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Methods for determining the characteristics of biocomposites

Jovana Bojković[1*], Vesna Bulatović[2], Branko Radičević[1], Nenad Stojić[1], Miljan Marašević[1]
[1]Faculty of Mechanical and Civil Engineering, University of Kragujevac, Kraljevo (Republic of Serbia)
[2]Faculty of Technical Science, Department for Civil Engineering, University of Novi Sad, Novi Sad (Republic of Serbia)

The use of biocomposite materials in the world is rapidly developing. The production of new biomaterials has made a big shift towards sustainable production, which also has a positive effect on ecology. Biocomposites are defined as a combination of two or more different materials, each of which has its characteristic properties. To define the use of a new biocomposite, it is necessary to examine its characteristics as a whole. The characteristics of that biocomposite are defined by the application of methods for extracting physical and mechanical properties, more precisely, porosity, bulk density, water absorption, airflow resistance, and heat flux, from which the coefficient of thermal conductivity is calculated. In this way, it is possible to determine the exact application of the material and compare it with long-used materials in the defined area of application. Therefore, new biocomposite materials can be used as an alternative to conventional materials.

Keywords: Biocomposite, Porosity, Bulk density, Thermal conductivity, Airflow resistance

1. INTRODUCTION

Reduction of CO_2 emissions, energy saving, and increasing application of biocomposite materials is becoming increasingly common area of research for many researchers around the world [1]–[4]. To improve the previously mentioned facts, the properties of biocomposite materials are tested. These tests are performed to define the area of application of the material. Pochwała et al. [5] studied the thermal conductivity of biocomposites, while Curto et al. [6] studied the thermal and acoustic characteristics of composite materials made from lime as binder and hemp as aggregate. Brzyski et al. [7] examined the physical and mechanical properties of the same biocomposites. Tests have shown that changing the ratio of the amount of binder and aggregate affects the mechanical (flexural strength) and physical properties (porosity, density, thermal conductivity...) [8], [9]. By examining the characteristics of composite materials on a biological basis, it can be established whether they can be used as an adequate substitute for already used materials with similar properties. Such materials can be used as components in the production of lightweight concrete [10]–[12] or as insulating materials from a thermal and acoustic point of view [13], [14]. How the properties of biocomposites, such as porosity, affect their application was investigated [15]. The effect of the thickness of the samples, the type of aggregate filler, and its volume fraction in the mixture, on the thermal and acoustic properties of the biocomposite was examined by Bojković et al. [16].

The results presented in the previously mentioned works define the possibility and area of application of the new biocomposite material, most often created by the maximum utilization of biomass. In this way, sustainable management of the used type of waste will be ensured, to protect the environment and give new use value to this material following the principles of sustainable development in construction. This paper aims to present methods for determining the characteristics of biocomposite to more easily define its application, durability, and the possibility of replacing the previously used but expensive construction material. On the other hand, since they are biomaterials, their application would have a positive effect on the environment.

2. MATERIALS AND METHODS

2.1. Material

The experimental part of the research, which is carried out to determine the characteristics of the material to define its application, is shown on a biocomposite made by combining wood waste (sawdust), Styrofoam whose granule size is 0.3 cm, and a mixture of lime and gypsum as a binder. The samples were formed using circular cross-section molds, 30 mm thick and 110 mm in diameter (Figure 1). The test was performed after 28 days of drying of the samples at a room temperature of 20±2°C.

Figure 1. Samples

2.2. Methods for determining airflow resistance

One of the main non-acoustic parameters that show the behavior of porous materials (biocomposites), which are used in sound insulation systems, is airflow resistance. A characteristic such as this is determined before the material enters the production process. According to the SRPS ISO 9053 [17] standard, there are two methods for measuring airflow resistance, namely the constant flow method and the variable airflow method. The constant air flow method (Figure 2) is based on the passage of a one-direction airflow through a sample of the tested material that is in the shape of a rectangular parallelepiped or a circular cylinder. The resulting pressure drop is measured between the two free

*Jovana Bojković: Dositejeva 19, 36000 Kraljevo, Republic of Serbia, bojkovic.j@mfkv.kg.ac.rs

surfaces of the sample while the airflow is created using a vacuum pump. The pressure drop is measured using a differential pressure gauge. Different from the previous method, the method with variable airflow (Figure 3) is based on a slow changing of airflow through a material sample of the same shape as in the previously mentioned method and measuring the alternating components in the volume enclosed by the sample. An alternating airflow rate is produced by a piston while the alternating pressure in the sample holder is measured by a side-mounted condenser microphone connected to a measurement amplifier.

