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*Research Article*

# COMPACTION STUDIES OF TORREFIED WILLOW

**Michał Rejdak[1], Agata Czardybon[1], Karina Ignasiak[1], Aleksander Sobolewski[1], Jolanta Robak[1]**

[1] Institute for Chemical Processing of Coal, Zamkowa 1 St., 41-803 Zabrze, Poland, e-mail: mrejdak@ichpw.pl

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**ABSTRACT**
The article presents the results of studies of torrefied willow (*Salix viminalis* L.) compaction. Densification tests were performed using a hydraulic press with a maximum pressure of 216 MPa. The effect of basic parameters of the briquetting process (pressure and temperature) on mechanical parameters of manufactured briquettes were determined. On the basis of the research, it was found that the increase in pressure and temperature of the densification process increases the density and strength of pressed briquettes. The positive effect of temperature is particularly noticeable at lower pressing pressures (36 MPa – 72 MPa). In the case of a temperature of 300 $^{o}$C, the increase in a pressure from 144 MPa to 216 MPa resulted in the decrease in the density and strength of the briquette. It was also found that the briquettes manufactured at this temperature are characterized by lower density and strength than the briquettes obtained at a temperature of 200 $^{o}$C.

**Keywords:** biomass, torrefaction, briquetting, briquette

## INTRODUCTION

Biomass torrefaction is a process of low-temperature pyrolysis carried out at temperatures from 200 to 300°C, under atmospheric pressure and in inert atmosphere, which aims at the processing of the raw biomass into fuel with properties similar to coal. The result of the torrefaction process is the removal of moisture and parts of volatile organic materials from the biomass, as well as depolymerisation of long polysaccharide chains (mainly hemicelluloses), to form a hydrophobic solid product with the trade name "torrefied biomass" [Prins 2005, Kiel 2007]. Temperature from 150 to 200°C results in the dissolution of hemicellulose, which is manifested by the deformation of the structure of biomass cell walls, as well as the change in the chemical composition due to the degradation of C-H and C-C bonds, and the emission of lipophilic compounds, such as: saturated and unsaturated fatty acids, sterols and terpenes. In the temperature range from 225 to 325°C it comes to intensification of hemicellulose depolymerisation processes, recondensation of resulting products, as well as release of volatile compounds. Disintegration of lignin takes place at the temperature range from 250 to 500°C and the cellulose range from 300 to 325°C [Bridgeman et al. 2008]. As a result of the thermal decomposition, it comes to the total destruction of cell walls, and in terms of molecules, the disintegration of bonds, such as C-O and C-C, with the simultaneous formation of high molecular carboxylic acids, alcohols, aldehydes, ethers and gaseous products, including: $CO_2$, CO and $CH_4$ [Prins et al. 2006, Tumuluru et al. 2011] .

The torrefied biomass has more favorable physiochemical properties compared to the thermally-unprocessed biomass, in terms of its use in the energy sector [Li et al. 2014]. Torrefied biomass has properties more similar to low-calorie coals, rather than raw biomass [Bergman et al. 2005a, Kopczyński and Zuwała 2013a]. It is a homogeneous material characterized primarily by a greater grindability index and energy density, which in turn reduces the costs associated with grinding [Bergman et al. 2005b]. Storage and warehousing of the torrefied biomass is much safer compared to the raw biomass, due to reduced risks of biological degradation.

Calorific value of the torrefied biomass depends on the thermal conversion process conditions, as well as the properties of the material used in the process [Bergman et al. 2005a, Kopczyński and Zuwała 2013b]. Low density of the torrefaction product, porous structure and excessive fragility may cause logistical problems (high transport costs, large storage areas), as well as excessive dust emissions, resulting in an increased explosion hazard. However, these problems can be eliminated by the pressure agglomeration, the most commonly used method for compaction of powdery materials particularly biomass [Kosturkiewicz et al. 2014, Hycnar et al. 2014, Rejdak and Winkler 2015, Stelte et al. 2011, Bazagran et al. 2014, Obidziński 2014]. After the completion of process, the raw material takes the form of briquettes or pellets.

