Indirect evaluation of the porosity of waste wood briquettes by measuring the surface roughness

Daniela Sova\textsuperscript{1*}, Lidia Gurau\textsuperscript{2}, Mihaela Porojan\textsuperscript{2}, Olivia Florea\textsuperscript{3}, Venetia Sandu\textsuperscript{1}, Monica Purcaru\textsuperscript{3}

\textsuperscript{1} TRANSILVANIA University of Brasov, Department of Mechanical Engineering, 1 Politehnicii Str., 500024 Brasov, Romania, Tel. +40 268 474761

\textsuperscript{2} TRANSILVANIA University of Brasov, Department of Wood Processing and Wood Products Design, 1 Universitatii Str., 500068 Brasov, Romania, Tel. +40 268 415315

\textsuperscript{3} TRANSILVANIA University of Brasov, Department of Mathematics and Computer Science, 50 Iuliu Maniu Str., 500090 Brasov, Romania, Tel. +40 268 414016

\textsuperscript{*} Corresponding author: Tel. +40 726153965.

E-mail address: sova.d@unitbv.ro (Sova Daniela).

Address: TRANSILVANIA University of Brasov, Department of Mechanical Engineering, 1 Politehnicii Str., 500024 Brasov, Romania.
Abstract

The briquette porosity is a quality characteristic known to be important for combustion analysis, heat and mass transfer processes during combustion stages, determination of effective thermal conductivity or other related properties. This paper describes a method to quantify the briquette porosity by some surface roughness parameters that can be useful for alternative, inexpensive and at hand evaluations. Porosity of briquettes manufactured with a hydraulic press from waste wood from secondary processing was calculated with three methods suggested in the literature for wood; of these, one was adapted here for a wet porosity model (called “general relation”) proposed for wood briquettes. Briquettes density was obtained by using two stereometric methods and a liquid displacement method. Correlations were examined between porosity, surface roughness parameters and density of briquettes. Very strong correlations with surface roughness were identified for porosity calculated with all three methods, when density was measured by one of the stereometric methods. These correlations can serve as a method to indirect evaluation of the briquettes porosity by measuring the surface roughness.

Statement of novelty

Porosity is important for the analysis of briquettes combustion; therefore it would be interesting to see if this property can be indirectly evaluated by another method, such as by measuring some roughness parameters of the briquette surface in connection with briquette density. Based on previous studies carried out on wood, it can be assumed that both porosity and roughness parameters are properties depending on density. Porosity and density were determined in the present study by using three different methods. The experimental data contribute to the existing literature on briquettes properties by adding the surface roughness. The novelty of the present paper consists in extending the applicability of the porosity models originally developed for wood, to the wood briquettes. To the best knowledge of the present authors, the wet porosity model applied to briquettes has not been reported before.
Keywords: waste wood briquette, density, porosity, surface roughness

Nomenclature

$m, M$: mass (kg)

$MC$: moisture content (%)

$n, P$: porosity

$Ra$: arithmetical mean deviation of the assessed profile (arithmetic mean of the absolute ordinate values within a sampling length) ($\mu$m)

$Rk$: core roughness depth (depth of the core profile within an evaluation length, excluding the height of the protruding peaks and deep valleys) ($\mu$m)

$Rpk$: reduced peak height (average height of the protruding peaks above the roughness core profile) ($\mu$m)

$Rvk$: reduced valley depth (average depth of the valleys projecting through the roughness core profile) ($\mu$m)

$Rk+Rpk+Rvk$: combines the previous three profile heights, based on the Abbot-curve height distribution. This is not a standard parameter, but it was derived from the standard parameters above, with the purpose of covering the magnitude of all three. ($\mu$m)

$V$: volume ($m^3$)

$V\%$: volume fraction

Greek symbols

$\Delta$: error

$\phi$: porosity

$\rho$: density ($kg/m^3$)

Subscripts
1. Introduction

Biomass, like agricultural straws and grasses, wood chips and sawdust, is an attractive feedstock because of its renewability, abundance and positive environmental impact. Biomass is difficult to handle, transport, store and utilize in its original form due to the high moisture content, irregular shape and sizes and low bulk density. Densification can generate products with uniform shape and sizes that can be more easily handled and thereby reduce cost associated with transportation, handling and storage [1, 2, 3, 4]. Conventional processes for biomass densification can be classified into baling, pelletizing, extrusion, and briquetting. Among them, pelletizing and briquetting are the most common processes used for biomass densification for solid fuel applications. During briquetting the biomass particles self-bond to form a briquette, due to the thermoplastic flow. The briquettes’ densities generally range from 900 to 1300 kg/m³ [5]. The five basic categories of biomass materials include: virgin wood, energy crops, agricultural residues, food wastes, industrial wastes and co-products [6]. Biomass from wood originating from forest residues or as waste resulting from wood industries is one of the most universal renewable sources of energy [7, 8].
Usta [9] defined wood as a cellular/porous material composed of cell wall substance and cavities containing air and extractives. Without cavities and intercellular spaces the density of the cell wall substance is constant for all timbers (1530 kg/m³ on an oven-dry mass and volume basis). However, wood does not entirely consist of cell wall substance because it contains air pockets in the cell lumens. Therefore, the amount of cell wall substance (K) is a function of wood density, d (K = d/1530). The void volume (porosity, P) is defined in relation to the cell wall substance (P = 1-K).

Siau [10] regarded wood cells as a rectangular model of square cross section with unit overall dimensions (Fig.1a). All cells are equally sized and the ends of the cells are neglected. The cell lumen also has a square cross section. The model refers to cells at oven-dry conditions, where the lumen has only dead air. A more general model of wood cells that considers the wood moisture content was described by Siau [10], as well as by Hunt et al. [11], where the bound water is added as a surrounding area to the outside of the squared cell cross section having the side equal to unity (Fig.1b).

