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Two-stage agglomeration of fine-grained herbal nettle waste**

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A b s t r a c t. This paper compares the densification work necessary for the pressure agglomeration of fine-grained dusty nettle waste, with the densification work involved in two-stage agglomeration of the same material. In the first stage, the material was pre-densified through coating with a binder material in the form of a 5% potato starch solution, and then subjected to pressure agglomeration. A number of tests were conducted to determine the effect of the moisture content in the nettle waste (15, 18 and 21%), as well as the process temperature (50, 70, 90°C) on the values of densification work and the density of the obtained pellets. For pre-densified pellets from a mixture of nettle waste and a starch solution, the conducted tests determined the effect of pellet particle size (1, 2, and 3 mm) and the process temperature (50, 70, 90°C) on the same values. On the basis of the tests, we concluded that the introduction of a binder material and the use of two-stage agglomeration in nettle waste densification resulted in increased densification work (as compared to the densification of nettle waste alone) and increased pellet density.

K e y w o r d s: agglomeration, nettle waste, binder, starch

INTRODUCTION

One of the types of post-production waste produced in herbal companies is the nettle waste obtained during herb processing. The amount of waste left over from the process of herbal production is relatively large, with its utilisation being problematic due to the very small particle sizes of waste fractions.

In the case of Herbapol Białystok S.A., the annual production of herbal (usually nettle) waste, amounting to approx. 30 t, is usually sold cheaply or given away for a refund of the transport price.

The most common method of herbal waste management is using herbal waste as a fodder additive. Depending on the species of the plant used, herbs exhibit bactericidal (*e.g.* sage), immunostimulating (Echinacea), antioxidant (*e.g.* rosemary), sedative, and soporific (*e.g.* melissa) properties. They can also lower cholesterol levels in the body. In addition, some of these improve the aroma and flavour of fodder. In order to take advantage of this range of properties, mixtures of different plants are often used (Hanczakowska, 2007). The effect of herbal additives in pig fodder on nutritional results is presented in a paper by Paschma (2004), among other studies.

One of the methods of plant biomass waste management, including fine-grained herbal waste, is its pelleting or briquetting into a solid fuel (pellets, briquettes), and then using the resulting product as fodder or fuel (subjecting it to combustion). Various kinds of post-production plant waste have been densified into the form of pellets or briquettes: olive tree pruning residues (Carone, 2010), cork powder waste (Montero *et al*., 2014), blends of poplar and pine sawdust (Monedero *et al*., 2015), corn stover and switchgrass (Mani *et al*. 2006a, 2006b), rice straw and rice bran (Chou *et al*., 2009), herbaceous crops (Gilbert *et al*., 2009), mixtures of fine-grained tobacco and lemonbalm waste (Obidziński, 2012a), mixtures of potato pulp and buckwheat hulls (Obidziński, 2014a; Obidziński *et al*., 2016), mixtures of potato pulp and oat bran (Obidziński, 2014b), different mixtures of vine shoots and cork (Mediavilla *et al*., 2009), spent mushroom compost-coal (Ryu *et al*., 2008), and other plant mixes (Niedziółka *et al*., 2008).

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The main issue with the utilisation of fine-grained herbal waste is its low bulk density (<300 kg m^{-3}) and the extremely small particle sizes. This is hugely problematic as far as its use as a material for pressure agglomeration is concerned, due to the significant effect of densification work on the process, as compared to the densification work for larger particles of the same material. It is as well problematic because of the issues caused by the dusty fraction's coating (gluing) of the elements of the working system (densification rolls, matrix, piston) during densification. The effect of these issues is that such materials are usually subjected to non-pressure agglomeration.

According to Sobczak (2004), non-pressure agglomeration makes it possible to produce dusty particles in small volumes, which, as noted by Obraniak and Gluba (2011), is a cost-effective solution due to the relatively small investment expenditures and operational costs it involves, provided that a suitable binding liquid is selected and appropriate process parameters are set.

Gluba (2003) argues that the course of non-pressure agglomeration, and, in consequence, the properties of the obtained product, depend on the properties of both media involved in the process, in addition to the design parameters of the device and the processing conditions. This is corroborated by Wildeboer *et al*. (2005).

According to Gluba and Obraniak (2009), the commonly used pelleting methods provide ready-made products with the desired quality parameters. However, if the range of parameters is narrowed down, the means and methods for refining the existing pelleting processes need to be identified, in addition to designing new, more effective means and methods intended for a specific product.

