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Effects of Initial Moisture Content on the Physical and Mechanical Properties of Norway Spruce Briquettes

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***Abstract*—**The moisture content of densified biomass is a limiting parameter influencing the quality of this solid biofuel. It influences its calorific value, density, mechanical strength and dimensional stability as well as affecting its production process. This paper deals with experimental research into the effect of moisture content of the densified material on the final quality of biofuel in the form of logs (briquettes or pellets). Experiments based on the single-axis densification of the spruce sawdust were carried out with a hydraulic piston press (piston and die), where the densified logs were produced at room temperature. The effect of moisture content on the qualitative properties of the logs, including density, change of moisture, expansion and physical changes, and compressive and impact resistance were studied. The results show the moisture ranges required for producing good-quality logs. The experiments were evaluated and the moisture content of the tested material was optimized to achieve the optimum value for the best quality of the solid biofuel. The dense logs also have high-energy content per unit volume. The research results could be used to develop and optimize industrial technologies and machinery for biomass densification to achieve high quality solid biofuel.

***Keywords*—**Biomass, briquettes, densification, fuel quality, moisture content, density.

I. Introduction

PRODUCTION of high quality solid biofuel brings many advantages compared to non-densified fuels. The uneven, fluffy, and dusty characteristics of biomass often make the material difficult to store, transport, and utilize. Densified biomass fuel can ease these problems. The higher bulk and energy densities result, respectively, in lower costs of transportation, handling and storage, and higher energy conversion efficiency, while the reduced moisture content increases the long-term storage capability [1], [2].

The strength and durability of the densified products depend on the physical forces that bond the particles together. The binding forces that act between the individual particles in densified products have been categorized into five major groups [3]-[5]. They are (i) solid bridges, (ii) attraction forces between solid particles, (iii) mechanical interlocking bonds, (iv) adhesion and cohesion forces, and (v) interfacial forces and capillary pressure.

The final moisture content of biomass pellets or briquettes is highly dependent on processing conditions such as initial moisture content, temperature, and pressure. Water acts as both a binding agent and a lubricant. Moisture present in the biomass facilitates starch gelatinization, protein denaturation, and fiber solubilization processes during extrusion, pelleting, or briquetting [6], [7]. Water helps develop Van der Waals' forces by increasing the area of contact between particles, according to [8]. According to [9], initial moisture content higher than 15 % and pressure higher than15 MPa has a negative effect on the final briquette quality, where cracks were observed. Pellets with a moisture content of less than 5 % can result in revenue loss for the pellet manufacturer, as they tend to break up, creating more fines during storage and transportation. Pellets with high moisture content can be subject to spoilage due to bacterial and fungal decomposition resulting in significant dry matter losses during storage and transportation [10], [7]. Steam-treated biomass is superior, as the additional heat modifies the physiochemical properties (gelatinization of starch, denaturation of protein) to such an extent that binding between the particles is significantly enhanced, resulting in improved densification quality [11]. In the study [12], it was found that increasing the moisture content of spruce wood sawdust from 7 to 15 % significantly increased the strength of the pellets. The authors of the study [1] researched the compaction of tree bark, sawmill waste, wood shavings, alfalfa hay, fresh alfalfa, and grass in a punch, die assembly, and found that an optimum moisture content of approximately 8% was required to produce high-density briquettes. They also recommended that a moisture content of 5–12% is necessary to produce good quality (in terms of good density and long-term storage properties) logs from hardwood, softwood, and bark in the forms of sawdust, mulches, and chips. They also noted that pellets or briquettes tend to become fragile in just a few days if the moisture content is less than 4 % due to absorption of moisture from the environment [1]. Densification of alfalfa having lower moisture content and fewer long fibers (more fines) had resulted in more stable wafers due to limited expansion. Alfalfa pellets made with 19% moisture content had the highest durability [13]. The optimum moisture content for pelleting cellulosic materials is 8–12% [14]. The study of briquetting sawdust, sander dust, wood shavings, and peanut hulls concluded that 15 % moisture content is favourable [15]. Results of [16] show initial moisture content of the raw material is the most important variable for controlling moisture uptake in production of Norway spruce pellets. In the study [17] of Scots pine pelleting, the moisture content is the dominant factor for bulk density and current for the pelletizer, which is explained by the change in friction in the die due to the

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lubricating properties of water. The study also reported that an optimum pellet quality was obtained from storage times in excess of 120 days and within a range of moisture content of 11–13%. Many researchers have found that the optimum moisture content for densification of biomass is different for each individual raw material and operating conditions.