Briquetting and pelletisation are processes in which the material with suitable characteristics is subjected to high pressure and optionally high temperature. Higher temperature of lignocellulosic materials, such as wood or straw, causes the partial hydrolysis of hemicellulose, as well as cellulose recrystallization, leading to the plastic deformation of the material [Bergman et al. 2005a]. As a result of high pressure and temperature, the effect of binding intermolecular forces will be intensified, mainly between external layers of the material. Upon completion of the briquetting process, the material retains its features, including: shape, density and mechanical properties [Boyd et al. 2011]. Biomass densification is accompanied by both elastic and plastic deformation of the grains under high pressure [Tumuluru et al. 2010]. The biomass agglomeration process is mainly based on the mechanism formation of solid bridges, which are the result of chemical reactions, curing of the binder, as well as crystallization of the dissolved materials. An important role in the process of biomass densification is also played by the presence of water, due to the influence of the effect of interfacial forces (water bridges) and capillary forces [Sastry and Furstenau 1973]. Addition of water lowering the temperature of lignin softening also [Thran et al. 2016].

According to the author [Mani et al. 2002], the biomass densification process under higher pressure involves three stages. In the first stage, the particles form the tightly-packed material, in which the majority of them retain the original properties. In the next stage, it comes to the increase in contact between the particles bound together by electrostatic forces of van der Waals, which is accompanied by both plastic and elastic deformation. The third phase is the significant reduction in the volume under a high pressure, resulting in the increase in the density of the pellet. Deformed particles are no longer able to change the position, due to the reduced number of voids in the structure of the pellet.

The mechanism of biomass particles rearrangement under the applied pressure is presented in Figure 1 [Tumuluru et al. 2010, Comoglu 2007, Denny 2002].

The process of simultaneous torrefaction and densification of the material is the subject of intense research conducted for many years. Reed [Reed 1997] observed the phenomenon of synergy, the result of which is the possibility to reduce the pressure required in the integrated process by half, compared to the density of the raw biomass,

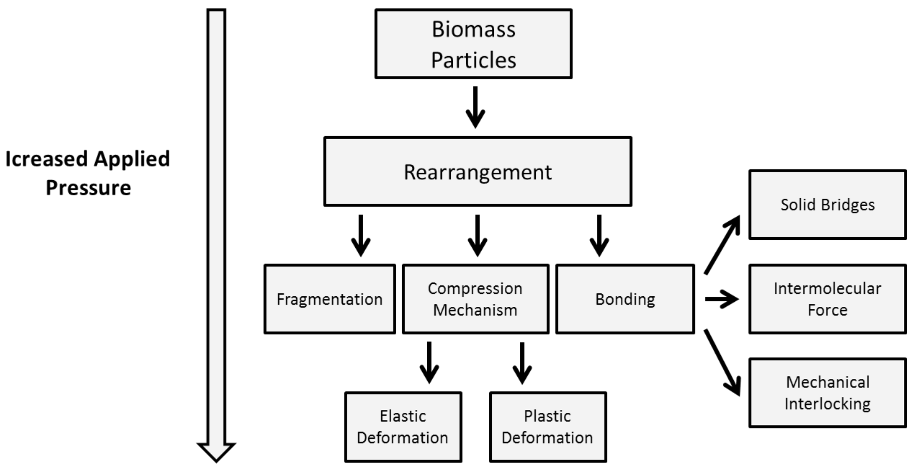


**Figure 1**. The mechanism of biomass particles rearrangement under the applied pressure [Comoglu 2007, Denny 2002, Tumuluru et al. 2010]

while maintaining the same quality of pellets. Also the power consumption required for densification turned out to be double lower at the much higher density and the calorific value of fuel.