Porosity is one of the physical properties of wood and wood briquettes, which is important for combustion analysis and modeling, heat and mass transfer processes during combustion stages, determination of effective thermal conductivity and effective heat capacity or other related properties, such as density or durability, transportation and storage of briquettes, and wood impregnation with preservatives [1, 11, 12, 13, 14].

Fig. 1. Wood cell model (cross section)
a) oven-dry conditions, b) wet conditions
Wood porosity can be determined by application of pycnometric methods, displacement of various liquids and mercury intrusion porosimetry [14, 15, 16]. However, wood has a different configuration than wood briquettes and can be submitted to different methods of porosity measurement. Mercury porosimetry and gas pycnometry are usually used to estimate the pore size distribution and porosity of wood. According to Moura et al. [15], the mercury porosimetry is suitable to evaluate the porosity of wood, pulp and paper, being a valuable tool to anticipate properties like surface roughness, air permeance or coating distribution. A limitation of the porosimeter that the authors have used is that voids with diameters below about 0.007 μm are not detected.

Brewer et al. [17] calculated the porosity of biochar by means of skeletal density, determined by helium pycnometry and envelope density, by using an envelope density analyzer. Additional methods were required since some macro-voids were inaccessible to helium gas.

More recently, research work [18] applied computed tomography (CT) and backscattered electron (BSE) imaging methods to investigate and quantify the porosity of wood (bamboo cross section). The authors of the research compared different methods of porosity measurement, such as mercury intrusion porosimetry, gas pycnometry, microscopy image processing and computed tomography, and evaluated the strengths and the limitations of each method. Considering the strengths and limitations of the existing porosity measurement techniques, they decided to simultaneously use two methods, SEM and CT scanning methods, in order to investigate the porosity distribution as function of orientation within bamboo wood.

In contrast, fewer methods have been reported for the measurement of briquettes or pellets porosity (bulk and individual porosity). The individual porosity (also called porosity index) of briquettes made of coal and corn cob in different ratios, with binder, was determined by Ikelle and Ivoms [19] based on the amount of water each sample is able to absorb. The porosity index was calculated as the ratio of the mass of water absorbed to the mass of the sample immersed in water. The authors did not describe very accurately the measurements they performed and question marks can arise regarding briquettes water absorption, whether all voids were filled or not with water after
immersion, as well as regarding the risk of swelling occurrence. The bulk porosity or macro-porosity is specific to cylindrical wood pellets and it is determined, for example, by using the method of stereometric measurements [20, 21].

A correlation between surface and volume (bulk) characteristics was reported by Suliman et al. [22]. They described the relationships existing between porosity and surface functionality of different wood biochars and soil water retention characteristics. One of their conclusions was that the capability of biochar to retain soil water is a function of the combination of its porosity and surface functionality, i.e. generation of oxygenated functional groups on the surface.

Since porosity is important for the analysis of briquettes combustion, it would be interesting to see if this property can be indirectly evaluated by another method, such as by measuring some roughness parameters of the briquette surface in connection with briquette density. Based on previous studies, it can be assumed that both porosity [9] and roughness parameters [23] are properties depending on wood density.

Therefore, this paper examines correlations between the following properties: porosity and density of briquettes, surface roughness parameters and density of briquettes, as well as surface roughness and porosity of briquettes. Porosity and density were determined by using three different methods.

2. Material, methods and equipment

The briquettes used for this research work were obtained from beech and spruce chips, in uncontrolled proportions, originating as waste material from secondary wood processing in the faculty workshop. The wood chips were compressed using a MB4 GOLDMARK type hydraulic briquetting press with the main characteristics indicated in Table 1.

| Press power | 4 kW |
|-------------|------|

Table 1. Characteristics of the briquetting press
### Table

| Parameter                        | Value                        |
|----------------------------------|------------------------------|
| Pressure                         | 15 MPa                       |
| Maximum capacity                 | 40 kg/h                      |
| Diameter of briquette            | 40 mm                        |
| Length of briquette              | 30-75 mm                     |
| Maximum moisture content         | 17%                          |
| Tank diameter                    | 800 mm                       |
| Press dimensions (LxHxW)         | 1200x980x1300 mm             |

The mixture of chips was compressed without binders. Cylindrical briquettes with uniform circular cross section and different lengths were obtained.

#### 2.1. Roughness parameters of briquettes

Ten briquettes were randomly taken from the press container and stored in a controlled environment (22±1 °C temperature and 40±2 % RH). Firstly, they were subjected to roughness measurements. The measurements were performed by using a MarSurf XT20 instrument manufactured by MAHR Gottingen GMBH, equipped with a scanning head MFW 250 with tracing arm in the range of ±500 μm and a stylus with 2 μm tip radius and 90° tip angle, which measured the briquettes lengthwise at a speed of 0.5 mm/s and at a low scanning force of 0.7 mN. The instrument had MARWIN XR20 software installed for processing the measured data.

The briquettes were scanned on tracing lengths of 15 mm. Four profiles were scanned for each specimen, at every 90° angle of the briquette cross-section, so that a total of 40 profiles were available for further evaluation of parameters. The lateral measuring resolution was 5 μm and the instrument provided a vertical resolution of 50 nm.

First, the software removed the form error and after that, the waviness. The roughness profiles were obtained by filtering each profile by using a robust filter RGRF (Robust Gaussian Regression Filter).
specified in ISO 16610-31 [24]. The cut-off used was 2.5 mm, as recommended in previous research by Gurau [23]. This filter was tested and found useful for wood surfaces.