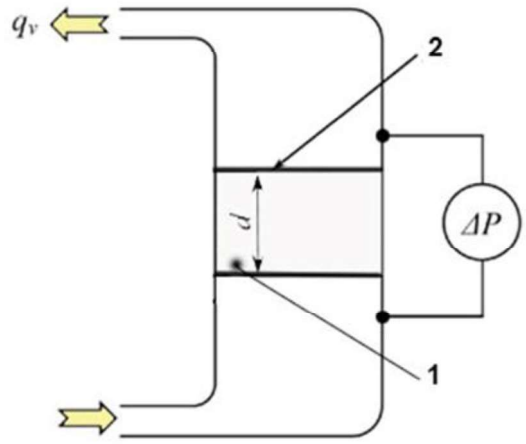

Figure 2. Constant airflow method

Description of Figure 2:
1. Porous material-sample
2. Cross-section of the sample
qv - volumetric air flow
d - thickness of the material sample
ΔP - pressure change

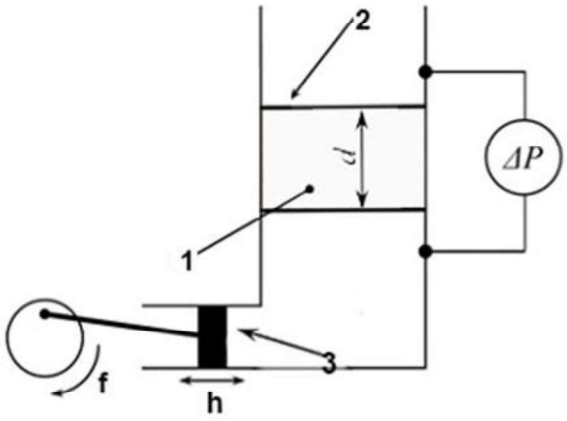

Figure 3. Variable airflow method

Description of Figure 3:
1. Porous material - sample
2. Cross-section of the sample
3. Cross-section of piston
d - sample thickness
ΔP – pressure change
f – frequency
h – the stroke of the piston

2.2.1. Apparatus for measuring flow resistance with constant airflow

In the method with constant airflow, which is more often applied in practice, the airflow is realized using a vacuum pump consisting of two air flow meters that work on the principle of a rotameter with a ball. Through the throttle valves, the airflow is adjusted for each rotameter individually. With one meter, the airflow varies in the range of 0.2-6 l/min, while air flows of 5-32 l/min are adjusted on the other meter. The vacuum pump makes it possible to reach the speed of the airflow in the "measuring cell" in which the sample of the material to be tested is placed and which is following the recommendations of the SRPS ISO 9053 standard [17]. "Measuring cell" is a plexiglass tube that is closed on one side to ensure conditions for maintaining negative pressure. The length of the tube is 300mm and the inner diameter is 100mm. By placing the sample in the cell and turning on the vacuum pump, negative pressure is created on one side while atmospheric pressure is on the other side of the sample. The difference between these two pressures is measured using a differential gauge. The measuring chain for determining the airflow resistance through the sample is shown in Figure 4.