Moreover, it is advisable to seek new methods of dusty-material pelleting, since non-pressure agglomeration fails to deliver products with adequate durability properties, and which are resistant to storage and transport conditions and hold sufficiently long life (in the case of fodder material).

Therefore, a method needs to be designed that would help to obtain a product with adequate durability properties, and, at the same time, alleviate the problems with the elements of the working system being coated (glued) during the densification process. These problems result in such materials being typically subjected to non-pressure agglomeration. The solution in this case is to use a specific type of two-stage agglomeration, *i.e.* non-pressure agglomeration, followed by pressure agglomeration.

A similar approach is presented in the literature dealing with the agglomeration of plant materials. Shipe *et al*. (2012) claim that one way to improve pellet quality is simply to supply more mechanical energy, either by using a thicker matrix, an expander, or double pelleting. All of these techniques increase the amount of electrical energy needed to produce a ton of pellets. According to Payne *et al*. (2001), double pelleting is a process whereby a conditioned feed enters the top press, where it is pre-densified through a 'thin' matrix. The pre-formed pellets then pass to the bottom press fitted with a matrix whose specification is determined by the requirements of the finished product. However, some feeds do not require double pelleting and therefore pass only through the bottom press. Payne *et al*. (2001) claim that if an excessive amount of short ends is found in the finished pelleted product when double pelleting, then it is possible that the specification for the matrix of the top press is too strict. Payne *et al*. (2001) state that it is important to use optimum matrix specifications for efficient production when double pelleting.

Robohm and Apelt (1989) hold that double pelleting improves the hardness and durability of feed pellets with a high fibre content, as well as those with high fat levels. Robohm and Apelt (1989) explain that a typical double pelleting system consists of two presses connected to each other in series. The first press, equipped with a conventional barrel-type conditioner and a relatively thin matrix (*e.g.* 5.0 mm in diameter and 25.0 mm in length) is used to pre-densify the feed. The second press is equipped with a thicker matrix (*e.g.* 5.0 mm in diameter and 40.0 mm in length). Robohm and Apelt (1989) claim that the drawback of a double pelleting system is that it requires more specific amounts of energy for pelleting.

According to Hryniewicz *et al*. (2008) and other researchers (Flore *et al*., 2009; Herting and Kleinebudde, 2007), two-stage pelleting is a novel process whereby a fine-grained material is consolidated, usually in roll presses, and then crushed and divided into grain classes. The half-product thus obtained has a larger bulk density and a lower required degree of density. As two-stage pelleting is used in the pharmaceutical industry, it is being continuously improved. Also, it is increasingly used to manufacture final products, *e.g.* certain fertilisers.

The aim of this paper is to compare the densification work during the process of pressure agglomeration of fine-grained dusty nettle waste and the properties of pellets obtained under various conditions, with the densification work and the properties of pellets obtained during a two-stage pressure agglomeration of the same material, *i.e.* material pre-densified in the process of non-pressure agglomeration with a binder addition and then subjected to pressure agglomeration.

MATERIALS AND METHODS

Post-production nettle waste (from Herbapol Lublin S.A., Branch Office in Białystok; a by-product of the manufacturing of herbal additives to medicines) was used as the test material subjected to pressure agglomeration (pelleting).

Bulk density tests showed that the tested nettle waste is characterised by a low bulk density of approximately 234 kg m^{-3}. This is of significance as far as long-haul transport of waste is concerned, as such a low density makes transport difficult. By extension, it is advisable to aim at reducing the volume of the transported raw material.

The average moisture content in nettle waste obtained from the manufacturer (Herbapol Lublin S.A. Branch Office in Białystok) was 8.6%, a value too low for the densification process to take place. Therefore, the material was moistened to assume the desired moisture content of 15, 18, and 21%. This was done by adding a specific (pre-calculated) amount of water to a known mass of nettle waste (initial moisture content: 8.6%). This involved spraying the nettle waste with a (pre-calculated) amount of water and then enclosing it in sealed plastic packages and putting it aside for 24 h to equilibrate the moisture throughout the volume.

The two-stage agglomeration of nettle waste consisted of preliminary densification (non-pressure pelleting) and secondary densification (pressure pelleting). During preliminary densification, in the process of coating, a binder in the form of a 5% potato starch solution was added. The starch used in the tests was obtained from the Peepes S.A. plant in Łomża.

The preliminary densification of nettle waste with binder content was done using non-pressure pelleting through coating; for non-pressure pelleting, a test stand was used. The design and operation of the stand is explained in a paper by Hejft and Leszczuk (2011).