The Energy Research Centre of the Netherlands ECN has developed the method for obtaining the pellets from torrefied biomass (TOP). The estimated cost of the entire project of the integrated system is about 20–30% higher than the process of pelletisation of the raw biomass, and the operating costs of the production of the pellet from the torrefied biomass are about 25% higher, compared to the pellets from the raw biomass [Jakubiak and Kordylewski 2010, Zuwała et al. 2014]. The higher cost of production of the torrefied pellets is, however, redeemed by higher energy values of this product. The obtained fuel has the high bulk density of 750–850 kg/m$^3$, energy density of 16–20 GJ/m$^3$, as well as calorific value of 20–25 MJ/kg [Zuwała et al. 2014]. For conventional wood pellets, these values are as follows: bulk density of 520–620 kg/m$^3$, energy density of 16–20 GJ/m$^3$ and calorific value of 8–11 GJ/m$^3$ [Obernberger and Thek 2004]. Comprehensive information in aspect of biomass torrefaction and densification has been presented in paper [Thran et al. 2016] describing the results of SECTOR project. The authors [Saeed et al. 2015] observed that torrefaction of biomass caused increase of bulk density of the obtained briquettes.

The Institute for Chemical Processing of Coal (ICHPW) under the project entitled „Biocoal For Power Generation" (BIOPOGEN), funded by the KIC InnoEnergy, conducts research on the torrefaction process in autothermal torrefaction installation [Zuwała et al. 2016] as well as densification of the resulting torrefied biomass.

This article presents the results of research of the process of briquetting of the torrefied willow (*Salix viminalis* L.). The influence of the basic parameters of the briquetting process (pressure and temperature) on the mechanical properties of the manufactured briquettes was identified.

## EXPERIMENTAL PROCEDURE

### Characteristics of the raw material

The raw material used for research was the torrefied willow (*Salix viminalis* L.) obtained during the autothermal process of torrefaction carried out in the IChPW installation at 280°C. Table 1 summarizes the results of both the proximate and elemental analysis of the raw and the torrefied willow, converted into dry and ash-free states, as well as the grain composition of the obtained torrefied biomass.

The torrefied willow was subjected to the densification process, both without pre-grinding and addition of the adhesive and water. The moisture content was approximately 3%.

## COMPACTION TESTS OF TORREFIED WILLOW

For compaction of torrefied willow the hydraulic press PW-1 with a maximum pressure of 30 T (Figure 2) was used. The weighted portion of torrefied willow was filled into the cylindrical metal mold equipped with heating elements, and then the following pressure was exerted: 36, 72, 144, 216 MPa. The tests were performed for various temperatures of the raw

**Table 1**. Properties of the raw and torrefied willow

| Parameter | Proximate Analysis | | | Ultimate Analysis | | | | |
|---|---|---|---|---|---|---|---|---|
| Fuel | $FC^d$ [%] | $V^d$ [%] | $A^d$ [%] | $C^d$ [%] | $H^d$ [%] | $N^d$ [%] | $S^d$ [%] | $O^d$ [%] |
| Willow | 19.17 | 78.53 | 2.30 | 50.10 | 6.56 | 0.78 | 0.13 | 40.12 |
| Torrefied willow | 29.61 | 67.79 | 2.60 | 56.10 | 5.29 | 0.76 | 0.04 | 37.81 |
| - | Sieve Analysis torrefied willow | | | | | | | |
| Class | >10mm | 10–8 | 8–3.15 | 3.15–2 | 2–1 | 1–0.5 | 0.5–0.2 | <0.2 |
| Content, % | 1.2 | 1.7 | 26.8 | 18.9 | 19.9 | 13.0 | 11.2 | 7.3 |

FCd [%] – the percentage content of carbon bound in the sample, converted to the dry state
Vd [%] – the percentage content of volatile matter in the sample, converted to the dry state
Ad [%] – the percentage content of ash in the sample, converted to the dry state
Cd, Hd, Nd, Sd, Od [%] – the percentage content, respectively of: carbon, hydrogen, sulphur and oxygen in the sample, converted to the dry state

material, including: 17°C (ambient temperature), 100°C, 200°C and 300°C. The initial density of the sample placed in the mold was 160 kg/m$^3$. Upon the completion of the densification process, the resulting briquettes were embossed in order to estimate their density and mechanical strength. The density of the briquettes was determined based on the quotient of the sample weight to its volume. The procedure of research was presented in Figure 3.