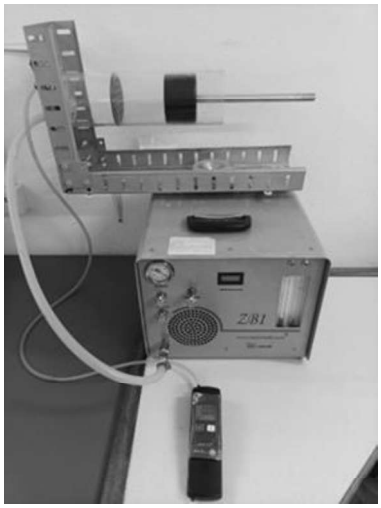

Figure 4. Measuring chain for determining airflow resistance

The pressure drop is a difference between the atmospheric pressure on one end of the sample and the sub-pressure created by the vacuum pump on the other. The ratio of the measured pressure drops (Δp) to the volumetric flow rate (q_v) that drop is the airflow resistance (R), which is calculated by the following formula:

$$R=\Delta p/q_v \quad (1)$$

The ratio of the airflow resistance to the sample cross-section area is the specific airflow resistance (R_s),

$$R_s=R/A \quad (2)$$

The specific airflow resistance (R_s) is the basis for the calculation of the longitudinal (specific) airflow resistance (r) for the corresponding sample thickness by means of the formula:

$$r = R_s/d \quad (3)$$

2.3. Methods of measuring thermal conductivity

There are two groups of methods for experimental testing of thermal conductivity, stationary and non-stationary. In stationary methods, thermal conductivity is obtained by direct measurement of heat flux and temperature on the surface of the sample when a stationary state is reached. In the second group of methods, non-stationary, the temperature distribution changes over time, and the rate of temperature change is also measured. This measurement determines the thermal diffusivity of the material. By knowing the density of the tested material, specific heat, and thermal diffusivity, its thermal conductivity can be calculated. Both groups of methods for determining thermal conductivity have numerous advantages and disadvantages [18]. In general, stationary methods are somewhat more complex than non-stationary ones, that is, the time interval for measuring thermal conductivity is quite long, the measuring equipment is complex and there are often difficulties during measurements that may arise as a result of contact thermal resistance [19]. In contrast to stationary, non-stationary methods are characterized by a shorter time interval of measurement, simpler apparatus, and smaller samples [20]. Depending on the type of material, the geometry of the sample, and the demanding measurement accuracy, the appropriate test method is selected.

The group of stationary methods for determining thermal conductivity include [16]:

- Method with flux meter,
- Method with direct electric heating,
- Method of a protected hot plate,
- Method of radial heat flow,
- Method with axial heat flow,
- Comparative method with axial heat flow (cut-bar method),

while the group of non-stationary methods includes:

- Pulse transient method,
- Laser pulse method,
- Transient flat source method,
- Line heat source method (hot wire method),
- 3ω method.

Among the listed methods, the stationary method with a fluxmeter is most often used in practice. The advantages of this method are the wide range of tested materials as well as the speed of obtaining results. The fluxmeter method is following the European standard EN12667 [21]. The measuring apparatus consists of two plates between which the sample is placed. One plate is heated, the other is cooled, and the heat flux is measured using a flux meter. A schematic representation of the method is given in Figure 5.

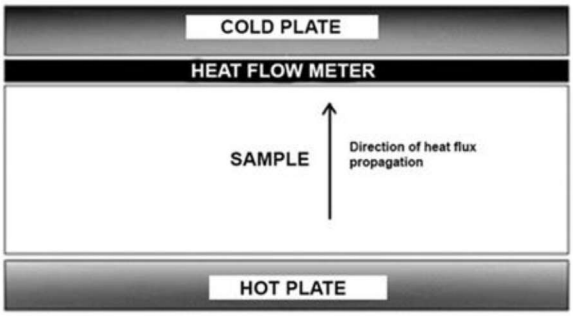

Figure 5. Schematic representation of the method

Based on the measured heat flux values, the thermal conductivity coefficients can be calculated according to the following formula:

$$q = \frac{t_1 - t_2}{\frac{\delta}{\lambda}} \quad (4)$$

$$\lambda = \frac{q \times \delta}{t_1 - t_2} \quad (5)$$

where q represents the heat flux, λ the coefficient of thermal conductivity, δ the thickness of the sample, while t_1 and t_2 are the measured temperatures on one and the other surface of the sample.