Commercially, the most widely-used pressure agglomeration machines for densification of biomass materials are ram (piston-type) extrusion presses, screw extruders, and pelletizers (ring-die, flat-die) [18]. In piston presses and screw extruders, the raw material is conveyed and compressed by a piston or a screw through a die to form a briquette of cylindrical or other shape. In a pelletizer, the material is pressed through open-ended cylindrical holes (dies) made in a flat plate or in the surface periphery of the ring. Between one and three small rotating rollers, push the feed material into the die holes. The surface friction between the feed particles and the wall of the die resist the free flow of the feed, and thus the particles are compressed against each other inside the die to form pellets [18], [6].

A high-pressure piston and die compaction process was used to densify the biomass materials in this study. The hydraulic press used is a conventional production briquetting press. The densification process of this press can achieve high and uniform pressure so that dense and strong briquettes can be produced under normal temperature without having to use a binder or heat. Materials can be compacted rapidly at room temperature to form good-quality products.

The selection of the type of physical quality testing method (compressive resistance, abrasive resistance, impact resistance or water resistance, etc.) used to test a particular densified product depends on the type of conditions encountered between manufacturing and use. An appropriate test is one that simulates the way the densified products are handled, transported and stored, and results in approximately the amount of fragmentation and fines that are produced in these manipulating processes.

II. Material and Methods

A. Raw Material

Fresh sawdust from Norway spruce (*Picea abies*) originating from a region of West Slovakia was obtained from sawmill where it had been dried to a moisture content of 8.2% and processed in a hammer mill. The sawdust contained no bark.

Approximately 1.5 tons of the prepared raw materials were transported to the Biomass Laboratory of the Faculty of Mechanical Engineering of the Slovak University of Technology in Bratislava, Slovakia. The material was stored in the laboratory in large bags for six months before experiments began. The particle size distribution was analysed for a representative seven samples of 100 g by the Retsch Vibratory Sieve Shaker AS 200 Digit. The raw material particle size distribution was measured as in Table I.

Nine samples of the raw material with different initial moisture contents were prepared six days before densification. Individual material samples were weighed, and the exact amount of water at a temperature of 15°C was added by spraying. Each sample was mixed to homogenize the moisture, and then stored in plastics bags. The values of the initial moisture content of the samples measured immediately before the densification process were 7.4%; 9.1%; 10.3%; 11.7%; 12.6%; 14.5%; 16.5%; 19.6% and 22.0% w.b. The range of moisture content was designed to include the whole range where the densification of biomass is possible, and distribution of chosen values of moisture content is uniform enough to obtain precise results.

TABLE I
RAW MATERIAL PARTICLE SIZE DISTRIBUTION OF NORWAY SPRUCE SAWDUST

Particle size (mm)	Proportion by weight (%)
≥ 4.00	2.56
2.00 - < 4.00	12.69
1.00 - < 2.00	35.92
0.50 - < 1.00	26.06
< 0.50	22.77

B. Densification Process

Briquettes were produced in an industrial scale process with a hydraulic briquetting press, the BrikStar 200 (BRIKLIS, s.r.o, Czech Republic) equipped with a cylindrical open die with a diameter of 50 mm and length of 340 mm. The densification process of biomass is created by a moving piston from one side of the die, and the frictional resistance of the briquette as it is pressed into the die. Increased, friction depending on the length of the die causes higher compacting pressure on the raw material.