In the process of non-pressure pelleting, nettle waste was fed to the charging hopper above the feeder to direct the stream of shredded ingredients to the appropriate spots on the pelleting plate. The pelleting liquid (a 5% starch solution) was fed over the pelleting plate (at two points) from a pressure tank through a set of nozzles. The stream of raw material dosed from the feeder and the stream of sprayed pelleting liquid met on the rotating plate of the pellet mill. The rotational speed of the plate was controlled by means of a single-phase motor with a converter. The contact between the shredded raw material and the pelleting liquid, coupled with the pouring motion of the material, produced pellets with a distinct round shape.

The non-pressure pelleting of nettle waste with binder content was done with the technological parameters set as follows: the inclination angle of the pelleting plate at 50°; the angle of the blade set in the pelleting plate at 90°; the rotational speed of the plate at 48 r.p.m.; the mass of nettle waste on the plate – 500 g; binder mass – 150 g; pelleting time – 16 min; the flow rate of the pelleting liquid – 0.025 $l\ min^{-1}$.

Pressure agglomeration (pelleting) tests of nettle waste and mixtures of nettle waste and a binder pre-densified in the process of coating were performed on an SS-3 stand (with the 'open densification chamber – densification piston' working system), as presented in Obidziński (2012a, 2012b).

The stand consists of a hand press mounted on the base of an open densification chamber with a 9 mm opening. The densification chamber is heated (by a heating band placed from the top down a special heat exchanger), allowing control over the process temperature. The preset temperature is reached by means of a temperature controller coupled with the heating band. The material is then densified by means of a densification piston with a strain gauge. This set-up allows the recording of the forces on the piston. Signals from the strain gauge system attached to the densification piston and the side pistons, as well as signals from the displacement sensor, are transmitted by a Spider 8-type multichannel recorder coupled with a computer, where they are recorded in the form of binary files, and then processed further.

The pressure agglomeration tests consisted of the following stages:

– preparation (moistening) of nettle waste to achieve the desired moisture content levels;
– preliminary densification of nettle waste through non-pressure agglomeration in a flat-plate pellet mill, with binder content in the form of a 5% starch solution;
– pressure agglomeration (pelleting) of nettle waste and pre-densified pellets (after 24 h) on an SS-3 test stand;
– determination of the density of the pellets obtained through pressure agglomeration (24 h after leaving the working system).

Once these stages had been completed, we determined the moisture contents of the nettle waste, the pellets obtained through non-pressure agglomeration, and the pellets obtained through pressure agglomeration.

The moisture content in the tested material (nettle waste and a mixture of nettle waste and a 5% potato starch solution) was assessed in line with PN-EN 14774-1:2010, by means of a WPE 300S scale-dryer with an accuracy of 0.01%. Each time, we determined the moisture content in five samples. The average values thus obtained were considered the final results of moisture content determination.

The density of the obtained pellets was determined 24 h after process completion. It consisted of measuring the diameter and length of 15 randomly picked pellets (with an accuracy of ± 0.02 mm) and then weighing these on a WPS 360 laboratory balance (with an accuracy of ± 0.001 g). The density of pellets (ρ_g) was calculated as the pellets mass to volume ratio.

This paper presents the results of tests of the influence of the moisture content of nettle waste (w_1=15%, w_2=18%, and w_3=21%) and the process temperature (t_1=50°C, t_2=70°C, t_3=90°C) on the values of densification work and the density of pellets produced in the pelleting processes of nettle waste and pre-densified mixtures of nettle waste and potato starch. In the course of the densification of pre-densified pellets from a mixture of nettle waste and potato starch, the impact of pellets particle size (d_{f1}=1 mm, d_{f2}=2 mm, and d_{f3}=3 mm) and process temperature (t_1=50°C, t_2=70°C,

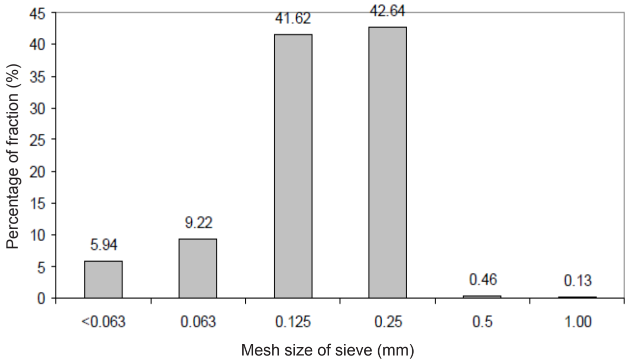

Fig. 1. Granulometric distribution of particles of shredded nettle waste subjected to a sieve analysis.

t_3=90°C) on the values of densification work and density of pellets were determined. The tests were performed at a matrix length of l = 47 mm and a mass of the densified mixture of m_p = 0.6 g.