After generating the roughness profiles, $Ra$, representing the arithmetic mean deviation of the assessed profile irregularities, was calculated on sampling lengths according to ISO 4287 [25]. Other calculated parameters were the material ratio curve (Abbot curve) parameters $Rpk$, $Rk$ and $Rvk$ from ISO 13565-2 [26]. $Rk$ is the depth of the roughness core profile, $Rpk$ is the average height of the protruding peaks above the roughness core profile and $Rvk$ represents the average depth of the profile valleys projecting through the roughness core profile. $Rvk$ may be especially sensitive to the species’ anatomical valleys or to various gaps caused during the briquettes pressing process. $Rpk$ is a measure of fuzziness protruding above the core roughness. The sum $Rk+Rpk+Rvk$ was also determined for comparisons, because of the cumulative effect on surface roughness and together with $Rvk$ should be sensitive to variations in briquette density (and porosity).

For each briquette and roughness parameter, a mean value and the standard deviation were calculated.

2.2. Briquettes density

In order to evaluate the briquettes density, two stereometric methods and a liquid displacement method were applied. The reason for applying different methods was to evaluate the best correlation of density with both porosity and roughness parameters. The first stereometric method (St1) was based on the measurement of the length and diameter of each briquette and on calculating the volume of a cylinder as regular geometrical shape. Two lengths (at right angle of each other) and three diameters (at each end and in the middle) of each briquette were measured using a digital pocket caliper (ULTRA, 0.01 mm accuracy). The average values and the volume were then calculated. The briquettes were weighed by using a KERN-EW 3000 g technical balance (0.01 g accuracy). The density was calculated as the ratio of mass to briquette volume.
The second stereometric method (St2) consisted in estimating the cross-section area of each briquette by means of a paper sheet of known area density (80 g/m²), as described in [27]. The briquette was placed on the paper, its contour was drawn on the paper and the cross-section surface was accurately cut. The piece of paper was then weighed and its surface was calculated from the mass and the area density of the paper. The paper surface approximating the cross-section of the briquette was multiplied by the average length of the briquette and the volume of the briquette was thus obtained. Again, the density was calculated as the ratio of mass to briquette volume.

After that, the briquettes were oven dried at 103±2°C to constant mass in order to determine the moisture content (dry basis). The moisture content was calculated based on wet and oven-dry briquette masses (SR EN 13183-1-2003/AC-2004 [28]). Finally, the oven-dry briquettes’ dimensions were measured again by using the two stereometric methods described before. Oven-dry and wet briquettes densities were calculated based on relations (1) and (2):

\[
\rho_0 = \frac{m_0}{V_0} \quad (1)
\]

\[
\rho = \frac{m_{br.}}{V_{br.}} \quad (2)
\]

where: \(\rho_0\) (kg/m³) and \(\rho\) (kg/m³) are the densities of the oven-dry and wet briquettes, \(m_0\) (kg) and \(m_{br.}\) (kg) are the oven-dry and wet briquettes’ masses, \(V_0\) (m³) and \(V_{br.}\) (m³) are the oven-dry and wet briquettes’ volumes. Wet briquettes are considered those briquettes which have the moisture content in equilibrium with the relative humidity of the surrounding air, that is, equilibrium moisture content (EMC).

The volume of oven-dry and wet briquettes by the liquid displacement method was estimated by immersing (Im) each briquette in toluene (\(C_6H_5CH_3\)) with a density equal to 865.5 kg/m³ at 20°C. The change of the toluene density with slight environmental temperature changes was neglected. The volume of the briquette was obtained from the mass of the volume of toluene displaced while immersing the briquette in the liquid. Firstly, a Berzelius glass beaker was filled with toluene to a
fixed volume (Fig. 2a). The beaker containing toluene was weighed. Then, the briquette was placed in
a metallic (copper) cage that was submerged in the Berzelius glass beaker with toluene. The part of
the liquid that exceeded the fixed initial volume was removed. The mass was determined again by
weighing. The cage was fixed by means of a wire on a glass rod placed on the glass top (Fig. 2b). The
volume of the briquette was calculated from the density of toluene and the difference in the masses
of toluene before and after briquette immersion. The density of the briquettes was evaluated by
using this method, following Fig. 2b and the equations indicated below:

\[ V_l = V_{li} + V_{br} + V_{cage} \]  

where: \( V_l (m^3) \) is a fixed volume of liquid (toluene) in the glass beaker, \( V_{li} (m^3) \) is the volume of the
liquid in the glass beaker when the briquette and cage were immersed (the part of the liquid that
exceeded the initial volume was removed), \( V_{br} (m^3) \) is the volume of the briquette, \( V_{cage} (m^3) \) is the
volume of the cage.

Fig. 2.
Briquette volume determination by using the liquid displacement method
a) before briquette immersion, b) after briquette immersion

If rearranging the terms of Eq. (3), the volume of the briquette becomes:

\[ V_{br} = V_l - V_{li} - V_{cage} \]  

Eq. (4) can also be written in terms of masses and liquid density \( (\rho_l) \), as:

\[ V_{br} = \frac{m_l - m_{li} - V_{cage}}{\rho_l} \]
and the expression of the briquette’s density is therefore:

\[
\rho = \frac{m_{\text{br.}}}{m_l - m_{l1} - V_{\text{cage}}} \tag{6}
\]

The terms \(m_l\) (kg) and \(m_{l1}\) (kg) refer to the masses of liquid corresponding to the volumes \(V_l\) and \(V_{l1}\).

The mass of the glass rod was every time subtracted from the performed mass measurements.

Briquettes are porous materials. During immersion, a part of the pores (voids developed during chips compression) was filled with liquid. In order to identify possible errors, the mass of the liquid, briquette and cage was measured first, as indicated in Fig. 2b; then, separate measurements were made of the masses of the liquid \((M_{l1})\), the briquette filled with liquid \((m_{w.br.})\) and, respectively, of the cage \((m_{cage})\).