2.4. Determination of porosity and water absorption

Porosity is defined as the presence of empty spaces in the structure of a material and can be divided into two groups: pores, which are not visible to the naked eye, and cavities, which are much larger and clearly visible. Pores and cavities can be open or closed depending on their connection and are most often irregular in shape. The porosity of the material is expressed by the porosity coefficient φ. In order to determine the porosity of the biocomposite, the samples are first placed in a tank where they are vacuumed under pressure for a time interval of 3 hours. Then water is poured into the tank, which completely covers the samples of the biocomposite being tested, while the vacuuming continues for another 1 hour. After that, the pump is turned off and the vacuuming process continues for another 18±2 hours until the samples are completely saturated with water. Then the saturated weight of the samples is measured in air (m_{sat}) and hydrostatic balance (m_{hydr}). Finally, the samples are dried in an oven until their weight stabilizes, after which they are measured again (m_{dry}). This procedure for determining the porosity of a material is in accordance with the research done by Lagouin et al. [22].

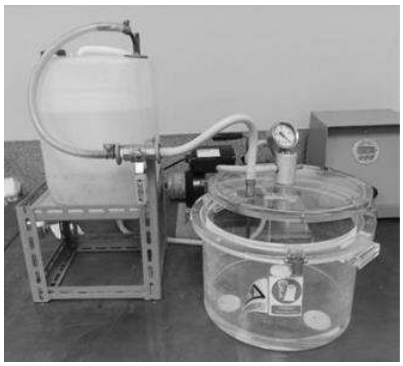

Figure 6. Vacuuming system

Figure 7. Sample holder

Material porosity and water absorption are calculated based on measured mass values according to the following formulas:

Porosity

$$\varphi = \frac{m_{sat} - m_{dry}}{m_{sat} - m_{hidr}} \cdot 100 \quad (6)$$

Water absorption

$$U_v = \frac{m_{sat} - m_{dry}}{m_{dry}} \cdot 100 \quad (7)$$

Bulk density is determined based on the volume of an irregularly shaped body (V) according to [23]:

$$\gamma = \frac{m_{dry}}{V} \quad (8)$$

3. RESULTS AND DISCUSSION

Based on the measured and calculated values shown in Table 1, it can be concluded what kind of biocomposite it is, that is, its application can be defined. The mutual dependence of all parameters starting from porosity, water absorption, heat flux, and longitudinal resistance of airflow is noticeable in the tested samples. For example, water absorption depends on porosity, that is, the higher the porosity of the samples, the higher the water absorption, and vice versa. As the porosity increases, the longitudinal resistance to airflow and the heat flux decrease. The differences occurring in the measured and calculated values for three samples of the same thickness can be subsumed under the standard deviation (SD) (Table 2).

By determining the characteristics listed in Table 1, it can be defined whether this biocomposite can be used as a sound or heat insulator. More precisely, based on the value for the longitudinal (specific) resistance of the airflow, it can be concluded what the material is in terms of sound insulation. The values of the absorption coefficient of the material can be calculated based on the longitudinal (specific) resistance of the airflow, using various theoretical models. On the other hand, heat flux and thermal conductivity coefficient define this biocomposite from the aspect of thermal insulation. Comparing it with Styrofoam, whose thermal conductivity coefficient ranges from λ = 0.035 - 0.040 W/mK, it can be concluded that this biocomposite has a three times higher thermal conductivity coefficient, that is, it is a worse thermal insulator than Styrofoam. In a similar way, this biocomposite can be defined in terms of sound insulation.

By comparing and analyzing the obtained results, linear correlations between porosity and flow resistance as well as porosity and coefficient of thermal conductivity were made. Based on the values, it can be concluded that an excellent correlation has been established between the previously mentioned values for the mean value of biocomposite P with a thickness of 3 cm, where the values of the correlation coefficients are R^2 = 0.9986 and R^2 = 0.9857 (Figures 8 and 9). However, the correlation between porosity and volumetric mass did not turn out to be good and amounts to R^2 = 0.3229 (Figure 10).