**Figure 2**. Hydraulic press PW-1

## TEST OF MECHANICAL STRENGTH OF THE BRIQUETTES

The tests of mechanical strength were performed using the mechanical strength testing press 5kN, based on the Brazilian Tensile Strength Method [Borowski 2002] (Figure 4 and Figure 5) also known as Indirect Tensile Strength Method [Mwanga et al. 2015]. The method consists in placing the cylindrical sample between the clamping plates of the press and applying the load (speed of 2 mm/min was used). The measurement was performed until the breakdown of the sample into two parts (Figure 6). The mechanical strength was estimated by the following equation

$$F = \frac{2F_{max}}{\pi d h} \quad (1)$$

where: $F$ – mechanical strength of the briquette, Pa
$F_{max}$ – maximum recorded force, N
$d$ – sample diameter, m
$h$ – sample thickness, m

## DISCUSSION OF RESULTS

Based on the obtained test results shown in Table 2 and Figure 7 – 9, it was found that together with the increase in a pressure applied to the sample, both the density of the briquette, as well as its mechanical strength increase. The increase in the density of briquettes depending on the applied pressure is logarithmic. By analyzing the results of the densification process in the range of 0–216 MPa, one observed the similar level of the increase in the density of the resulting briquettes, regardless of temperature. The final density stood at the level 4.7 to 5.2 times higher than the initial density of the sample. The significant difference was observed in the range of lower pressing pressures (36 – 72 MPa), for which positive influence