The total mass is:

\[
M = m_{l1} + m_{w.br.} + m_{cage} \tag{7}
\]

and the sum of the individual masses is:

\[
M' = M_{l1} + m_{w.br.} + m_{cage} \tag{8}
\]

During the successive measurements of masses, some toluene may evaporate and thus, \(m_{l1}\) may be different from \(M_{l1}\). The difference

\[
M - M' = m_{l1} - M_{l1} = \Delta \tag{9}
\]

represents the error that occurs during mass measurements. The errors that were calculated for all briquettes are very small, below 1 g. The ratio \(\Delta/M_{l1}\) was also evaluated and it is lower than 0.2%, showing that, during the experiment, liquid mass losses were insignificant. The liquid that fills a part of the pores does not influence the briquette’s volume determination by the liquid displacement method. Corrections in calculating the density were made for slight equilibrium moisture content changes of the briquettes.

The volume of the cage was also determined by using the liquid displacement method and it was calculated from the following relations:
\[ V_t = V_{l_2} + V_{cage} \]  

(10)

or

\[ V_{cage} = V_t - V_{l_2} = \frac{m_t - m_{l_2}}{\rho_t} \]  

(11)

where: \( V_{l_2} (m^3) \) is the volume of the liquid existing in the glass beaker when the cage was immersed and \( m_{l_2} (kg) \) is the corresponding mass (Fig. 3).

Fig. 3. Determination of the cage volume by using the liquid displacement method

2.3. Briquettes porosity

Further on, the briquettes’ porosity was calculated by using three methods. One method is very often mentioned in literature, for example in [16, 11, 18], and the other two are recommended in two publications, Siau [10] and Hunt et al. [11], as presented below.

The first method applied in the research reported in this paper is based on relations indicated by different authors. Plötze and Niemz [16] calculated the oven-dry porosity (\( n \)) of different wood types from the oven-dry density (\( \rho \)) and the solid cell wall density (\( \rho_s \)), as:

\[ n = 1 - \frac{\rho}{\rho_s} \]  

(12)

Hunt et al. [11] determined the oven-dry wood cell porosity (\( P_d \)) from the oven-dry density (\( \rho_{OD} \)), the density of the cell wall (\( \rho_{cw} \)) and the density of air (\( \rho_{air} \)) using the following equations:

\[ \rho_{OD} = \rho_{cw}(1 - P_d) + \rho_{air} P_d \]  

(13)
They assumed that with the increase of the moisture content, the wood cell lumen size remains the same, because the moisture content (bound water) is added as an outside layer to the cell wall (Fig. 1b). Even so, they calculated a wet porosity (see third method described below), since the cross-section side of the cell wall increases with an increase in moisture content (the dimensional change due to the increase in moisture content is added to the outside of the cell wall dimension).

Similarly to Hunt et al. [11], Huang et al. [18] calculated the porosity of oven-dried bamboo wood ($\phi$) from the bulk density ($\rho_{\text{bulk}}$) (including wood substance and cavities) and skeletal density ($\rho_s$) (excluding wood cavities), as:

$$\rho_{\text{bulk}} = \rho_s (1 - \phi) + \rho_{\text{air}} \phi$$  \hspace{1cm} (15)

Despite the different notations used, the oven-dry porosity has, according to the afore-mentioned authors, a similar expression that takes or does not take into account the density of the air. Since the density of air is around 1 kg/m$^3$, it can be neglected.

A second method, calculating the wood porosity in wet conditions, is based on Siau’s equation, as indicated in [10]:

$$P = 1 - \frac{m_0}{1000 V_{br.}} (0.653 + 0.01MC)$$  \hspace{1cm} (16)

where: $P$ is the porosity or the fractional void volume of wood, 0.653×10$^{-3}$ (m$^3$/kg) is the specific volume of the wood substance, MC (%) is wood moisture content.

Eq. (16) was obtained by subtracting the cell wall (wood substance) volume fraction ($V\%_{ws}$) and moisture volume fraction ($V\%_M$) from unity:

$$P = 1 - V\%_{ws} - V\%_M$$  \hspace{1cm} (17)
Similar to wood composition consisting of dry cellular walls, bound water and lumens filled with air, one can consider a similar situation in the case of briquettes, and Eq. (17) can be considered valid for wood briquettes as well.

The third method used for wood porosity calculation, proposed by Hunt et al. [11], is based on dry cell porosity and moisture content. The oven-dry density of the cell was expressed in Eq. (13) in terms of cell wall density, density of air and oven-dry porosity, and the dry porosity was defined in Eq. (14). The oven-dry cell wall density $\rho_{cw} = 1530 \text{ kg/m}^3$ used in the calculation of the dry porosity (Eq. (14)) is based on its determination by water displacement, as described by Siau in [10]. The density of dry air at 20°C is $\rho_{air} = 1.18 \text{ kg/m}^3$ [29].

Wet porosity is obtained by the same authors from:

\[ P = \frac{(1 - V\%_{bw})P_d}{1 - V\%_{bw}P_d} \quad (18) \]

where: $V\%_{bw}$ is the bound water volume fraction.

The bound water volume fraction is calculated with respect to the wood moisture content, according to Eqs. (19) and (20), [11]:

\[ MC = \frac{V\%_{bw}\rho_{bw}}{\rho_{cw}(1 - V\%_{bw})} \quad (19) \]

or

\[ V\%_{bw} = \frac{\rho_{cw}MC}{\rho_{cw}MC + \rho_{bw}} \quad (20) \]

where: $\rho_{bw}$ is the bound water density; for bound water volume fraction calculation, the authors [10, 11] considered that $\rho_{bw} = 1115 \text{ kg/m}^3$. The equations above, developed by Hunt et al. [11] for wood, were used to calculate the effective thermal conductivity of wood briquettes with the moisture content ranging from 0% to the equilibrium moisture content [30]. Again, the wood cell is the structural component of both wood and wood briquettes, showing the validity of the method in the case of briquettes.
The first method of porosity calculation, mentioned above, referred only to the calculation of porosity characteristic to the dry conditions. However, in order to be able to compare it to the second and third methods (for wet conditions), it was considered appropriate to develop a modified equation valid for the calculation of porosity in wet conditions. As such, considering Eqs. (13) and (14), similar equations can be written for the density and wet porosity of wood cells. The density can be expressed as:

\[
\rho = \rho_{cw} (1 - P) + \rho_{air} P
\]  

(21)

where: \( \rho_{cw} \) is the density of the cell wall with bound water.