After the raw material was fed by the screw and pre-compacted by the side piston, pressure was applied at a constant rate by the main piston. When the pressure reached the desired value, the main piston moved back immediately, without any time lag.

The hydraulic press makes it possible to programme the length of briquettes and to use permit continued constriction of the die. The length of briquettes depends on the raw material and feeding parameters. A constant briquette length (30% of its maximum), constant feeding parameters and an ultimate densification pressure of 16.5 MPa were used in this study. In addition, constriction of the die was not used during the densification process. The temperature of the briquettes was measured up to 30°C during processing.

C. Sampling

Once the briquetting process had reached a stable state and briquettes with the same initial moisture content had achieved a similar appearance, sampling of 40 briquettes was conducted. Thirty of them were selected for measurement of their physical characteristics, and ten of them for measurement of their moisture content. The sampling was repeated nine times separately for each prepared raw material with different initial moisture content. All samples were marked and stored at 20°C in the laboratory. In total, 9x40 briquettes were collected.

D. Moisture Content

Moisture content of the briquettes was measured at three stages: (i) 5 minutes after ejection, (ii) 5 days after ejection, and (iii) 90 days after ejection. The determination of the moisture content was based on heating the material (disintegrated briquette) to 105 ± 2°C until a constant weight was achieved according to the standard procedure described in EN 14774-3:2009 [19].

E. Weight, Dimensions and Density

The weight, length and diameter of each briquette were measured using a digital caliper and an electronic balance. Briquette density was calculated by means of the ratio between briquette weight and its volume including pore volume. The volume of briquettes was calculated as the volume of a cylinder with dimensions (length and diameter) measured as previously described, according to EN 16127:2012 [20]. The average density was calculated for each sample of 30 briquettes with different initial moisture content of raw material.

The densities of the briquettes were measured 5 min. after they were ejected from the die. Other properties of the briquettes were tested 5 days and 90 days after the briquettes were made and stored in air in the laboratory. The 5-min density measurement test was chosen because most of the briquettes underwent expansion after ejection, with the most rapid expansion occurring within the first 5 minutes.

F. Compressive Resistance

Compressive resistance represents the maximum crushing load a briquette can withstand before cracking or breaking. The compressive resistance test simulates the compressive stress due to the weight of the top briquettes on the lower briquettes during storage, transport and handling. Compressive resistance of the densified products is determined by a diametrical compression test. A single briquette is placed between two flat parallel platens, which have faces with a greater surface area than that of the area of the briquette. An increasing load is applied at a constant rate, until the test briquette fails by cracking or breaking. The load at the point of fracture is read off a recorded stress–strain curve, which is the compressive strength, and reported as force or stress [5].

A hydraulic press was used to determine the compressive resistance in this study. Ten briquettes from each sample with different initial moisture content were tested five days after ejection. The compressive resistance was calculated for each briquette as a ratio between the maximum force at the point of fracture and the length of the briquette. After that, the two extreme values of compressive resistance (maximum and minimum values) were excluded from each sample group of tested briquettes. The resulting value of compressive resistance for each tested sample group of briquettes was calculated as an arithmetical average of eight particular values.

The compressive resistance test provides a quick measurement of the quality of briquettes as they are produced from the briquetting press, and assists in adjusting the briquetting process in order to improve the briquette quality. Li and Liu [1] used the ASTM method C39-96 developed for concrete briquettes [21] to measure the compressive resistance of biomass logs.

G. Impact Resistance

An impact resistance (or drop resistance or shatter resistance) test can simulate the forces encountered during loading and emptying of densified products from trucks onto the ground, or from chutes into storage bins. Pietsch [3] suggested that drop tests could be used to determine the safe height of drop of briquettes production.