The sieve analysis of fine-grained nettle waste was performed on an LPz-2e Multiserw Morek laboratory vortex mixer (in line with PN-89/R-64798), using a set of 5 sieves: 1, 0.5, 0.25, 0.125, and 0.063 mm. 100 g of the prepared material was weighed, poured onto the uppermost sieve and covered with a lid, and the vortex mixer was switched on for 5 min. After this time, each fraction was weighed. The obtained results represented the proportional content of a given fraction.

The LPz-2e Multiserw Morek vortex mixer was also used to divide into fractions the pellets (of a mixture of nettle and binder material) which had been pre-densified in the process of coating. Sieves with openings of 1, 2, and 3 mm were used to this end.

RESULTS AND DISCUSSION

Figure 1 shows the granulometric distribution of particles of nettle waste subjected to sieve analysis on an LPZ-2e vortex mixer.

On the basis of the sieve analysis, we can conclude that the highest mass content was obtained for the fraction with a 0.25 mm opening size, comprising 42.64% of the total mass of the tested sample. A similar fraction mass was recorded on a sieve with a 0.125 mm opening size, comprising 41.62% of the total mass. The smallest amounts were observed for fractions on the sieves with opening sizes of 0.5 and 1 mm. Both fractions combined amounted to less than 1%.

The results of the effect of the moisture content in waste material and the process temperature on the densification work of nettle waste in an open chamber are presented on Fig. 2.

The tests showed that an increase in the temperature of the densification process from 50 to 90°C coincided with a reduction in densification work. For example, an increase in the process temperature from 50 to 90°C, at a moisture content of 15%, caused a significant reduction in densification work, from 18.53 to 1.91 J (*i.e.* by approximately 89%).

The test results allow the conclusion that densification work decreases as the moisture content in waste material increases from 15 to 21% (at each of the tested process temperatures). For example, an increase in moisture content from 15 to 21% (at a process temperature of 70°C) causes a reduction in densification work from 28.09 to 23.37 J (by approximately 17%).

The effect of moisture content in waste (w_o) and the temperature of the densification process (t_p) on the densification work (L_z) of nettle waste in an open chamber is described by the following formula:

$$L_z = -198.32+10.40w_o +4.25\, t_p -0.15w_o^2-0.07w_o\, t_p -0.03t_p^2. \quad (1)$$

The test results are confirmed by the results obtained by Laskowski and Skonecki (2001), who pelleted grains of four barley cultivars. They discovered that the values of densifying pressures and specific densification work decrease with increasing temperature.

According to Filbakk *et al.* (2011), an increase in the temperature of the material improves its flexibility and makes densification easier, particularly when combined with an increase in moisture content. Moreover, higher matrix temperatures are mainly caused by friction during the pelleting process.

Figure 3 presents the results of the tests of the effect of material and process factors (process temperature and the moisture content in pellets) on the density of pellets obtained from nettle waste in the process of open-chambered pressure agglomeration.

An analysis of test results shows that as the process temperature increases from 50 to 90°C, pellet density decreases (at each of the tested moisture content values). For example, an increase in the process temperature from 50 to 90°C, at a moisture content of 15%, brings about a significant reduction in pellet density, from 1123.97 to 698.28 kg m^{-3} (*i.e.* by approximately 38%).

The tests demonstrated that pellet density increased as the moisture content in pellets increased from 15 to 21% (at each of the tested process temperatures). For example, an increase in the moisture content from 15 to 21% (at a temperature of 90°C) induces an increase in pellet density from 698.28 to 1183.23 kg m^{-3} (*i.e.* by approximately 41%).

