Properties of *Pinus* sp. Pellets Prepared after In-line Pre-compaction with Torrefaction

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Pelletizing and torrefaction increase biomass energy density, generating a more homogeneous fuel with lower moisture, enabling it to burn in equipment with high energy efficiency. This work evaluated the quality of pellets made from wood particles that had been densified and torrefied in an extruder-type system. Particles of the *Pinus* sp. wood were torrefied at 200, 250, and 300 °C for six minutes and then compacted to produce pellets. The physical, chemical, and mechanical properties of these pellets were evaluated and compared with standard ones. Torrefaction modified the pellets properties by increasing ash, fixed carbon, higher heating value, and the energy density, while reducing the volatile matter and equilibrium moisture content. The mechanical durability of the pellets was lower than that defined by the European, German, and American standards. The torrefaction pre-compaction route with torrefied particles at a temperature of 300 °C was the most efficient for energetic use, compared to the *in natura* biomass. The latter has negative aspects such as great variation in size (length and diameter) and density besides high moisture content.

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INTRODUCTION

Biomass is a clean and renewable material that can replace conventional energy sources such as oil and natural gas. This substitution would reduce greenhouse gas emissions and dependence on fossil fuels (Luque *et al*. 2008), through use of biofuel such as pellets and briquettes (Lee *et al.* 2011; Stelte *et al.* 2011). The formation of pellets increases the energy density, reduces moisture content, and reduces transport and storage costs, allowing the standardization in size and composition and facilitating the use of this material in domestic and industrial ovens (Warajanont and Soponpongpipat 2013; Boschetti *et al*. 2019). The pelletization of elephant grass, eucalyptus wood, and sugarcane bagasse resulted in pellets with energy density between 9 and 10.2 GJ/m^3 (da Silva *et al.* 2018). The gain in energy density can be attributed to the increase in the density of the material (Araújo *et al.* 2016).

Torrefaction increases the energy potential of the biomass; however it reduces the density of the material (Wang *et al.* 2017; Silva *et al.* 2018; Dufourny *et al.* 2019). Compaction of material by pelleting minimizes density loss from torrefaction, enhancing the use of agricultural, forestry, or industrial residues as biofuel, reducing the negative aspects of *in natura* biomass, such as variation in size and density and high moisture content of this material (Chen *et al*. 2015; Whittaker and Shield 2017; Röder and Thornley 2018).

The biomass torrefaction is similar to a slow pyrolysis: subjecting the material to a temperature with oxygen restriction with the elimination of part of the volatile compounds, increasing the carbon content and heating value, and reducing the moisture content (Li *et al*. 2016; Atreya *et al*. 2017; Wang *et al*. 2017). Torrefaction is a process between drying and pyrolysis of the biomass and is faster than the latter (Peng *et al.* 2013; Dhyani and Bhaskar 2018; Dufourny *et al*. 2019). The torrefaction of *Eucalyptus grandis*, *Pinus maximinoi*, *Pinus patula*, and *Gmelina arborea* wood at 300˚C showed yield between 55 and 75%, resulting in material with 70% volatile matter (70 wt%), 18.7 MJ/kg heating value and less than 1% ash content (Perez *et al.* 2019).

Pelletizing and torrefaction increase the biomass energy density, generating a more homogeneous fuel with lower moisture and allow the use of modern and more efficient burning equipment (Stelte *et al.* 2011). The combined use of these processes is indicated for the production of a more competitive product in relation to non-renewable fuels.

Torrefaction may add quality to the pellet produced, but the process during the preparation of the particles or after pelletizing needs further research, as the pellet industry does not have the ideal equipment that is capable of pelletizing in co-production with torrefaction.

This work evaluated the physical, mechanical, and chemical properties of pellets produced with torrefied *Pinus* sp. wood particles.

EXPERIMENTAL

Pinus sp logs were sawn on a circular saw and the sawdust produced was air-dried on a flat surface, covered and protected from rain and winds until reaching 20% moisture (dry basis). The initial sawdust moisture was 220% on a dry basis, the drying period lasted 55 days and samples were weighed daily to monitor moisture loss. The particles of these residues were sieved and selected the fraction between a 3.0 and 1.0 mm.