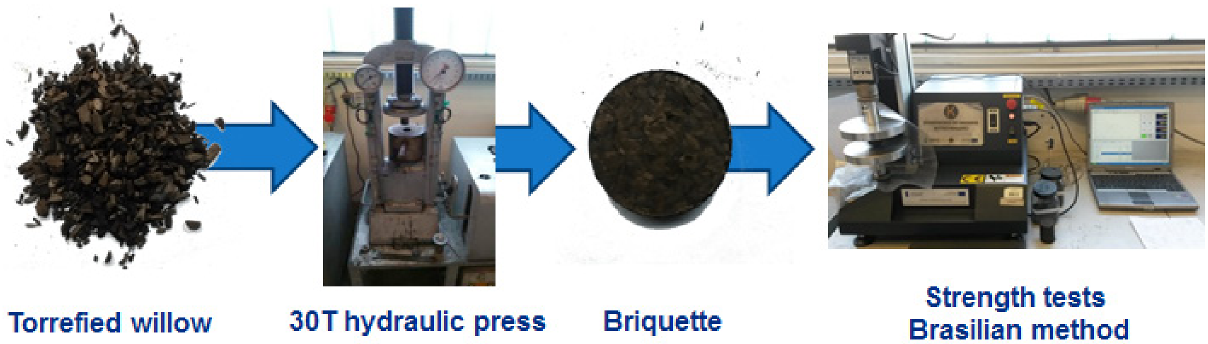


**Figure 3.** Procedure for conducting research

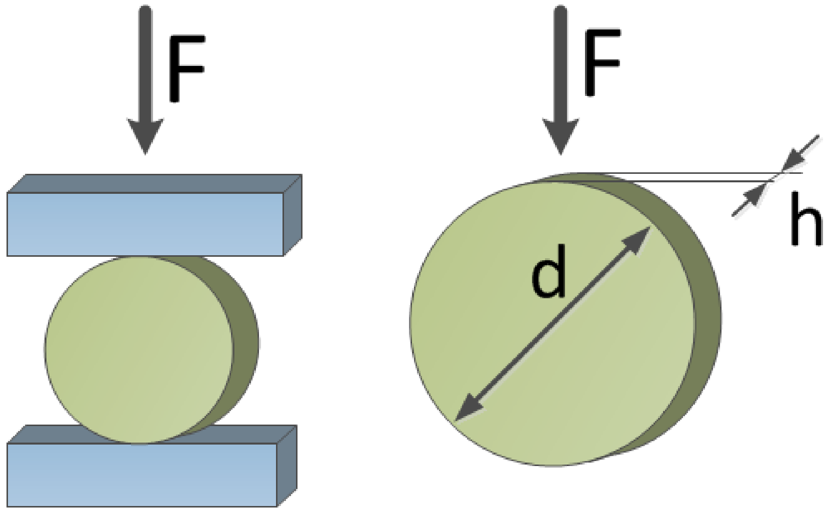


**Figure 4**. Scheme of the Brazilian Tensile Strength Method

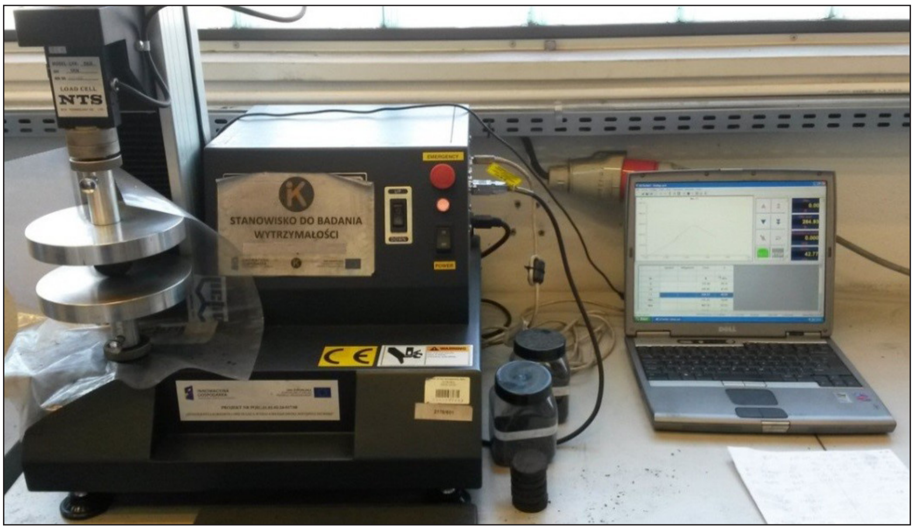


**Figure 5**. Stand for mechanical strength testing

**Figure 6**. Briquettes after completion of strength tests

**Table 2**. The aggregate list of results

| Temperature, °C | Press pressure, MPa | Briquettes density, kg/m³ | Mechanical strength, kPa | The average density, kg/m³ | Average strength, kPa |
|---|---|---|---|---|---|
| 17 | 0.0 | 160 | - | 160 | - |
| 17 | 36.0 | 541 | 28.9 | 543 | 30 |
| 17 | 36.0 | 545 | 30.2 | | |
| 17 | 72.0 | 689 | 111.3 | 699 | 131 |
| 17 | 72.0 | 708 | 151.0 | | |
| 17 | 144.0 | 902 | 451.3 | 902 | 405 |
| 17 | 144.0 | 902 | 358.8 | | |
| 17 | 216.0 | 997 | 850.3 | 972 | 833 |
| 17 | 216.0 | 947 | 815.6 | | |