Larsson and Rudolfsson (2012) claim that controlling matrix temperature is an efficient method of obtaining high bulk density and high pellet durability. This is corroborated by Nielsen *et al.* (2009), who claim that during the pelleting process, process temperature and moisture content were the key parameters determining the pelletisability of sawdust, and pellet hardness. Generally, increased temperature and moisture content reduced the densification work of the

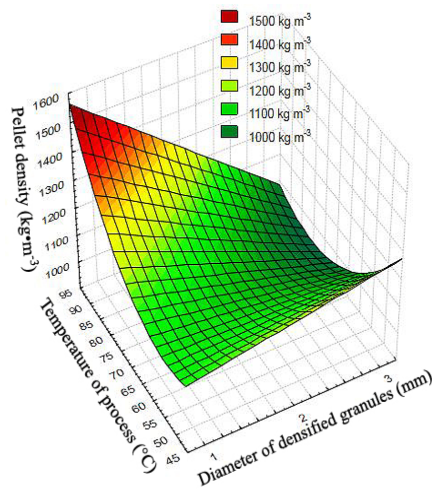

Fig. 2. Effect of material (the moisture content of pellets) and process (the temperature of the process) factors on the work of open-chambered nettle waste pressure densification.

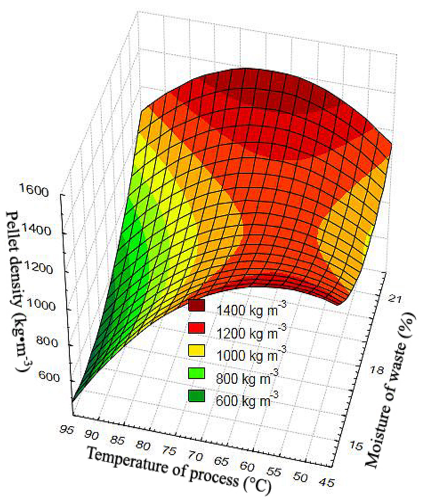

Fig. 3. Influence of material and process factors (the temperature of the process and the moisture content in pellets) on the density of pellets obtained in the process of open-chambered nettle waste pressure densification.

process. A higher temperature produced stronger pellets, while an increased moisture content may result in weaker pellets due to lowered wall friction.

According to Razuan *et al*. (2011), who investigated the physical properties of pellets made from palm kernel cake, the most favourable temperature for pellet production was 80-100°C. These pellets had densities of 1 184-1 226 kg m^{-3} and tensile strengths of 930-1007 kPa. Carone *et al*. (2010) investigated the effect of key process parameters (pressure and temperature) and biomass characteristics (moisture content and particle size) on selected mechanical properties (density and durability) of pellets produced from olive tree pruning residues. They pelleted biomass with three different hammer mill screen sizes (1, 2, and 4 mm), at different moisture contents (5, 10, 15, and 20% w.b.), process temperatures (60, 90, 120, and 150°C), and pressures (71, 106, 141, and 176 MPa) in a lab-scale pellet press. They discovered that temperature was the most important variable influencing the mechanical properties of pellets, followed by initial moisture content and raw material particle size. In particular, a high process temperature, a low moisture content, and a reduced particle size provided high quality pellets.

Li *et al*. (2015), who investigated energy input and physical properties of pellets made from sewage sludge and biomass, concluded that temperature and moisture content both had an impact, to various extents, on the properties of the discussed pellets, which were found to have been independent of pressure. For co-pelleting and an optimum pellet quality, they recommended a pressure of 55 MPa, a temperature of 90°C, and a moisture content of 10-15% as the optimum parameters.

The effect of moisture content in pellets (w_o) and process temperature (t_p) on the density of pellets (ρ_g) obtained during open-chambered nettle waste densification is described by the following formula:

$$\rho_g=5511.41-587.38w_o+26.67t_p+13.34w_o^2+1.91w_o\,t_p-0.49t_p^2. \qquad (2)$$

Tests of the pressure densification of nettle waste (moistened up to moisture contents of, respectively, 15, 18, and 21%) in an open chamber (on an SS-3 stand) show that nettle waste is too fine-grained a material and thus not susceptible to densification. An agglomerate of a sufficient quality could not be obtained at any of the different values of moisture content and temperature. Indeed, at the highest temperature of 90°C, the pellets crumbled immediately after leaving the matrix. For this reason, we decided to use binder material, in the form of a 5% starch solution, in further tests, and to employ two-stage pelleting, *i.e.* pelleting which involves non-pressure agglomeration with the use of a flat-plate pellet mill in the first stage, followed by, *i.e.* pressure agglomeration, in the second stage.

Figure 4 shows the results of tests of the effect of material (fraction particle size) and process (process temperature) factors on the open-chambered densification work of pellets (as obtained in a non-pressure manner through coating) from a mixture of nettle waste and a 5% potato starch solution.