Table 1. Measurement results for biocomposite P

Mark	Sample thickness	Porosity	Bulk density	Absorbing water	Heat flux	Thermal conductivity coefficient	Longitudinal specific resistance to air flow
	d (cm)	**φ (%)**	**γ (g/cm³)**	**Uv (%)**	**q (W/m²)**	**λ (W/m·K)**	**r (kPa·s/m²)**
P1	3	57.268	0.637	91.652	40.3	0.121	480.636
P2	3	57.881	0.632	91.857	40.18	0.119	479.980
P3	3	56.913	0.620	89.897	40.51	0.122	481.221
P_{medium}	3	57.3	0.629	91.135	40.3	0.121	480.612

Table 2. The standard deviation for measured values

		STDEV
Porosity	φ	0.490
Bulk density	γ (g/cm³)	0.009
Absorbing water	Uv	1.077
Heat flux	q (W/m²)	0.167
Thermal conductivity coefficient	λ (W/mK)	0.002
Longitudinal specific resistance to air flow	r (kPa·s/m²)	0.621

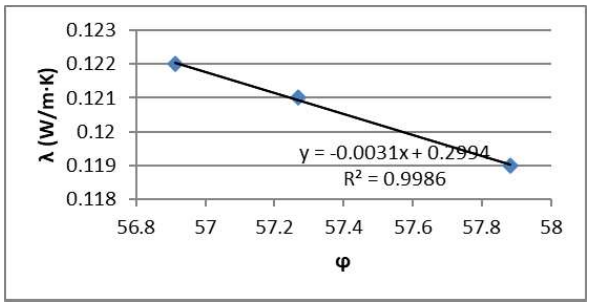

Figure 8. Linear correlation φ-λ

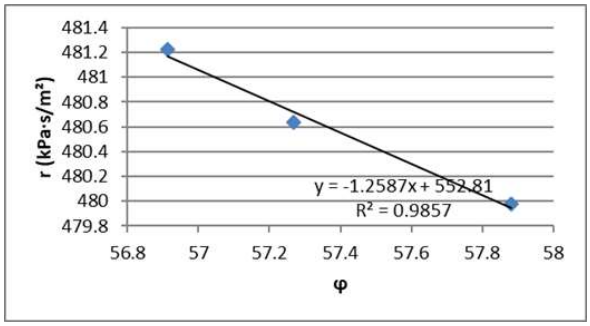

Figure 9. Linear correlation φ-r

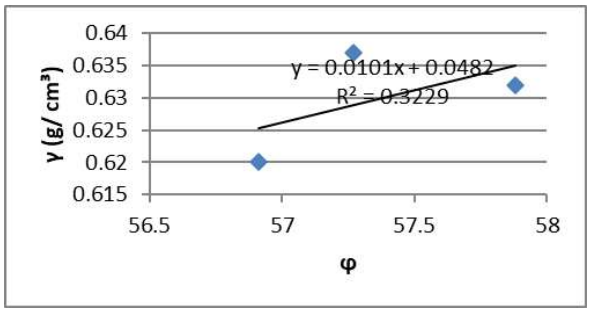

Figure 10. Linear correlation φ-γ

4. CONCLUSION

The increasingly frequent use of bio-based composites makes a big shift towards sustainable production and has a positive impact on the environment. Using waste and by-products from other industries, such as sawdust, which is generated as waste from the wood industry, enables additional use value for waste material and creates conditions for sustainable development in this segment of the economy. The tests mentioned in this paper can define the exact application of the new biocomposite. However, the same tests can also define the negative side of the tested material. More precisely, it was determined that the biocomposite made from wood industry waste material, Styrofoam granules and lime and gypsum as a binder, is a good sound and poor heat insulator. High values of air flow resistance and thermal conductivity coefficient support this key. This fact can determine the path of further research. By changing the ratio of materials in the biocomposite, different values of the tested properties will be obtained and, in this way, the optimal mixture can be defined in terms of sound or thermal insulation.

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