0.110 W m^{-1} K^{-1}, compressive strength of 3.5 MPa, electrical conductivity of 29.8 S m^{-1}, and electromagnetic shielding efficiency of 46 dB.

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