The density of the cell wall with bound water can be obtained from the rule of mixtures:

\[
\rho_{cw,w} = \rho_{cw} (1 - V_{bw}^\% ) + \rho_{bw} V_{bw}^\%
\]  

(22)

The wet porosity is obtained from Eq. (21), as follows:

\[
P = \frac{\rho_{cw,w} - \rho}{\rho_{cw,w} - \rho_{air}}
\]  

(23)

The new developed equation (Eq. 23), based on the first method, was applied in this research, in order to calculate the wet porosity of briquettes. It was named in the porosity analysis as “general relation”. Eq. (23) is explained in detail in the Appendix to this paper.

With the porosity and the briquettes’ density determined by means of the three methods described above, correlations were further examined between: porosity and briquette density, surface roughness data and briquette density, as well as surface roughness and porosity of briquettes.

3. Results and discussion

The results obtained from the density determination of the ten briquettes at equilibrium moisture content (EMC) are shown in Table 2. The equilibrium moisture content (dry basis) of the briquettes ranged from 8.13\% to 8.74\%, with an average at 8.41\%.

The highest density results were obtained by using the first stereometric method (St1) and lowest density results were obtained by using the second stereometric method (St2) (Table 2).
Table 2. Experimental, mean values and standard deviations of briquettes density (at EMC), porosity and roughness

| Density (kg/m³) | Porosity | Roughness (µm) |
|----------------|----------|----------------|
|                | Siau     | Hunt et al.    | General relation | Ra | Rvk | Rk+Rpk+Rvk |
| First stereometric method |          |                |                  |    |     |            |
| 906.0          | 0.39     | 0.39           | 0.39             | 26.7| 89.1| 194.0     |
| 888.6          | 0.40     | 0.40           | 0.40             | 25.2| 84.9| 177.1     |
| 823.2          | 0.44     | 0.44           | 0.45             | 34.5| 95.6| 249.1     |
| 894.0          | 0.39     | 0.40           | 0.40             | 23.6| 61.7| 178.6     |
| 855.7          | 0.42     | 0.43           | 0.43             | 32.0| 109.4|231.6      |
| 864.2          | 0.41     | 0.42           | 0.42             | 25.5| 92.8| 201.1     |
| 864.2          | 0.41     | 0.42           | 0.42             | 32.7| 108.9|222.3      |
| 909.2          | 0.38     | 0.40           | 0.39             | 25.0| 68.7| 192.4     |
| 832.9          | 0.44     | 0.44           | 0.44             | 28.4| 104.7|200.0      |
| 907.5          | 0.38     | 0.40           | 0.39             | 21.7| 52.0| 150.2     |
| Mean           | 874.6    | 0.41           | 0.42             | 0.41| 27.6| 86.8    | 199.6     |
| stdev          | 31.3     | 0.021          | 0.018            | 0.021|0.021|0.205    | 28.79     |
| Second stereometric method |          |                |                  |    |     |            |
| 834.0          | 0.43     | 0.45           | 0.44             | 26.7| 89.1| 194.0     |
| 825.4          | 0.44     | 0.45           | 0.45             | 25.2| 84.9| 177.1     |
| 757.5          | 0.49     | 0.48           | 0.49             | 34.5| 95.6| 249.1     |
| 830.7          | 0.44     | 0.44           | 0.44             | 23.6| 61.7| 178.6     |
| 776.8          | 0.47     | 0.47           | 0.48             | 32.0| 109.4|231.6      |
| 804.1          | 0.46     | 0.46           | 0.46             | 25.5| 92.8| 201.1     |
| 780.4          | 0.47     | 0.47           | 0.48             | 32.7| 108.9|222.3      |
| 841.7          | 0.43     | 0.44           | 0.44             | 25.7| 68.7| 192.4     |
| 808.4          | 0.45     | 0.46           | 0.46             | 28.4| 104.7|200.0      |
| 879.0          | 0.40     | 0.42           | 0.41             | 21.7| 52.0| 150.2     |
| Mean           | 813.8    | 0.45           | 0.46             | 0.45| 27.6| 86.8    | 199.6     |
| stdev          | 35.9     | 0.024          | 0.019            | 0.024|0.024|0.020    | 28.79     |
| Liquid displacement method |          |                |                  |    |     |            |
| 820.6          | 0.44     | 0.44           | 0.45             | 26.7| 89.1| 194.0     |
| 833.2          | 0.43     | 0.44           | 0.44             | 25.2| 84.9| 177.1     |
| 796.7          | 0.46     | 0.47           | 0.47             | 34.5| 95.6| 249.1     |
| 805.6          | 0.45     | 0.45           | 0.46             | 23.6| 61.7| 178.6     |
Table 2 indicates a high variability of the density (at EMC), which is influenced by the measurement method and the briquette. The largest mean density difference was encountered between the first and the second stereometric method, while the second stereometric method and the liquid displacement method (Im) showed statistically similar values, as tested by ANOVA single factor, for a confidence level $p<0.05$. However, regression analysis of density data has shown a weak correlation between individual density values calculated with St2 and liquid displacement method ($R^2=0.469$) and a better correlation with St1 method ($R^2=0.7$). Rabier et al. [27] have also obtained a high variability of the density of different types of briquettes, especially for the stereometric methods. They explained this variability through the intrinsic physical properties of the briquettes, such as the surface roughness. They noticed that stereometric methods led to more variable results, compared to immersion methods. Also, from the statistical results they concluded that the two stereometric methods cannot be regarded as equivalent, which is in agreement with our findings.

Slight differences in briquettes moisture content can also have an influence on density variability.