Torrefaction Process and Particles Characterization

Torrefaction was conducted with an endless screw reactor developed in the Panels and Wood Energy Laboratory (LAPEM/UFV in Viçosa, Minas Gerais, Brazil) (Carneiro *et al.* 2016). The prototype of this equipment was a semi-continuous screw reactor reusing volatile gases in the heating system (Fig. 1) (Silva *et al.* 2018). The prototype was developed in the laboratory, and the patent was subjected to INPI (National Institute of Intellectual Property – Brazil) under the process number BR10 2016 010484 0. The primary structure of this reactor has three systems essential to most reactors that facilitate the dry torrefaction: (I) transport; (II) heating; and (III) cooling. The first system moves the biomass for the homogenization process, which can be classified as continuous, intermittent, or mixed. The second system transfers indirect heat to the biomass under controlled conditions for indirect heating produced in furnace/burner. The third system releases the torrefied biomass within the safe temperature limits. Temperatures of 200, 250, and 300 °C with residence time of 6 min were used for pellet production.

Fig. 1. Lateral view of the screw reactor for thermal treatment of lignocellulosic biomass: 1 - input biomass; 2 - heating system; 3 - cooling system; 4 – burner; 5 - connection chimney with the burner; 6 - torrefied biomass exit

The *Pinus* sp. samples were ground in a Wiley knife mill with a 2 mm sieve opening, and the material that passed the 40-mesh sieve and was retained in the 60-mesh sieve was used to energy characterization.

The content of volatile matter, ash, and fixed carbon were determined according to ABNT NBR 8112 (1986), and the higher heating value was measured according to ABNT NBR 8633 (1984).

Production, Evaluation and Classification of Pellets

The pellets were produced with “in natura” *Pinus* sp. wood and torrefied at 200, 250 and 300 ºC. Pellets were produced in laboratory pelletizing machine press with a 30 kg.h^{-1} production capacity. The average pelletizing temperature was 100 ± 3 °C. The pelletizing matrix (Amandus Kahl, model 14-175) was pre-heated in oil at 200 °C for approximately 30 min. The matrix was heated in an external heat source, which decreased the material expenditure to reach the minimum pelletizing temperature, that was near to 100 °C ± 3 °C.

The pelletizing machine is a system with a motor reducer, speed controller, and continuous screw. Four nozzles were installed, along the continuous screw, to inject at a pressure of 0.8 kgf/cm^2, the steam produced by autoclaving.

The pellet moisture content in dry basis was determined according to DIN EN 14 774-2 (2010). The bulk density (kg/m^3) of the pellets was obtained according to DIN EN 15103 (2010) in samples conditioned in a climatic chamber at 65% relative humidity and 20 °C.

The samples of pellets made from torrefied wood were transformed into sawdust in a knife mill according to TAPPI 257 om-85 (1985) to determine the higher heating value and percentage of ash and volatile matter. The material was sieved and the fraction that crossed the sieve with 40 mesh and was retained in the sieve with 60-mesh was used.

The higher heating value of the pellets was obtained according to DIN EN 14918 (2010) using adiabatic calorimeter IKA300, the energy density (GJ/m^3) by the product of the useful heating value useful and bulk density, the diameter (mm) and length (mm) of the pellets according to DIN EN 16127 (2012) and hardness (kg) in a durometer. The mechanical durability and percentage of pellet fines were determined according to DIN EN-15210-1 (2010).

Pellets were classified according to CEN/TS 14961-2 (European), DIN 51731 (German), SS 18 71 20 (Swedish), Önorm M 7135 (Austrian), CTI-R 04/05 (Italian), USA PFI (American) and ISO 17225-2 (international standardization) for the commercialization of this material.

Statistical Analysis

The results of the properties evaluated for the pre-torrefied pellets were analyzed according to a randomized design with four treatments (control and particle torrefied temperatures). The averages were grouped with Tukey's test ($p \leq 0.05$) to evaluate the physical and mechanical pellets properties. Statistical analyzes were performed with STATISTICA 8.0 software (StatSoft, Tulsa, USA). Statistical testing is necessary to accurately assess if the means of each treatment are different.