The impact resistance was tested using two methods in this study. Briquettes were tested by adapting the ASTM method D440-86 of drop shatter for coal [22]. The briquettes were dropped twice from 1.83 m onto a concrete floor. An impact resistance index (IRI) introduced by Richards [23] was used to evaluate the impact resistance of the briquettes. The IRI is calculated from IRI=(100 x N)/n, where N is the number of drops, and n is the total number of pieces after N drops. Because two drops were used as standard, the number of drops N in the above equation is always 2, and maximum value of IRI is 200 [1]. It should be mentioned that frequently when a briquette hits the concrete floor it breaks into pieces of various sizes ranging from large pieces to fine particles. When the number of pieces was counted in a test, the small pieces that weighed less than 5% of the initial weight of the briquettes were not included in the calculation of the IRI. After the first drop, all the pieces that weighed less than 5% of the original weight of the briquette were not collected and dropped for a second time. This method was also used by Li and Liu [1].

The other method used for measuring shatter resistance is to calculate the percentage loss of weight from shattering. Each briquette was subjected to 10 repeated drops from a height of 1 m onto a concrete surface. The percentage loss was then calculated. This method was also used by Lindley and Vossoughi [24]. Raghavan and Conkle [25] developed a similar test for coal pellets.

III. Results and Discussion

A. Effect of Moisture Content on Density

The effect of moisture content on briquette density was studied extensively at two periods: (i) five minutes after ejection, and (ii) five days after ejection. Fig. 1 shows both densities of the briquettes (5 minutes after ejection and 5 days after ejections) made at 16.5 MPa without holding time as a function of the initial moisture content. When the moisture was lower than 11.7%, the briquettes had low densities and disintegrated easily when subjected to small handling forces. They tended to absorb moisture from the air and expanded significantly, becoming fragile in a few days. On the other hand, the briquettes with an initial moisture content of more than 16.5% were not able to maintain good quality for long because their volumes grew markedly, immediately after ejection from the die, and significant cracks appeared on the surface. Fig. 1 also shows the rather equal change of the briquette density for different values of initial moisture

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content after 5 days through the whole test period. Briquettes made at around 12.6% initial moisture content had the highest density and the surface was smooth without visible cracks.

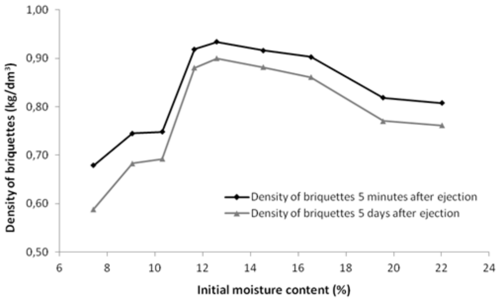

Fig. 1 Variation of briquette density of spruce sawdust compacted at 16.5 MPa by initial moisture content

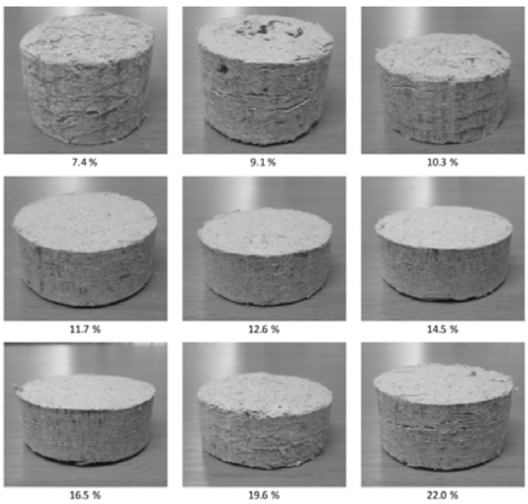

Fig. 2 Biomass briquettes with different initial moisture content of spruce sawdust compacted without holding time at 16.5 MPa and at room temperature using the BrikStar 200 industrial manufacturing hydraulic briquetting press; optical comparison of the surface quality

The surface quality of briquettes with different initial moisture content is compared visually in Fig. 2. Cracks and porosity decreased with increasing initial moisture content up to 11.7% w.b. Moisture content higher than 14.5% caused an increase in the surface porosity and cracks appeared again. The best surface quality was achieved in the moisture range from 12.6% to 14.5%.