The relationship between individual porosity and density of briquettes at EMC is shown in Figs. 4, 5 and 6 and the calculated values, mean values and standard deviations are included in Table 2. As expected, all figures indicate the decrease of the porosity with an increase in density. This is in agreement with the results obtained for wood, as indicated by [9] and [16]. There was a strong correlation between the porosity determined according to Siau’s and the general relation methods and density, regardless the measurement method. According to the method described by Hunt et al.
for the determination of the porosity, the density measurement method has an influence on the
porosity results. While the porosity determined by Hunt et al.’s method showed high correlation with
density for St1 and St2 methods (0.976 and 0.939, respectively), it decreased to 0.5016 for the liquid
displacement method (Fig. 6). This result shows that determination of porosity with Hunt et al.’s
method is less reliable when measuring density via the liquid displacement method. The regression
analysis has revealed a weaker correlation of porosity data from Hunt et al.’s equation with porosity
calculated with the other two relations, when density was determined by immersion.

Fig. 4. Porosity of briquettes as a function of the density obtained from the first stereometric method
(St1)

Fig. 5. Porosity of briquettes as a function of the density obtained from the second stereometric
method (St2)
A preliminary analysis of the roughness parameters results showed a high variability and a weak inverse correlation with density. This may be a result of variable local density of the briquettes on their circumference. The measured profiles were taken so that two profiles corresponded to the generatrix with high briquette density and the other two on the generatrix with low briquette density, after a visual assessment. Given the high density variation, it was considered that the selection of measuring lines for each briquette can have an influence on the assessment of its overall roughness. Previous studies on sanded solid wood found an inverse relationship between density and surface roughness [23]. It was reasonable to expect that density of briquettes might have a similar relation with their surface roughness. In order to check this assumption, a mathematical procedure was applied that selects means of roughness parameters from combinations of three profiles from the measured data. As such, although 4 profiles were measured, resulting in one mean value of the four profiles (1,2,3,4), the calculations took into consideration more means taken from groups/combinations of 3 profiles, which were further checked for their best correlation with density and indirectly with porosity, respectively. For example, the combinations were means of profiles: 1+2+3; 1+2+4; 2+3+4 and 1+3+4. The more measurements are performed, the more are the means available and the better the chance of a more reliable approximation of the surface quality.
The mathematical procedure is looking to find the linear regression roughness parameters – density, with negative slope and maximum coefficient of determination. For the ten briquettes there were ten densities and four different average roughness parameters (the four means mentioned above) per briquette, that is, a matrix with ten rows and four columns. For the matrix, the following function is considered $f : \{l_1, l_2, \ldots, l_{10}\} \rightarrow \{c_1, c_2, c_3, c_4\}$, where $l_i, i = 1\ldots10$ represent the matrix rows and $c_j, j = 1\ldots4$ are the matrix columns. The total number of possible combinations is $4^{10} = 1048576$, as stated in the following theorem: the total number of functions $f : D \rightarrow E$ is $N = |E|^{|D|}$, where $D$ and $E$ are finite sets [31]. The algorithm implemented in Python programming language (version 3.7, Python Software Foundation) contains a procedure of conversion of a number from the $10^{th}$ base into the $2^{nd}$ base. The algorithm does not allow selecting two elements from the same row. Another procedure used in Python, called padding, concatenates the values obtained in the $4^{th}$ base; that is, the values $\{0,1,2,3\}$ are arranged on the ten positions so that, at the end, the array $S_i, i=1\ldots10$ can be read in order to calculate the sum. The procedure padding sets zeroes in front of the values until the array reaches a certain given dimension.

Table 3 shows the correlations between the average roughness parameters and the density of briquettes at EMC, as well as with the briquettes porosity.

Table 3. Coefficients of determination of the correlations between the porosity and density with roughness of briquettes

| Density method | Porosity method | Coefficient of determination ($R^2$): porosity-roughness parameter | Coefficient of determination ($R^2$): density-roughness parameter |
|----------------|----------------|---------------------------------------------------------------|---------------------------------------------------------------|
|                |                | $Ra$ | $Rvk$ | $Rk+Rpk+Rvk$ | $Ra$ | $Rvk$ | $Rk+Rpk+Rvk$ |
| First stereometric method | Siau | 0.576 | 0.541 | 0.582 | | | |
| | Hunt et al. | 0.619 | 0.546 | 0.604 | 0.580 | 0.537 | 0.584 |
| | Gen. rel. | 0.579 | 0.539 | 0.585 | | | |
In order to analyze the correlations of density with roughness, as well as of porosity with roughness, the Regression analysis tool was used. This involved performing a linear regression analysis by using the "least squares" method to fit a line through a set of observations. This function analyzes how, for example, briquette surface roughness is affected by the values of briquette density or porosity. High correlations indicate a strong dependence of the two properties.

The roughness parameters values decreased with an increase in density. Fig. 7 shows an example of correlation of roughness parameters and density of briquettes obtained by using the St2 method. From Table 3 and from the regression analysis it can be concluded that the correlations were reasonable in the case of the first stereometric density measurement method; however, significantly stronger correlations were obtained for the second stereometric density measurement method. This was observed especially in the correlation of the parameter $R_k + R_{pk} + R_{vk}$ and density, where the coefficient of determination was 0.911. The correlations were statistically not significant when the liquid displacement method was applied. This shows that the selection of the density measurement method has an important influence on the roughness parameters.

Table 3, too, indicates the correlations between the surface roughness and porosity obtained from the three methods of calculation and for each density measuring method. From Table 3 and the regression analysis it was observed that the surface roughness increases with briquette porosity increase.
Fig. 7. The roughness parameters of briquettes as a function of the density obtained from the second stereometric method (St2).

In case of density measured by the St1 method, the porosity determined with all three relations, has shown a similar moderate positive correlation with briquettes roughness, \( R^2 > 0.5 \), statistically significant for a confidence level \( p < 0.05 \).