RESULTS AND DISCUSSION

The volatile matter content of the pellets decreased as the torrefaction temperature of the particles increased, mainly at the treatment at 300 °C, with 4.3% reduction, compared with the control (Table 1). Polar extractives and hemicelluloses are volatile materials and represent a considerable part of the wood chemical composition. These compounds are unstable under high temperatures and removed during torrefaction (Mészáros *et al.* 2007; Brito *et al.* 2008). This is important because these compounds are rich in oxygen (mainly CO and CO_2), and its removal increases the fixed carbon content and, consequently, the energy characteristics of the material, such as higher heating (Peng *et al.* 2013; Park *et al.* 2013; Pereira *et al.* 2013). The volatile compounds oxidize and release energy faster than the oxidation of the biofuel fixed carbon, which increases the rate of biomass burning (Santos *et al*. 2013).

The ash content increased with the torrefaction temperature. Thus, the ash content after torrefaction at 300 °C was 31.3% higher than the control (Table 1). The increase in the ash content is due to the thermal degradation and volatilization of the hemicelluloses (Pereira *et al*. 2013; Korus and Szlek 2015). The thermal degradation process eliminates organic components without affecting the inorganic ones (Phanphanich and Mani 2011), but the increase in ash content of the biomass decreases its densification, energy properties, and the cost of obtaining biomass energy (McKendry 2002). In addition, ash contributes to combustion residues, and higher concentrations require more frequent cleaning of the biomass deposits (Thek and Obernberger 2010). Ash content indicates the biofuel quality as solid fuels (pellets and briquettes) by reducing the higher heating value, increasing the frequency of cleaning of industrial furnaces, and corroding them (Pereira *et al.* 2013).

The fixed carbon of the pellets increased with the torrefaction temperature. For instance after torrefaction at 300 °C, the fixed carbon was 20.6% higher than the control (Table 1). The increased fixed carbon is due to the degradation of compounds rich in oxygen and hydrogen (Phanphanich and Mani 2011). This includes the lignin, which is a more thermally stable compound with a complex structure and lower degradation during torrefaction (Cai *et al.* 2017). The increase in fixed carbon is also due to the proportional increase in the lignin, a compound with a high carbon content and lower hydrogen and oxygen, due to the elimination of the hemicelluloses and part of the cellulose (Pasangulapati *et al*. 2012; Pereira *et al.* 2013; Dhyani and Bhaskar 2018). The increased fixed carbon content can be used as a basis to select biomass for combustion (Reis *et al.* 2012). Fuels with high fixed carbon content and low volatile materials tend to burn slower with energy released for a longer time during burning (Furtado *et al.* 2010). The main advantage of torrefaction is to concentrate the carbon in the fuel to reduce iron ore and provide energy to the blast furnace in the steel industry (Protásio *et al.* 2015). The energy potential of pellets produced with particles torrefied at 300 °C was higher due to their higher net heating value (in the absence of moisture), energy density, and fixed carbon.

The equilibrium moisture content (EMC) of the pellets produced with particles pre-torrefied at 300 °C was 29.7% lower than the control (Table 1). The lower EMC of the pellets produced with particles pre-torrefied at 300 °C can be attributed to the faster degradation of cellulose and hemicellulose at this temperature, reducing water adsorption capacity and EMC (Waters *et al.* 2017). The pellet EMC is directly related to the water amount connected to the cell wall by hydrogen bonds (Whittaker and Shield 2017), and its reduction makes the particles more cohesive and increases the period that they can be stored (Tumuluru *et al.* 2011). The lower equilibrium moisture increases the useful heating value of the biomass, and it reduces the damage by microorganisms in stored biomass (Swithenbank *et al.* 2011; Tumuluru *et al.* 2011).

The bulk density of the pellets manufactured with the torrefied material at 300 °C was 13.1% higher than the control (Table 1). The smaller density of the pellets produced with torrefied material explains its higher bulk density and increases the surface area and the contact and cohesion between the particles (Boschetti *et al*. 2019). The higher lignin content of *Pinus* also increases the agglutination between the particles during compaction and explains the higher bulk density of the pellets made with densified and torrefied material from this plant. This makes unnecessary the addition of natural binding agents to produced pellets and briquettes compacting *Pinus* spp. (Brito *et al.* 2008). The bulk density determines the storage and transport conditions of pellets and it is directly linked to the concept of energy density, *i.e.*, the amount of energy transported per unit volume (Sotannde *et al*. 2010).