The results of experiments also showed that the weight of individual briquettes decreased slightly with the increasing moisture of the raw material. All control parameters of briquetting press and particle size distribution were constant. This phenomenon is caused by changes in the bulk density of sawdust when changing its moisture content.

B. Expansion, Physical Changes and Long-Term Performance of the Briquettes

Briquettes made from raw material of around 12.6% moisture content had the highest density and good long-term performance. This phenomenon can be seen from Fig. 3, which shows the expansion of the briquettes made of spruce sawdust at different initial moisture contents after 5 days and 90 days of storage. The longitudinal expansions (elongations) were calculated based on the lengths of the briquettes measured 5 min after ejection from the die, and then re-measured 5 days and 90 days later. The elongation was reduced when the initial moisture content increased. The longitudinal expansions of briquettes after 5 days were less significant than after 90 days of storage. Experiments proved the briquette length shrinking (negative elongation) during the first 5 days when the initial moisture content exceeded around 21%. After 90 days, the briquette length shrank when initial moisture content exceeded 15 %. The reason for this negative extension was the reduction of briquette moisture as a result of air drying during storage.

As Fig. 3 shows, the elongation also depends on the storage time. The longer the storage time after ejection the more significant the elongation will be. The study shows that only elongation of briquettes made of material with 12.6% initial moisture content were independent of storage time.

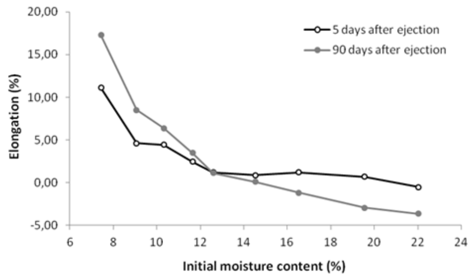

Fig. 3 Variation in elongation of briquettes by initial moisture after 5 days and 90 days

Fig. 4 shows the percentage change in density and moisture of briquettes of spruce sawdust by initial moisture content after five days. This behavior represents the global physical changes of the briquettes. Density change does not describe the physical changes completely. Although density is a function of mass and volume, not only do changes in weight (water included) and dimensions need to be controlled, but also the moisture changes need to be controlled separately. To evaluate the most stabile state of briquettes, changes to the moisture content also need to be included. Results shown in Fig. 5 as a cumulative absolute value of physical changes reveal that the briquettes stored for 5 days at indoor conditions (relative humidity from 50% to 60%, temperature around 20°C) achieved a slight change of cumulative physical changes up to 16.5% of initial moisture content. The percentage change after 5 days of storage was calculated based on the density and moisture of the briquettes, measured 5 min after ejection from the die, and then re-measured 5 days later. Absolute values of both physical changes - percentage change in briquette moisture and percentage change in briquette density, during the storage time between the fifth and the ninetieth day after ejection can be seen in Fig. 6. During the mentioned storage time, the physical changes of the briquettes showed the minimum values in the range of initial moisture content from 10.3% to 14.5%. The best value of initial moisture content for the storage process is 12.6% when the absolute value of physical changes of briquettes was up to 3.6%.

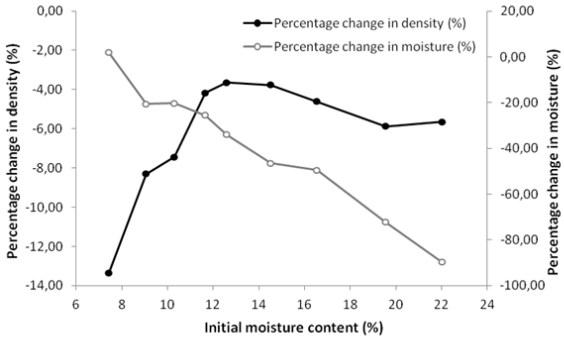

Fig. 4 Percentage change in density and moisture of spruce sawdust briquettes by initial moisture content after 5 days