However, when the density was calculated with the St2 method, the porosity determined with all three methods showed strong positive correlations with briquettes roughness (Table 3). The correlations were almost similar between the three methods and were statistically significant for a confidence level \( p < 0.05 \). Very good correlations were met by all three roughness parameters measured, but were best for \( R_k + R_{pk} + R_{vk} \). The coefficient of determination \( R^2 \) was greater than 0.9 for porosity determined by Siau and general relation methods and the roughness composed parameter \( R_k + R_{pk} + R_{vk} \) (see Fig. 8). The correlation of the porosity with \( Ra \) did not differ considerably with respect to the method of porosity calculation. The correlation of the porosity with \( R_{vk} \) was weaker in comparison with the other two parameters, but was better when Hunt et al.'s method of porosity calculation was applied.
Among the three porosity relations, only relation of Hunt et al. determined a weak positive correlation with surface roughness, for a confidence level $p<0.05$, when density was obtained by immersion, while the other porosity relations produced no correlation with surface roughness (Table 3).

If the porosity of briquettes is to be estimated by measurements of surface roughness, the best correlation can be obtained when measuring the roughness parameters $Rk+Rpk+Rvk$, followed closely by $Ra$. Very strong correlations with roughness were obtained for porosity calculated with all three relations, but when density was determined by St2 method. The findings are encouraging as they provide an alternative method to estimate the briquette porosity based on measured surface roughness.

4. Conclusions

Correlations were analyzed between porosity and density, three roughness parameters and density, and porosity and roughness parameters of briquettes. Porosity had a strong negative correlation with density when it was calculated from Siau’s equation or by using the general equation, regardless the method of density determination. The correlation was weaker if the method proposed by Hunt...
et al. was used and when the density was determined by the liquid displacement method. Strong negative correlations were obtained for the roughness parameters and density, if the density was determined according to the second stereometric method, while no correlation was found when the liquid displacement method was used. Very strong positive correlations porosity-surface roughness, were obtained for porosity calculated with all three relations, when density was determined by the second stereometric method. If the porosity of briquettes is to be estimated by measurements of surface roughness, the recommended parameter is $R_k + R_{pk} + R_{vk}$.

Although the number of the samples tested was rather low, the experimental data contribute to the existing literature on briquettes properties by adding the surface roughness, assisting in the selection of the most appropriate method for the study of porosity.

Further work is required to verify if those initial results remain consistent and repeatable for other briquettes from different batches and for other combination of wood species.

The novelty of the present paper consists in extending the applicability of the porosity models originally developed for wood, to the wood briquettes. To the best knowledge of the present authors, the wet porosity model applied to briquettes has not been reported before; it shows promising results in terms of its application to combustion analysis and heat and mass transfer processes.

**APPENDIX**

The mass of a wood cell that consists of the cell wall, bound water and air in the lumen is:

$$m = m_{cw} + m_{bw} + m_{air} \quad (A1)$$

where $m_{cw}$ is the mass of the cell wall, $m_{bw}$ is the mass of the bound water, $m_{air}$ is the mass of the air in the lumen of the wood cell. By replacing the masses by corresponding volumes and densities, the following equation can be written:

$$\rho \cdot V = \rho_{cw} V_{cw} + \rho_{bw} V_{bw} + \rho_{air} V_{air} \quad (A2)$$

If dividing each term of Eq. (A2) by $V$, Eq. (A3) becomes:
By definition, the porosity is:

\[
\frac{V_{\text{air}}}{V} = P \quad (A4)
\]

Also, the following relations can be written:

\[
V = V_{\text{cw}_{\text{at}}} + V_{\text{air}} \quad (A5)
\]

\[
V_{_{\text{cw}_{\text{at}}}} = V_{\text{cw}} + V_{\text{bw}} \quad (A6)
\]

and

\[
\frac{V_{\text{cw}_{\text{at}}}}{V} = 1 - P \quad (A7)
\]

where: \( V_{\text{cw}_{\text{at}}} \) is the volume of the cell wall with bound water.

The cell wall and bound water volume fractions are expressed as:

\[
\frac{V_{\text{cw}}}{V_{\text{cw}_{\text{at}}}} = 1 - V\%_{\text{bw}} \quad (A8)
\]

\[
\frac{V_{\text{bw}}}{V_{\text{cw}_{\text{at}}}} = V\%_{\text{bw}} \quad (A9)
\]

Considering Eqs. (A5)-(A9), Eq. (A3) becomes:

\[
P = \frac{\rho - \rho_{\text{cw}}(1 - V\%_{\text{bw}}) - \rho_{\text{bw}}V\%_{\text{bw}}}{\rho_{\text{air}} - \rho_{\text{cw}}(1 - V\%_{\text{bw}}) - \rho_{\text{bw}}V\%_{\text{bw}}} \quad (A10)
\]

But, from the rule of mixtures:

\[
\rho_{\text{cw}_{\text{at}}} = \rho_{\text{cw}}(1 - V\%_{\text{bw}}) + \rho_{\text{bw}}V\%_{\text{bw}} \quad (A11)
\]

By replacing Eq. (A11) in Eq. (A10), Eq. (A3) is therefore:

\[
P = \frac{\rho_{\text{cw}_{\text{at}}}}{\rho_{\text{cw}_{\text{at}}} - \rho_{\text{air}}} \quad (A12)
\]

Acknowledgement
We hereby acknowledge the structural funds project PRO-DD (POS-CCE, O.2.2.1., ID 123, SMIS 2637, ctr. No 11/2009) for providing the research infrastructure used in this work.