The heating value of pellets produced with particles torrefied at 250 and 300 °C was 1.1 and 2.6% higher, respectively, than in the control (Table 1). The higher net heating value (in the absence of moisture), of torrefied biomasses is due to the volatilization of hemicelluloses, which has low carbon content and degree of polymerization, this fact increases the lignin content (McKendry 2002; Peng *et al*. 2013), compound with higher carbon content and thermal stability (Panneerselvam *et al*. 2013; Nhuchhen *et al*. 2014). Therefore, the disruption of chemical bonds with higher binding releases more energy and, consequently, the biomass heat power. The higher net heating value is one of the main factors to select the biomass for energy purposes, mainly to produce pellets to release the maximum amount of energy in the combustion. The fixed carbon content of the biomass must be high due to its direct positive relation with the heating value (Peng *et al*. 2013).

The energy density increased by 16.0% in the highest temperature for pellets produced with particles torrefied at 300 °C in relation to the control (Table 2). The increase in the energy density of the pellets with the increase of the torrefaction temperature was expected because this process partially degrades the hemicelluloses, increasing the proportion of high carbon components connected by strong carbon-carbon bonds, such as the resulting fraction of lignin, increasing the higher heating value of biomasses (Kim *et al*. 2012). The energy density is an important parameter because it summarizes the physical and chemical characteristics of the product between higher heating value and density (Garcia *et al*. 2013; ÖzyuğUran and Yaman 2017).

Table 1. Mean Values of Volatile Matter (VM), Ash Content, Fixed Carbon (FC), Equilibrium Moisture Content on Dry Basis (EMCdb), Bulk Density (D), and Higher Heating Value (H) (mean ± standard error) of Pellets Produced with Torrefied Particles (pre-pelletizing torrefaction) at Different Treatments (Treat.)

Treat.	VM (%)	Ash (%)	FC (%)	EMCdb (%)	D. (Kg.m^{-3})	H (MJ.kg^{-1})
Control	$86.34^{0.60}$a	$0.32^{0.03}$c	$14.05^{0.42}$c	$10.73^{0.14}$a	$572.30^{3.65}$c	$16.92^{0.08}$b
200 ºC	$84.76^{0.60}$a	$0.35^{0.02}$bc	$14.06^{0.09}$c	$9.24^{*0.63}$ab	$620.63^{*5.39}$b	$17.04^{0.10}$ab
250 ºC	$85.55^{0.29}$a	$0.40^{*0.02}$ab	$14.88^{0.22}$b	$9.03^{*0.94}$bc	$637.76^{*5.43}$ab	$17.10^{0.23}$ab
300 ºC	$82.64^{*0.48}$b	$0.42^{*0.02}$a	$16.94^{*0.14}$a	$7.54^{*0.21}$c	$647.27^{*10.55}$a	$17.36^{0.04}$a

Means followed by the same letter, per column, do not differ by the Tukey test (α= 0.05). * Means differ from the control by the Dunnett test (α= 0.05).

Table 2. Energy Density (ED), Diameter, Length, Mechanical Durability (MD), Hardness (Hard.), and Fines Content (mean ± standard error) of Pellets Produced with Torrefied Particles Pre-pelletizing

Treatment	ED (GJ.m^{-3})	Diameter (mm)	Length (mm)	MD (Kg)	Hard. (kg)	Fines (%)
Control	$9.69^{0.08}$c	$6.08^{0.05}$a	$17.35^{1.43}$c	$20.67^{2.94}$c	$90.29^{1.42}$b	$0.44^{0.04}$a
200 ºC	$10.62^{*0.13}$b	$6.03^{*0.04}$b	$18.84^{*1.11}$b	$23.93^{5.26}$bc	$95.32^{0.34}$a	$0.37^{0.07}$ab
250 ºC	$10.87^{*0.15}$ab	$6.00^{*0.04}$b	$18.72^{*0.89}$b	$25.73^{*2.66}$b	$95.41^{0.61}$a	$0.28^{*0.01}$b
300 ºC	$11.24^{*0.20}$a	$6.02^{*0.03}$b	$20.23^{*0.77}$a	$36.33^{*3.81}$a	$95.46^{0.16}$a	$0.14^{*0.02}$c

Means followed by the same letter, per column, do not differ by the Tukey test (α= 0.05). * Means differ from the control by the Dunnett test (α= 0.05).