| 100 | 36.0 | 606 | 200.7 | 618 | 192 |
| 100 | 36.0 | 629 | 182.6 | | |
| 100 | 72.0 | 781 | 499.7 | 773 | 520 |
| 100 | 72.0 | 766 | 539.7 | | |
| 100 | 144.0 | 936 | 991.6 | 930 | 948 |
| 100 | 144.0 | 924 | 903.7 | | |
| 100 | 216.0 | 1010 | 12221 | 997 | 1267 |
| 100 | 216.0 | 984 | 1311.4 | | |
| 200 | 36.0 | 715 | 302.6 | 722 | 299 |
| 200 | 36.0 | 729 | 295.8 | | |
| 200 | 72.0 | 824 | 557.2 | 838 | 545 |
| 200 | 72.0 | 851 | 532.4 | | |
| 200 | 144.0 | 902 | 908.1 | 919 | 949 |
| 200 | 144.0 | 936 | 989.6 | | |
| 200 | 216.0 | 936 | 1304.2 | 947 | 1239 |
| 200 | 216.0 | 959 | 1174.0 | | |
| 300 | 36.0 | 689 | 238.7 | 681 | 220 |
| 300 | 36.0 | 673 | 200.4 | | |
| 300 | 72.0 | 878 | 613.8 | 878 | 582 |
| 300 | 72.0 | 878 | 549.4 | | |
| 300 | 144.0 | 911 | 806.3 | 917 | 838 |
| 300 | 144.0 | 923 | 870.4 | | |
| 300 | 216.0 | 878 | 521.0 | 868 | 529 |
| 300 | 216.0 | 857 | 536.3 | | |