**Declarations**

**Funding:** PRO-DD (POS-CCE, O.2.2.1., ID 123, SMIS 2637, ctr. No 11/2009)

**Conflicts of interest/Competing interests:** Authors have no conflict of interest to declare

**Availability of data and material (data transparency):** All data generated or analysed during this study are included in this published article

**Code availability (software application or custom code):** not applicable

**References**

[1] Karunanithy, C., Wang, Y., Muthukumarappan, K., Pugalendhi, S.: Physicochemical characterization of briquettes made from different feedstocks. Biotechnology Research International, 1-12 (2012)

[2] Chaney, J.: Combustion characteristics of biomass briquettes. PhD thesis, University of Nottingham (2010)

[3] Mani, S.: A systems analysis of biomass densification process. PhD thesis, University of British Columbia (2005)

[4] Christoforou, E.A., Fokaides, P.A.: Thermochemical properties of pellets derived from agro-residues and the wood industry. Waste and Biomass Valorization 8, 1325-1330 (2017)

[5] Tumuluru, J.S., Wright, C.T., Kenny, K.L., Hess, J.R.: A review on biomass densification technologies for energy application. Idaho National Laboratory, pp. 3-20 (2010)

[6] Al-Hamamre, Z., Saidan, M., Hararah, M., Rawajfeh, K., Alkhasawneh, H.E., Al-Shannag, M.: Wastes and biomass materials as sustainable-renewable energy resources for Jordan. Renewable and Sustainable Energy Reviews 67, 295-314 (2017)
[7] Križan, P.: The densification process of wood waste. De Gruyter Open Ltd., Warsaw/Berlin, pp. 1-11 (2015)

[8] Grover, P.D., Mishra, S.K.: Biomass briquetting: technology and practices. Food and Agriculture Organization of the United Nations, Bangkok, Thailand (1996)

[9] Usta, I.: Comparative study of wood density by specific amount of void volume (porosity). Turkish Journal of Agriculture & Forestry 27, 1-6 (2003)

[10] Siau, J.F.: Wood: influence of moisture on physical properties. Virginia Polytechnic Institute and State University (1995)

[11] Hunt, J.F., Gu, H., Lebow, P.K.: Theoretical thermal conductivity equation for uniform density wood cells. Wood and Fiber Science 40 (2), 167-180 (2008)

[12] Ragland, K.W., Aerts, D.J., Baker, A.J.: Properties of wood for combustion analysis. Bioresource Technology 37, 161-168 (1991)

[13] Saptoadi, H.: The best biobriquette dimension and its particle size. Asian Journal on Energy & Environment 9 (3, 4), 161-175 (2008)

[14] Dunlap, F.: Density of wood substance and porosity of wood. Journal of Agricultural Research 2 (6), 423-428 (1914)

[15] Moura, M.J., Ferreira, P.J., Figueiredo, M.M.: The use of mercury intrusion porosimetry to the characterization of eucalyptus wood, pulp and paper. Iberoamerican congress on pulp and paper research (2002)

[16] Plötze, M., Niemz, P.: Porosity and pore size distribution of different wood types as determined by mercury intrusion porosimetry. European Journal of Wood and Wood Products 69 (4), 649-657 (2011)
Brewer, C.E., Chuang, V.J., Masiello, C.A., Gonnermann, H., Gao, X., Dugan, B., Driver, L.E., Panzacchi, P., Zygourakis, K., Davies, C.A.: New approaches to measuring biochar density and porosity. Biomass and Bioenergy 66, 176-185 (2014)

Huang, P., Chang, W.S., Ansell, M.P., John, C.Y.M., Shea, A.: Porosity estimation of Phyllostachys edulis (Moso bamboo) by computed tomography and backscattered electron imaging. Wood Science and Technology 51, 11-27 (2017)

Ikelle, I.I., Ivoms, O.S.P.: Determination of the heating ability of coal and corn cob briquettes. IOSR Journal of Applied Chemistry 7(2), 77-82 (2014)

Igathinathane, C., Tumuluru, J.S., Sokhansanj, S., Bi, X., Lim, C.J., Melin, S., Mohammad, E.: Simple and inexpensive method of wood pellets macro-porosity measurement. Bioresource Technology 101, 6528-6537 (2010)

Guo, W., Lim, C.J., Bi, X., Sokhansanj, S., Melin, S.: Determination of effective thermal conductivity and specific heat capacity of wood pellets. Fuel 103, 347–355 (2012)

Suliman, W., Harsh, J.B., Abu-Lail, N.I., Fortuna, A.M., Dallmeyer, I., Garcia-Pérez, M.: The role of biochar porosity and surface functionality in augmenting hydrologic properties of a sandy soil. Science of the Total Environment 574, 139-147 (2017)

Gurau, L.: The roughness of sanded wood surfaces. Doctoral thesis, Forest Products Research Centre, Buckinghamshire Chilterns University College, Brunel University (2004)

ISO/TS 16610-31, Geometrical product specification (GPS) – Filtration. Part 31: Robust profile filters. Gaussian regression filters (2010)

ISO 4287 (1997) + Amd1 (2009) Geometrical product specifications (GPS). Surface texture. Profile method. Terms. Definitions and surface texture parameters
Profile method. Surfaces having stratified functional properties. Part 2: Height characterization using the linear material ratio curve

[27] Rabier, F., Temmerman, M., Böhm, T., Hartmann, H., Jensen, P.D., Rathbauer, J., Carrasco, J., Fernández, M.: Particle density determination of pellets and briquettes. Biomass and Bioenergy 30, 954-963 (2006)

[28] SR EN 13183-1 (2003) / AC (2004) Wood. Moisture content of a piece of timber. Determination by the drying method

[29] Incropera, F.P., DeWitt, D.P., Bergman, T.L., Lavine, A.S.: Fundamentals of heat and mass transfer. John Wiley & Sons, New York, 6th ed. (2007)

[30] Sova, D., Porojan, M., Bedelean, B., Huminic, G.: Effective thermal conductivity models applied to wood briquettes. International Journal of Thermal Sciences 124, 1-12 (2018)

[31] Smullyan, R.M., Fitting, M.: Set theory and the continuum problem. Dover Publications, New York (2010)