The diameter of the pellets that had been formed from particles prepared with different torrefaction temperature was similar. However, diameters became smaller as the temperature increased (Table 2). The similar diameter of the pellets at various torrefaction temperatures of the particles can be attributed to the elimination of unstable components (holocelluloses) from the wood, causing a rearrangement in the physical structure of the pellet (Silva *et al*. 2015). The constant pressing temperature of 120 °C plasticizes the lignin that acts as an adhesive, increasing the contact between the particles and reducing the expansion due to the lower hygroscopicity and, consequently, decreasing its diameter (Furtado *et al*. 2010).

The pellets produced with torrefied particles was 16.6% longer in the treatment at 300 °C (Table 2) with good adhesion of torrefied particles reducing the losses of material during pelletizing. The higher average length of the pellets produced with particles roasted at 300 °C can be explained by the increased adhesion force between the particles, and the consequent increase in the mechanical resistance of the pellets. (Liu *et al*. 2013). The higher compaction of the material increases the contact area between the particles, mass per unit volume and, consequently, the mechanical strength of the pellets (Zamorano *et al*. 2011), making them less brittle and longer.

The hardness value of the pellets produced with the particles torrefied was higher than in the control, with an increase higher than 5 % (Table 2). The higher hardness value of the pellets produced with the particles torrefied is possibly due to the decrease in their equilibrium moisture content. Torrefaction reduces the number of hydroxyl groups (-OH) and, consequently, the pellet hygroscopicity (Shang *et al*. 2014; Atreya *et al*. 2017). This allows a smaller number of hydrogen bonds between the molecules of the material, reducing those with the water and making the particles more cohesive, and consequently a more resistant pellet (Liu *et al*. 2014). Moreover, this gain was possible without the addition of a binding agent, which enhances the economic and environmental viability of the pellet torrefaction.

The mechanical durability of the pellets was higher with biomass torrefied at 300 °C. The increase in mechanical durability with torrefaction is mainly due to the reduction in the equilibrium moisture content as the temperature increases. The water present in the pellets, between 8 and 12%, occupies the place of hydrogen bonds and, with the lignin, is fundamental for the cohesion between the wood particles (Liu *et al*. 2014).

Analyzing the effect of torrefaction temperature in the quality of the pellets, the hardness, bulk density, and durability of the pellets produced with the treated particles at 300 °C were better, which can increase the competitiveness of the product on the market. The increased hardness and durability of torrefied particle pellets will reduce the mass loss during transport and handling (Kaliyan 2018).

The fines content of pellets of torrefied particles at 300 °C was 68.2% lower than in the control. Torrefaction process increases the compaction and improves the distribution of a larger mass per unit volume and, consequently, the adhesion between particles and the strength of the material by retracting the material of the particles (Liu *et al*. 2013). The lower equilibrium moisture content of the pellets with torrefaction reduces the generation of fines and increases the number of carbon-carbon bonds that are more energetic, making the material more resistant (Liu *et al*. 2014). The amount of fines generated by the pellets is important for the commercialization of this biomass, as they affect the density of the product, transport and, in large quantities, increases the chance of spontaneous combustion (Garcia *et al*. 2013).

Most pellets produced with torrefied particles met the conditions established by the standards (ISO 17225-2 2014, DIN Plus 2011, DIN 14961-2 2011 and US PFI standards), except for the mechanical durability. Pellets manufactured with torrefied particles at 200 °C did not meet the requirements of the *Premium*, *Standard* and *Utility* categories of the latter category. Those made of particles torrefied at 250 and 300 °C could be marketed in the American market, respectively, as Utility and Standard. This does not affect the economic viability of the pellets, as the pine wood can be obtained as waste in sawmills, reducing its price and the process of torrefaction of pelletizing is inexpensive. Adequacy to European and American international standards is important in specifying the minimum characteristics of the largest pellet market in the world.

CONCLUSIONS

1. Bulk density, moisture content, and mechanical durability were better at making the pellets suitable for export.
2. The heat treatment of the particles increased the higher heating value, fixed carbon, mechanical durability and reduced the fines content of the pellets.
3. Pre-pelletizing torrefaction improved the properties of fixed carbon, higher heating value and hardness of the pellets.
4. The value of mechanical durability of the pellets in all treatments did not meet the minimum requirements of ISO 17 225-2 (international standard), CEN/TS 14961-2 (European), DIN Plus 14961-2 (German) and USA PFI (American). Pellets of all treatments met at least one category of standards SS 18 71 20 (Swedish) and CTI-R 04/05 (Italian).

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