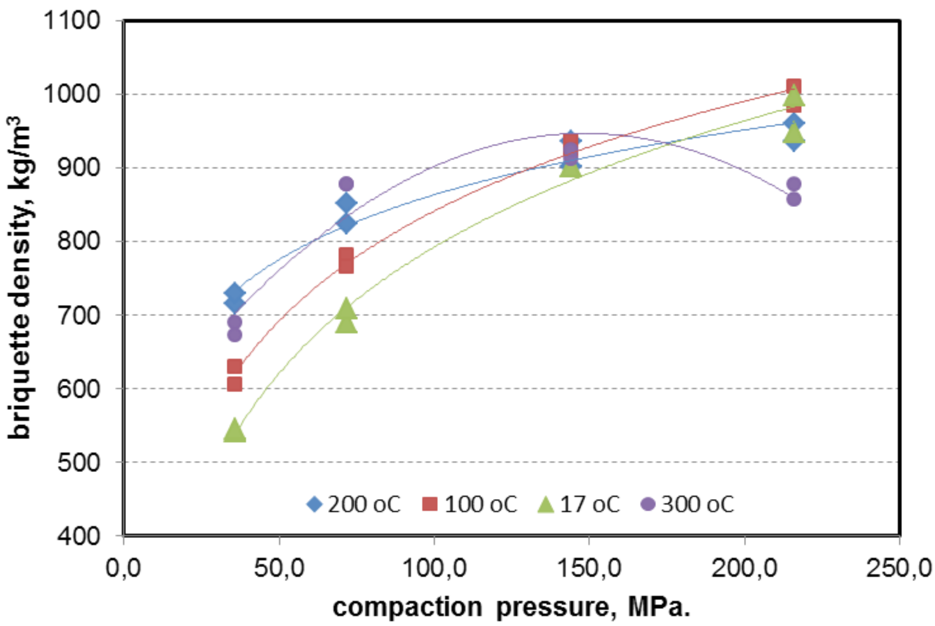


**Figure 7**. The dependence of the briquette density from the pressing pressure

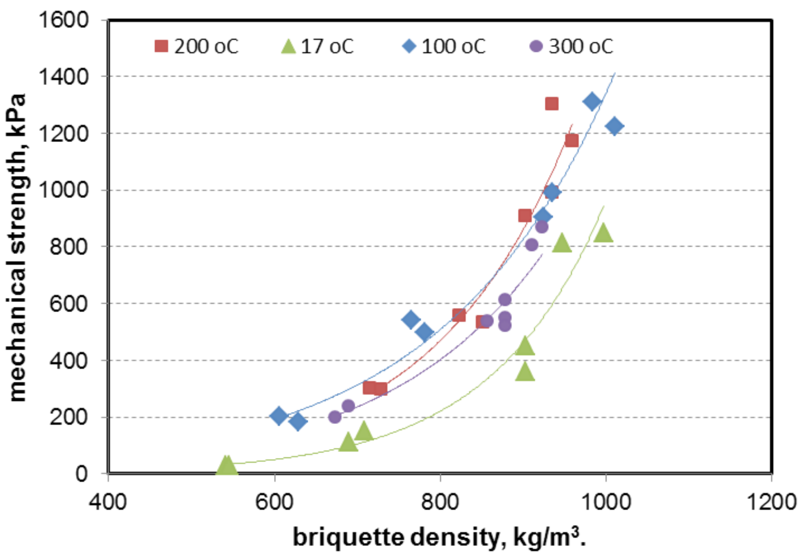


**Figure 8.** The dependence of the briquette strength from its density

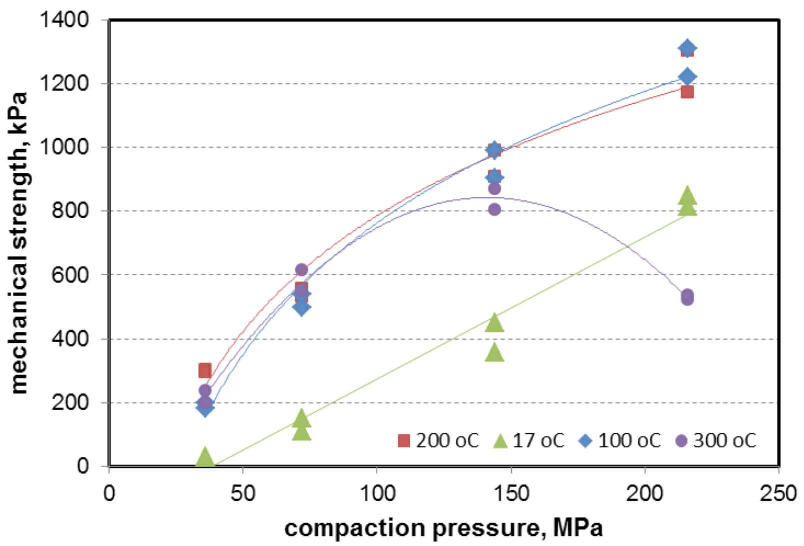


**Figure 9.** The dependence of the briquette strength from the pressing pressure

of temperature on the resulting density and mechanical strength was observed.

For a pressure of 36 MPa and temperature of 17, 100 and 200°C, the density of briquettes increased, respectively, to: 543, 618 and 722 kg/m$^3$. For 300°C slight decrease in the density of briquettes to the level of 681 kg/m$^3$ was observed. Similar trend was maintained for a pressure up to 72 MPa, for which the obtained density for temperatures of 12, 100, 200 and 300°C remained at the level of: 699, 773, 838 and 878 kg/m$^3$ respectively. The increase in the temperature of the process resulted in significant improvement in the mechanical strength of the obtained briquettes. At a pressure of 36 MPa, the increase in temperature from 17 to 100, 200 and 300°C resulted in the increase in strength from 30 to: 199, 299 and 220 kPa respectively. For a pressure of 72 MPa, the strength increased from 131 kPa for temperature of 17°C, to the value of: 520, 545 and 582 kPa, for temperatures of 100, 200 and 300°C respectively. The improvement in strength is associated with the increased density of the briquette, due to the increase in the process pressure (intensification of intermolecular interactions) and melting of lignin components of torrefied biomass (caused by growth of temperature) which act as natural binding agent and create the liquid bridges between particles. After solidification during cooling or drying process, liquid bridges turn into solid bridges [Kaliyan and Morey 2009, Toufiq et al. 2012].