On the basis of the tests, we found that the densification work (at each of the tested fraction particle sizes) increased as the temperature of the densification process increased from 50 to 70°C. For example, an increase in the densification temperature from 50 to 70°C at a fraction particle size of 1 mm brought about an increase in the densification work from 36.43 to 78.56 J (*i.e.* by approximately 54%). A further increase in the process temperature from 70 to 90°C engendered a reduction in the densification work. For example, an increase in the process temperature from 70 to

90°C at a fraction particle size of 1 mm caused a reduction in the densification work from 68 to 25 J (*i.e.* by approximately 63%).

Furthermore, increasing the particle size of densified pellets from 1 to 3 mm induced a reduction in the densification work at each of the tested process temperatures. The highest reduction in densification work, from 78.56 to 20.17 J (*i.e.* by approximately 74.32%), was obtained when the pellet particle diameter increased from 1 to 3 mm (at a temperature of 70°C).

The results of the tests concerning the effect of temperature and particle sizes of material on the process of the densification of nettle waste with potato starch content are comparable with the results obtained by other researchers.

Montero *et al*. (2014) who pelleted powder from cork industries, claimed that mixing different particle sizes was not an obstacle during the pelleting process, and no pretreatment (drying, sifting, *etc*.) was required in any of the cases, which in turn resulted in reduced processing costs. They claim that raw materials and different mixtures of cork can be pelleted regardless of the real demand for granulated cork (0.5-1, 1-2 mm, *etc*.).

Shaw *et al*. (2009) investigated the effect of a reduced particle size (an average of 0.3-0.6 mm after pelleting), two temperature values (70-100°C), and two moisture content levels (9-15%) on the density and mechanical resistance of pellets (produced in a laboratory press) from wheat straw and poplar wood. They discovered that under all these conditions, smaller particle sizes resulted in higher pellet densities and mechanical resistances. Gilbert *et al*. (2009) studied the effect of pressure and temperature on the density and mechanical resistance of pellets from wheat straw and grass. Their study showed that at temperatures between 75 and 100°C, lignin melts act as a natural binder and a maximum resistance is obtained. However, above 100°C, water evaporates and pellets become more brittle.

Nguyen *et al*. (2015) investigated the effect of process parameters and raw material characteristics on the physical and mechanical properties of wood pellets made from particles of sugar maple trees (of different vigour) in a single pellet mill, while controlling temperature (75, 100, and 125°C), moisture content (8.1, 11.2, and 17.2%), densifying force (1 500, 2 000, and 2 500 N), and particle size (<0.25, 0.25-0.5, and 0.5-1.0 mm). Particle size was the most important factor for the friction in the matrix, followed by moisture content, densifying force and temperature. They claim that in order to minimise friction, and, consequently, maximise the density and compressive strength of the produced pellets, the pelleting process should take place at a temperature of approximately 100°C and at a moisture content of approximately 11.2%.

According to Kashaninejad *et al*. (2014), the total specific energy required for the densification and ejection of pellets (a matrix diameter of 6.35 mm) made from wheat straw grinds, varied from 4.35 to 33.64 MJ t^{-1} and increased with compressive load and particle size (less than 1 mm).

The effect of fraction particle diameter (d_f) and densification process temperature (t_p) on the densification work temperature (L_z) of pellets (obtained in an open-chambered non-pressure manner from nettle waste with a starch solution) is described by the following formula:

$$L_z = -170.66 - 8.01d_f + 7.08t_p - 5.02d_f^2 + 0.21d_f t_p - 0.06t_p^2, \quad (3)$$

d_f – fraction particle diameter (mm).

Figure 5 illustrates the results of tests of the effect of material and process factors (process temperature and densified fraction particle diameter) on the density of pellets obtained in the process of open-chambered pressure agglomeration, from a raw material in the form of nettle waste pellets produced in a non-pressure manner through coating with a 5% potato starch solution.

An analysis of Fig. 5 leads one to the conclusion that the density of pellets increased (at each of the tested fraction particle sizes) as the densification process temperature increased from 50 to 90°C. For example, an increase in the temperature of the densification process from 50 to 90°C, at a fraction particle size of 1 mm, caused an increase in pellet density from 1 110.44 to 1 364.46 kg m^{-3} (*i.e.* by approximately 19%). Only at a particle size of 3 mm did an increase in temperature (from 50 to 70°C) result in a reduced pellet density. The obtained reduction was attributable to the increased moisture content in pellets (obtained in a non-pressure manner) (Fig. 6).

The excessive moisture content (approximately 23%) of the densified 3 mm fraction caused depressurisation – as a result of the high pressures and high temperatures in the chamber – of the pellets which had left the densification chamber.