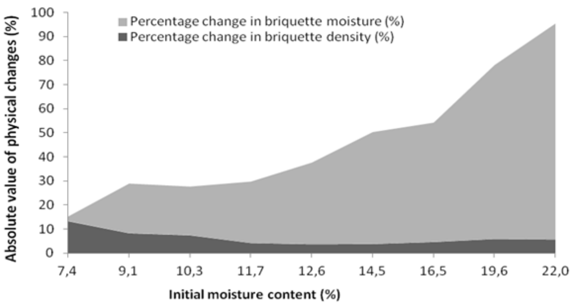

Fig. 5 Absolute value of both physical changes - percentage change in briquette moisture and percentage change in briquette density 5 days after ejection

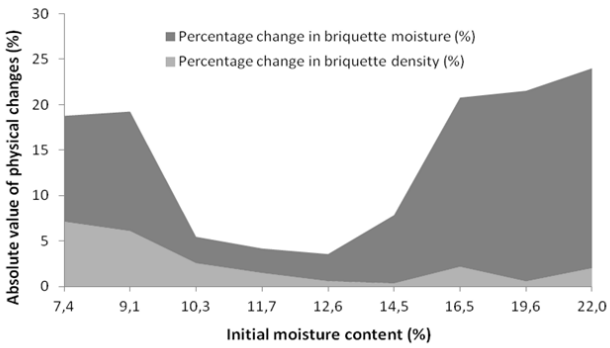

Fig. 6 Absolute value of both physical changes - percentage change in briquette moisture and percentage change in briquette density during the storage time between the fifth and the ninetieth day after ejection

C. Effects of the Change in Moisture Content

The initial moisture content of spruce sawdust was measured just before densification. The moisture content of briquettes was measured at three stages - 5 minutes, 5 days, and 90 days after the densification. The briquettes were stored under the same conditions all the time, at room temperature, and with a stabile relative humidity of 50-60% in the laboratory. Despite pressing at low temperature (up to 30°C) a slight reduction in moisture was already detected 5 minutes after ejection. A significant reduction in moisture was found 5 days after densification. These results are shown in Fig. 7. The briquettes with different initial moisture had a tendency to achieve a moisture content equilibrium after just 5 days, but the stable state of moisture content to near 9% for each sample with different initial moisture content, could be seen after 90 days.

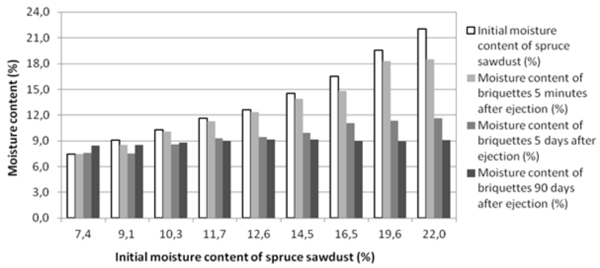

Fig. 7 Comparison of moisture changes during the densification process, and during storage times of 5 days and 90 days after ejection

D. Effects of Moisture Content on Compressive Resistance and Impact Resistance

The highest compressive resistance (27.4 $N.mm^{-1}$) was noted in the briquettes produced from the sawdust with an initial moisture content of 12.6%. As is shown in Fig. 8, the briquettes with initial moisture content lower than 11.7% had significantly low compressive resistance, something which could cause rapid disintegration of the briquettes under handling and storage. The compressive resistance of the briquettes slightly decreased with increasing initial moisture content from around 13% w.b.