For higher pressures from 144 to 216 MPa, the density of the resulting briquettes was at a similar level (902, 930, 919 and 917 kg/m$^3$). Slightly greater range of density was obtained for a pressure of 216 MPa and temperatures of 17, 100 and 200°C (respectively: 972, 997 and 947 kg/m$^3$). At a temperature of 300°C one observed the phenomenon of swelling of briquettes (Fig. 10), caused by the process of production of volatiles from the torrefied biomass, as a result of the increase in a temperature of the briquetting process above the final temperature of production of the tested torrefied willow (280°C). This phenomenon is also intensified by an increased pressing pressure. Swelling caused the decrease of their density, also leading to the significant reduction of the mechanical strength, which is particularly visible in the case of a pressure of 216 MPa (Fig. 9).

The higher density of the briquette (997 kg/m$^3$) was obtained for a temperature of 100$^o$C and a pressing pressure of 216 MPa. The resulting briquette was also characterized by the highest mechanical strength amounting to 1267 kPa. Similar strength was obtained for the briquette produced at a temperature of 200$^o$C (1239 kPa), wherein it was characterized by a lower density of 949 kg/m$^3$.

Based on the analysis of the data obtained by multiple regression method (while eliminating the data obtained for a temperature of 300$^o$C), the following equations describing the effect of the process temperature and the compaction pressure on the density and mechanical strength of the resulting briquettes was obtained:

$$r = -110.24 + 0.46 \, x \, T + 194.63 \, x \, lnP \quad (R^2 = 0.90) \tag{1}$$

$$F = -1955.7 + 2.192 \, x \, T + 513.9 \, x \, lnP \quad (R^2 = 0.89) \tag{2}$$

where: $r$ – the briquette density, kg/m$^3$
$F$ – the mechanical strength, kPa
$T$ – the process temperature (raw material), $^o$C
$P$ – the compaction pressure, MPa

The significance of the obtained empirical models was verified by the F-Snedecor test. The value of the F statistics for equation 1 and 2 is: 104, 15 and 91, 12 respectively, and is higher than the critical value $F_{kr}$ = 6.89, which indicates the statistical significance of the obtained equations. High determination coefficients also indicate a good matching of the model.

## CONCLUSIONS

The conducted research shows that the increase of pressure and temperature of the densification process increases the density and strength of the pressed briquettes. The beneficial effect of temperature is particularly visible at lower pressing pressures (36–72 MPa). In case of a temperature of 300$^o$C, the increase in pressure from 144 MPa to 216 MPa caused the decrease in the density and strength of the briquette. Briquettes produced at that temperature are characterized by lower density and strength than briquettes obtained at a temperature of 200$^o$C, which may be due to the removal of volatiles, resulting in swelling of the briquettes (Fig. 10). With the increase in pressure, the intensification of this phenomenon was observed.

Based on the conducted research, the following conclusions may be drawn:

1. The density of briquettes increases with the increase in compaction pressure, and the increase is logarithmic in nature,

**Figure 10**. The swollen briquette produced at a temperature of 300 $^o$C, at a pressure of 216 MPa

2. The increase in the density of briquettes significantly improves their mechanical strength,
3. The increase of the raw material temperature to 200°C has a beneficial effect on both density and mechanical strength of the briquette, which is particularly visible at lower compaction pressures.
4. Over the final temperature of the torrefaction process, worsening of the properties of briquettes, associated with the re-release of volatiles was observed,
5. From a practical point of view (density and strength), the most favorable effects in terms of high pressures are achieved at a temperature of 100°C, while in the case of lower pressures the increase of temperature to the value that does not cause the release of volatiles is favorable,
6. In the industrial conditions, it is possible to compact the torrefied biomass, without the necessity of binder addition.

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