Excessive moisture content (approximately 23%) of the densified 3 mm fraction caused depressurisation of the pellets that left the densification chamber, due to the high pressures and high temperatures in the chamber.

The tests (Fig. 5) showed that as the particle size of densified pellets increased from 1 to 3 mm, a reduction in pellet density (at process temperatures of 70 and 90°C) occurred. The obtained reduction was also attributable to the excessive moisture content in pellets obtained in a non-pressure manner (Fig. 6), with moisture levels being approximately: 23 and 21% for the 3 and 2 mm fraction, respectively. Such high moisture levels brought about the depressurisation of the pellets which had left the densification chamber, thus reducing their density. Only at a process temperature of 50°C does an increase in the particle size of pellets from 1 to 3 mm create an increase in pellet density, from 1 110.44 to 1 184.16 kg m^{-3} (*i.e.* by approximately 6%). The highest pellet density (1 364.46 kg m^{-3}) was obtained

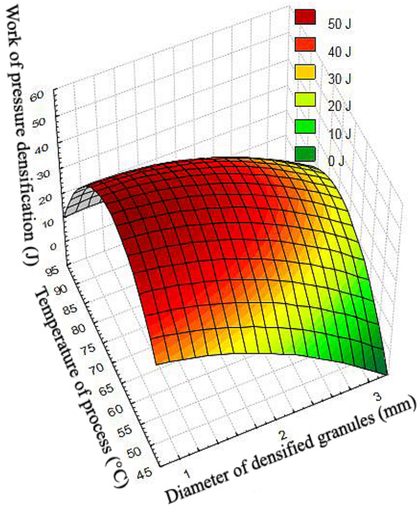

Fig. 4. Effect of material (the size of fraction particles) and process (the temperature of the process) factors on the work of open-chambered pressure densification of pellets (obtained in a non-pressure manner through enveloping) from a mixture of nettle waste and a 5% potato starch solution.

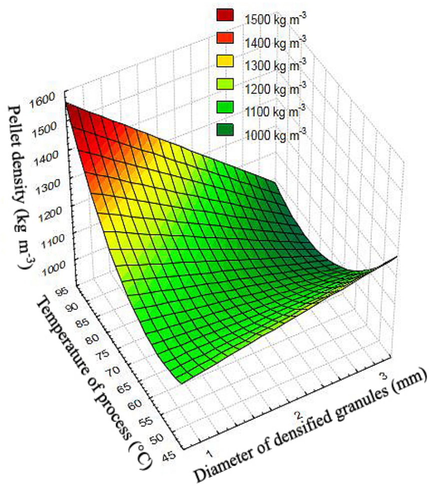

Fig. 5. Effect of material and process factors (the diameter of particles of the densified fraction and process temperature) on the density of pellets obtained in the process of open-chambered pressure agglomeration, of pellets (obtained in a non-pressure way) from nettle waste and a 5% potato starch solution.

at a temperature of 90°C and a fraction diameter size of 1 mm. The tests showed that increased pellet density was strongly correlated with a reduction in the moisture content in the densified pellets (as obtained in a non-pressure manner from a mixture of nettle waste and a starch solution).

The effect of fraction particle diameter (d_f) and process temperature (t_p) on the density of pellets (ρ_g) obtained through densification in an open chamber (produced in a non-pressure manner from a mixture of nettle waste and a starch solution) is described by the following formula:

$$\rho_g = 1683.69 + 314.17 d_f - 25.03 t_p + 0.73 d_f^{\,2} - 5.87 d_f\, t_p + 0.27\, t_p^{\,2} \quad (4)$$

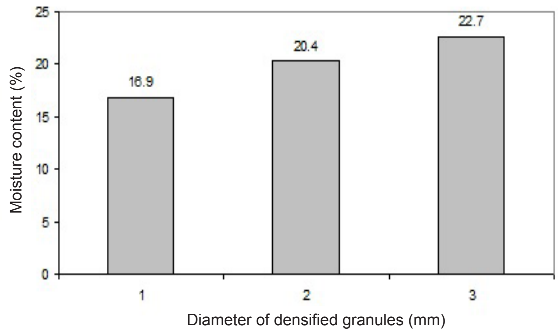

Fig. 6. Correlation between moisture content and the size of particles of pellets from a mixture of nettle waste and a 5% potato starch solution obtained in a non-pressure way.

The tests showed that at a temperature of 70°C, across the range of fraction particle diameters, pellets of a sufficient density failed to be produced. They crumbled at a slight press of the hand. Conversely, at a temperature of 90°C, across the range of pellet sizes, pellets of a sufficient density (from 968.78 to 1 364.46 kg m^{-3}) were successfully produced.