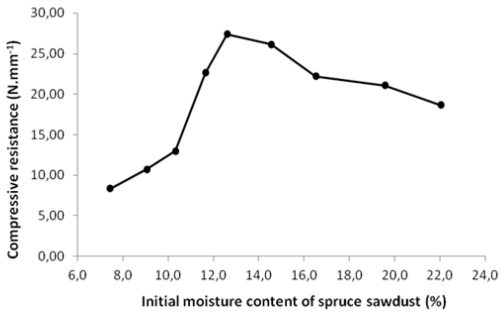

Fig. 8 Variation of compressive resistance with initial moisture content of spruce sawdust

The effect of initial moisture content on impact resistance was determined by two methods. The first one was carried out by adapting the ASTM method D440-86 of drop shatter for coal [22], where five briquettes from each sample were dropped twice from 1.83 m onto a concrete floor. A calculated average value of impact resistance index (IRI) was used to evaluate the impact resistance of the briquettes (Fig. 9). The maximum value of IRI (200) was reached by the briquette samples with initial moisture content of 11.7% and higher. Most of the briquettes with initial moisture content lower than 9% shattered, which is important to know when transporting, handling and storing.

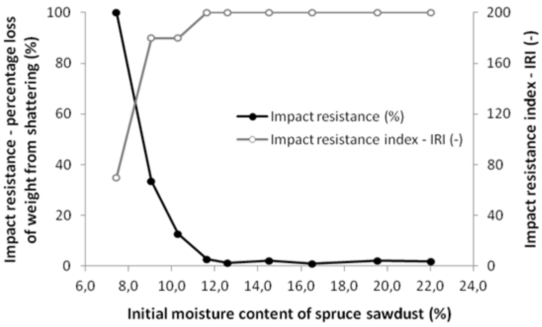

Fig. 9 Variation of impact resistance and impact resistance index with initial moisture content of spruce sawdust

The second method used to determine the impact resistance represents the percentage loss of weight from shattering. Five briquettes from each sample were subjected to 10 repeated drops from a 1 m height onto a concrete surface. The impact resistance as a percentage loss was then calculated. The results of this method, which are shown in Fig. 9, confirmed the results of the previous test method. The briquettes with initial moisture content equal to 11.7% and higher showed the smallest percentage loss of weight (only around 2%). Most of the briquettes with lower initial moisture content broke into many pieces after just a few drops. Surprisingly the percentage loss of weight did not increase by increasing the initial moisture content from 11.7% to the tested 22.0%.

IV. Conclusion

Spruce sawdust as a wood waste can be densified into high quality briquettes through high-pressure compaction. Briquettes were produced in an industrial scale process with a hydraulic briquetting press. For all briquettes studied, the same technical and technological conditions except the initial moisture content were maintained. The appropriate moisture range for producing good quality briquettes is from 11% to 16%, and the optimum is in the neighborhood of 13%. When the moisture content of spruce sawdust is close to the optimum (13%), a pressure of just 16.5 MPa can produce briquettes with a dry density higher than 0.8 kg.dm-3 (wet density higher than 0.9 kg.dm-3).

The best surface quality of briquettes can be achieved in the range of moisture content from 12.6% to 14.5%. The study shows that the expansion of briquettes made of material with 12.6% initial moisture content was independent of storage time, with a value of 1.2% of elongation.

In the range of initial moisture content of up to 16.5%, lower physical changes could be seen on the fifth day after ejection.

The smallest values in the physical changes of briquettes during storage time between the fifth and the ninetieth day after ejection were seen in briquettes with a range of initial moisture content from 10.3% to 14.5%. The best value of initial moisture content for the storage process is 12.6%, when the absolute value of physical changes was up to 3.6 %.

When the effect of moisture change was studied, all the briquettes with different initial moisture content had a tendency to achieve an equilibrium moisture content close to 9% after 5 days of storage. This phenomenon was confirmed by subsequent measurement after 90 days of storage.

The highest compressive resistance was achieved by the briquettes with an initial moisture content of 12.6%, but a suitable range of moisture content was 11.7%-16.5%. The two methods used for determining the impact resistance of briquettes showed that the best initial moisture content was 11.7% and higher.

When spruce sawdust has initial moisture content outside of the studied range of 11.7-16.5%, the production of good quality briquettes in industrial scale processes with a hydraulic briquetting press is very difficult. The best value of initial moisture content sufficient to cover all criteria was discovered to be 12.6%.

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