The tests allowed us to conclude that the two-stage pelleting of fine-grained nettle waste, in which the waste was pre-densified in the process of coating with a binder addition and then subjected to pressure agglomeration, resulted in increased work required for waste densification, from 0.87-34.28 J (in the case of pressure densification of nettle waste alone) to 6.58-78.56 J (in the case of the pressure densification of pellets obtained in a non-pressure manner from nettle waste and a 5% potato starch solution). Adding a binder material (in the form of starch) to nettle waste in the process of non-pressure agglomeration prior to pressure pelleting resulted in increased densifying pressures and, in consequence, increased densification work (compared to the densification of nettle waste alone). In their studies, Stasiak *et al*. (2013) corroborated the very good properties of starch as a natural binder.

The obtained results of tests of the pressure densification of nettle waste with potato starch content are comparable with the results obtained by other studies dealing with biomass densification with binder materials. According to Kaliyan and Morey (2010), natural binders in biomass can be expressed or activated (softened) under high pressures in the presence of moisture (*e.g.* water soluble carbohydrates) or, in some cases, an increased temperature. Pre-densified nettle waste, in the course of pressure densification, on contact with surfaces of matrix openings, resulted in increased resistance to forcing (the mixture no longer slid on opening surfaces, as was the case when nettle waste was densified alone), yielding high-density pellets.

The use of the two-stage pelleting of fine-grained nettle waste, in which the waste is pre-densified in the process of coating with a binder addition and then subjected to pressure agglomeration, substantially increases the density of pellets. During the pressure densification of nettle waste alone, densities ranging from 491.77 to 1 212.61 kg m^{-3} were obtained, while in the course of the pressure densification of pellets (obtained in a non-pressure manner) from nettle waste and a 5% potato starch solution, densities ranged from 968.78 to 1 364.46 kg m^{-3}.

The positive effect of binder addition to biomass on the quality of pellets in the pelleting process has been corroborated by many studies. Finney *et al*. (2009) concluded that the kinetic durability of pellets increased considerably when small amounts (over 1%) of binder (sodium hydroxide and maize starch) were added during biomass pelleting. Razuan *et al*. (2011) discovered that adding small amounts of caustic soda (1.5-2.0 wt.%) to palm kernel cake increased the tensile strength of the obtained pellets. Gilbert *et al*. (2009), who produced pellets from a mixture of switchgrass and heavy oil, concluded that heavy pyrolysis oil is of potential use as a binder material that can substantially increase pellet strength and durability. Ohman *et al*. (2006) observed that it was possible to improve the quality of pellets produced from biomass by adding hydrolytic post-production waste obtained during the production of ethanol from lignocellulosic materials. Sotannde *et al*. (2010) concluded that using binding materials (gum arabic and Cassava starch) in the process of the densification (briquetting) of carbonised (torrefied) forest wood waste allows the production of briquettes of a high kinetic durability. According to Filbakk *et al*. (2011), higher contents of lignin and extractives have a positive effect on binding mechanisms during the production of pellets from pinewood containing 0, 5, 10, 30, and 100% of bark.

CONCLUSIONS

The tests involving the non-pressure and pressure pelleting of a mixture of nettle waste and a binder in the form of a starch solution led to the following conclusions:

1. The addition of a binder to nettle waste yielded high density pellets, pressure-produced from pellets obtained earlier in a non-pressure manner. The densities of nettle waste with a starch solution pellets ranged from 972.15 to 1364.46 kg m^{-3}.

2. An increase in the fraction particle diameter of pellets obtained through non-pressure densification from nettle waste with a starch solution from 1 to 3 mm resulted in a reduced densification work and an increased density of pellets obtained in the process of pressure agglomeration.

3. An increase in the temperature of the densification process of nettle waste and starch solution pellets obtained in a non-pressure manner, from 50 to 90°C, brought about an increase in densification work and pellet density.

4. The most beneficial value of pellet density (1 364.46 kg m^{-3}) obtained in the process of pressure densification (in a non-pressure manner) from a mixture of nettle waste and a starch solution was achieved at a process temperature of 90°C and a fraction particle diameter of 1 mm.

5. The introduction of a binder material and two-stage agglomeration to the process of nettle waste densification resulted in an increased pellet density and increased densification work, when compared to the densification of nettle waste alone (at the same time, the greatest amount of energy was used for waste densification).

Conflict of interest: The Authors do not declare conflict of interest.

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